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# Jatropha Oil Refining Process and Biodiesel Conversion: Mass and Energy Balance

Jinjuta Kongkasawan*[1] and Sergio C. Capareda*

***Abstract –*** *Jatropha curcas L. is considered as an alternative energy source to help solve the energy crisis. The refining process is essential during the biodiesel making process because it can enhance the yield and quality of the product. Results revealed that the gross heating value of refined oil increased significantly from the crude oil with lower viscosity and lighter clearer color. The mass conversion efficiency and energy recovery of refined oil were obtained at 75% and 78%, respectively. NaOH was selected as a catalyst for the transesterification process of refined oil. For the mass conversion of the transesterification process, 83% of biodiesel and 13% of glycerin were obtained. Moreover, 99% of energy recovery was obtained from biodiesel and glycerin. According to the ASTM characterization of Jatropha biodiesel, only high viscosity problem was encountered. To solve this problem, blending Jatropha biodiesel with the commercial fuel was recommended to lower its viscosity. Furthermore, the gross heating value of biodiesel was obtained at 39.5 MJ/kg. Due to a high mass conversion efficiency and energy content of Jatropha products, Jatropha has the potential to serve as an alternative energy source.*

***Keywords*** – Jatropha biodiesel, mass and energy balance, oil refining, transesterification.

## 1. INTRODUCTION

Jatropha is a drought-resistant tropical tree with a minimum necessity of water compared to other fruit trees, and it can produce seed yield with an acceptable quality. It is spread in the wild and agricultural tropical zone of Central America, South America, Africa, India, South Eastern Asia, and Australia. Jatropha is likely to grow in an area with intensive sunshine as found in a savanna or desert region [1]. A Jatropha seed contains a high fraction of oil compared to other oil plants. Koh and Ghazi [2] reported the percentage of oil content in Jatropha seeds that was about 50-60 wt. percent non-edible oil.

Several researchers also investigated the properties of Jatropha crude oil. Achten et al. [3] suggested that the broad range in the values of the free fatty acids, unsaponifiables, acid number, and carbon residue indicated that the environmental conditions and Jatropha genetics affected the oil quality. The seed oil consisted of more than 70% unsaturated fatty acids with a higher fraction of oleic acid than linoleic acid. Moreover, the high content of monounsaturated oleic acid represented the semi-drying property, which could be applied to industrial surface coating [4]-[5].

Vegetable oils are one of the promising feedstock for biofuel production due to their high energy, lower sulfur and aromatic content, and capabilities of renewing and biodegrading [2], [6]. Triglycerides, the main component in vegetable oils, consist of three fatty acid molecules bonded to a glycerin. The bulk energy in triglycerides has made these molecules an interesting supply for biodiesel manufacturer [7]. Both edible and non-edible vegetable oils such as canola, soybean, corn, madhuca indica and Jatropha curcas are appropriate for diesel substitution.

Many researchers consider Jatropha oil a prospective fuel substitution, due especially to its ability to be cultivated in dry and marginal lands [8]. In contrast with other oil plants, Jatropha oil has low acidity, good oxidation stability compared to soybean oil, low viscosity compared to castor oil, and well cooling properties compared to palm oil [5], [8]. Numerous studies on Jatropha as a potential feedstock for biodiesel production have been conducted. Foidl et al. [9] studied the possibility of Jatropha curcas L. as a source for biofuel production in Nicaragua. They concluded that the Jatropha crude oil was suitable for biodiesel making. The biodiesel obtained from transesterification process had high qualities that reached the standards for vegetable oil- based fuels and could be used in diesel engines without modifications.

Biodiesel has already been verified as suitable fuel for diesel engines. Biodiesel from vegetable oils are produced via a transesterification process. Transesterification is the chemical reaction of triglycerides with alcohol in the presence of a catalyst. A catalyst is a supplement for the reaction, which helps the reaction to be completed in a short time. Typically, methanol is selected for biodiesel production because of its low price. The products from a successful transesterification process are crude glycerol and mono-alkyl ester, as we called it biodiesel [2].

However, the oil purification process is important prior to the biodiesel production. The acidity, water, phosphorous and peroxide value that are contained in the crude vegetable oil can reduce the biodiesel quality [10]. It has also been reported that refining the oil before performing a biodiesel production affects the yield and

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quality of the biodiesel [11]. The common refining process includes purification, degumming, neutralization, bleaching, and deodorization. Filtering or centrifugation is a simple way for purification in order to remove water and non-dissolved impurities. The gum will be removed by the treatment with $H_2SO_4$ or hot water or absorbent materials. The popular neutralizing method is the alkali neutralization that makes the oil Ph neutral. Finally, bleaching and deodorization will eliminate the pigments, odorous matters, and high melting acylglycerols. Thus the goal of the refining process is to improve the oil quality by removing the undesirable compounds such as phospholipids, free fatty acids and pigments [12]-[13].

Therefore, the main goal of this study is to perform a complete refining process on the Jatropha crude oil, conduct the transesterification process, and evaluate a complete mass and energy balance of the process. The American Society for Testing Materials (ASTM) standards D6751 will be performed on the resulting biodiesel product. The specific objectives were as follows:

a. Evaluate the efficiency of oil refining process.
b. Evaluate the efficiency of transesterification process and test the ASTM standards of the biodiesel.
c. Determine the energy and mass balance of Jatropha seed and energy potential in oil.

## 2. MATERIALS AND METHODS

### *2.1 Jatropha Oil Extractions*

Jatropha curcas L. seeds were obtained from the Bio-Energy Testing and Analysis Laboratory (BETA Lab) and used for the oil extraction by using a screw press machine (Komet Press, Model HFG 505 WN, Germany) at the BETA Lab. The operating temperature was selected at 140$^o$C since it was the condition that the maximum percent oil extracted was achieved [14]. The extracted oil was filtered using a 25-µm fiberglass filter membrane and then used for the refining process.

### *2.2 Oil Refining Process*

The oil refining processes of Jatropha was conducted in the same way as Santos' (2009) work. The details of oil refining procedures including degumming, neutralization, and dewaxing are provided in the next sections.

#### *Degumming*

The objective of the degumming process was to remove the phosphorus-based compounds, mainly lecithin and cephalin from the fresh oil before converting it into biodiesel. The removed substance was called "gums."

The filtered oil was heated to 55-60$^o$C and de-ionized water was added. The mixer was agitated for 1 hour with the temperature maintained at 55-60$^o$C. The hydrated gums were removed by centrifugation. Finally, the gums and the oil were separated, weighed and the results were recorded.

In order to make sure that all gums in the oil would be removed, the degumming process experiment was performed by varying the amount of de-ionized water added in the oil such as 2%, 4% and 6% by weight of the oil. If too much water were added in the oil, the water layer would appear after centrifugation. Therefore, the highest amount of de-ionized water was selected such that there was no water layer occurred.

#### *Neutralization*

In general, the crude oil contains free fatty acids from natural hydrolysis of the triglycerides. The neutralization process was performed in order that the free fatty acid would be removed prior to biodiesel making.

For the neutralization process, the degummed oil was mixed with potassium hydroxide (KOH) aqueous solution. The amount of KOH used was based on the free fatty acid content of Jatropha oil. Free fatty acids determination was done by following the AOCS Official Method Ca 5a-40. A contacting time of 30 minutes for the soap formation was allowed, and the mixture was separated by centrifugation. The neutralized oil was washed with a de-ionized water twice to make sure that all traces of soap were eliminated. Then the centrifuge was used to separate the oil and water. The oil and recovered soap were recorded.

#### *Dewaxing*

Vegetable oils usually contained high amount waxes that led to the cloud when refrigerated. Therefore, the waxes should be removed before a biodiesel conversion process.

The oil obtained from a neutralization process was mixed with a 5% (by weight of the oil) of NaOH aqueous solution and a 5% (by weight of the oil) of de-ionized water in a covered flask and placed in a chiller setting at 5$^o$C. The mixture was agitated for 4 hours. The soapy water wetted the waxes, which moved from oil to water phase. The waxes were then removed by centrifugation. The oil and the waxes were separated, and recorded their weights.

### *2.3 Transesterification Process*

The Jatropha refined oil was then converted into biodiesel via a transesterification process. The transesterification process was done by adding NaOH (0.4% wt of oil) which was used as catalyst, and methanol (12.5% wt of oil) into the refined oil. The reaction temperature was selected at 50$^o$C and allowed the contact for an hour. After that, the mixture was left in the separation funnel overnight. The glycerin was removed and the biodiesel was washed with warm de-ionized water before analyzing for its properties.

### *2.4 ASTM Characterization of Biodiesel*

Biodiesel obtained from the experiment was performed the ASTM characterization in accordance with ASTM D6751-08 standard. The ASTM testing procedures including cloud and pour point (ASTM D2500), flash point (ASTM D93), kinematic viscosity (ASTM D445),

acid number (ASTM D664) and gross heating value (ASTM D4809).

### *2.5 Mass and Energy Balance*

According to the initial oil input and the weights of the products, mass balance for the refining and transesterification processes were determined. The efficiency of Jatropha crude oil conversion into the by-products, refined oil, biodiesel, and glycerin namely "%Mass Conversion", and "%Mass Losses" were calculated using equations 1 and 2:

$$\%Mass\ conversion = \frac{W_{prod}}{W_{crude\ oil}} \times 100\% \quad (1)$$

$$\%Mass\ losses = 100\% - \sum(\%Mass\ conversion\ of\ all\ products) \quad (2)$$

where: $W_{prod}$ is the weight of products, kg
$W_{crude\ oil}$ is the initial weight of Jatropha crude oil, kg

For the energy distribution, the energy input to the system was computed based on the crude oil gross calorific value. Energy output was a summation of energy from the by-products including gums, soap, and wax, the refined oil, biodiesel, and glycerin acquired from a bomb calorimeter (refer to section 2.4 for heating value determination). The amount of energy recovery in the product and energy losses was calculated in terms of percentage using equations 3 and 4:

$$\%Energy\ recovery = \frac{W_{prod} \times HV_{prod}}{W_{crude\ oil} \times HV_{crude\ oil}} \times 100\% \quad (3)$$

$$\%Energy\ losses = 100\% - \sum(\%Energy\ recovery\ of\ all\ products) \quad (4)$$

where: $W_{prod}$ is the weight of products, kg
$W_{crude\ oil}$ is the initial weight of Jatropha crude oil, kg
$HV_{prod}$ is the gross heat value of the products, MJ/kg $HV_{crude\ oil}$ is the gross heat value of the crude oil, MJ/kg

## 3. RESULTS AND DISCUSSION

### *3.1 Degumming Process Experiment*

The degumming process was tested by adding different amounts of de-ionized water (2%, 4%, and 6% by weight of oil) into the heated oil. It was observed that a water layer occurred when 6% (by weight of oil) of de-ionized water was added. On the other hand, no water layer appeared with 2% and 4% of de-ionized water added. Therefore, 4% of de-ionized water was used to perform the degumming process during the oil refining procedure.

### *3.2 Oil Refining*

The Jatropha oil extracted from a screw press was then carried through a refining process, including degumming, neutralization, and dewaxing. After the completed refining process, approximately 75±0.5% of Jatropha refined oil was obtained with 4±0.6% gum, 8±0.6% soap, and 11±0.9% wax. The acid value of the crude Jatropha oil was found to be 0.2 mgKOH/g, which was relatively low. High free fatty acid composition would result in large amount of soap by-product in a neutralization process, which reduced the yield of biodiesel conversion [2].

### *3.3 Characterization of Refining Process*

The gross heating value of Jatropha crude oil, by-products from the refining process including gums, wax, and soap, refined oil, glycerin, and biodiesel were determined as shown in Table 1. It can be seen that the gross calorific value of refined oil increased a little from crude oil, changing from 37.6 MJ/kg to 39.0 MJ/kg. The waxes are high melting esters of fatty alcohol with long chain fatty acids. At low temperature, vegetable oils will transition from liquid to solid state (or waxes) because of the poor low temperature flow properties [15]. Therefore, this explains why wax still has a relatively high heating value.

The viscosity of the crude Jatropha oil and refined oil was determined according to the ASTM D 445-04. The viscosity of Jatropha crude oil was found at 37 cSt while the viscosity of Jatropha refined oil was reduced to 35 cSt. Adhvaryu *et al.* [15] stated that the deposition of waxy substances in oil led to the increase in viscosity. Therefore, removing the wax would result in a lower viscosity in refined oil. Moreover, it was found that the refined oil was clearer and had a lighter yellow color than the crude oil. This can be explained by the elimination of some unwanted compounds during the refining process.

### *3.4 Biodiesel Conversion and ASTM Characterization*

Refined oil was then converted into biodiesel via a transesterification process by using NaOH as a catalyst. As shown in Figure 1, the transesterification process yields the mass conversion of 83±1.0% wt biodiesel, 13±0.9% wt glycerin with only 4±2.0% wt losses. Mass losses could occur during the experimental process due to the chemical reaction and substance transfers. Then the energy losses can be calculated from the heating value of each product obtained. Moreover, Figure 2 demonstrates that 90±1.2% of total energy is obtained from biodiesel and 9±0.7% from glycerin. The degree of uncertainty in each of the HV values are quite small and usually less than one MJ/kg.

The ASTM characterization of biodiesel was conducted to ensure that the biofuel would meet the specification of standard fuels. The properties of the Jatropha crude oil and produced biodiesel are demonstrated in Table 2. It can be seen that converting crude oil into biodiesel can enhance the properties of the crude oil to meet the standard limitation. All properties of Jatropha biodiesel met the ASTM standard specification except for the kinematic viscosity, which was a little bit higher than the specification (7.8 cSt).

However, the addition of commercial fuel into Jatropha biodiesel can be the option to lower its viscosity and make it suitable for a diesel engine [16].

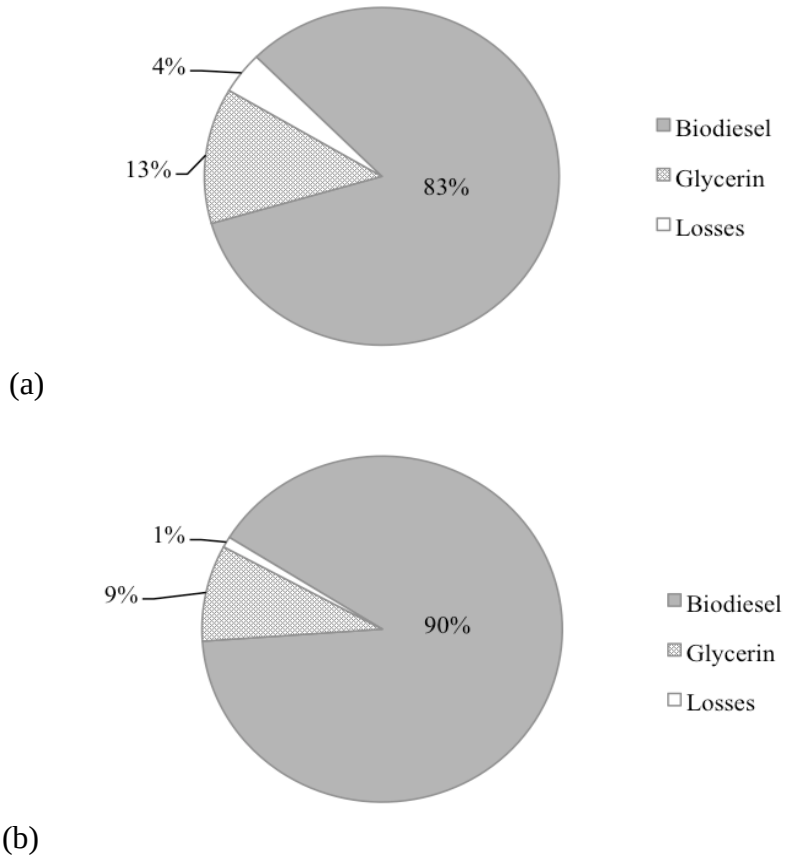


**Fig. 1. Mass and energy balance of transesterification process (a) mass distribution and (b) energy distribution.**

**Table 1. Gross heating value of crude oil, by-products, and refined oil.**

| Product | HV (MJ/kg) |
|---|---|
| Jatropha crude oil | 37.6 ± 0.5 |
| Gum | 11.7 ± 0.9 |
| Soap | 5.2 ± 0.6 |
| Wax | 20.2 ± 0.1 |
| Refined oil | 39.0 ± 0.6 |

**Table 2. ASTM characterization of fuels.**

| Property | Method | Specification | Jatropha Crude Oil | Jatropha Biodiesel |
|---|---|---|---|---|
| Flash Point, $^{o}$C | D 93 | 130 min. | > 210 | 192 |
| Kinematic Viscosity, 40 $^{o}$C, cSt | D 445 | 1.9-6.0 | 37 | 7.8 |
| Cloud Point, $^{o}$C | D 2500 | Report | 6 | 6 |
| Pour Point, $^{o}$C | D 2500 | Report | -4 | -2 |
| Acid Number, mgKOH/g | D 664 | 0.5 max. | 0.17 | 0.13 |
| Gross Heating value, MJ/kg | D 4809 | Report | 37.6 | 39.5 |

### *3.5 Mass and Energy Distribution*

Mass and energy balance for a refining process were examined based on the weights of initial crude oil and products obtained at each step, and their heating values. The mass and energy distribution for a refining process are provided in Figure 2 and Figure 3. Approximately 75% of mass conversion efficiency and 78% of energy recovery of refined oil were obtained with 2% mass losses and 14% energy losses. The percent energy losses was much higher than the percent mass losses; this can be explained by the gross calorific value of Jatropha crude oil, which was a lot higher than the energy value of the by-products obtained from refining process.

The refined oil was then converted into biodiesel via a transesterification process. Figure 4 and 5 show the mass and energy distribution for the completed process including the refining process and biodiesel conversion process. As mentioned before, the mass and energy conversion efficiencies of Jatropha biodiesel in the transesterification process were obtained at 83% and 90%, respectively (Section 3.4). By considering the whole process (refining and transesterification processes), the conversion of Jatropha crude oil into biodiesel would yield up to 63% of mass conversion efficiency and 70% of energy in biodiesel.

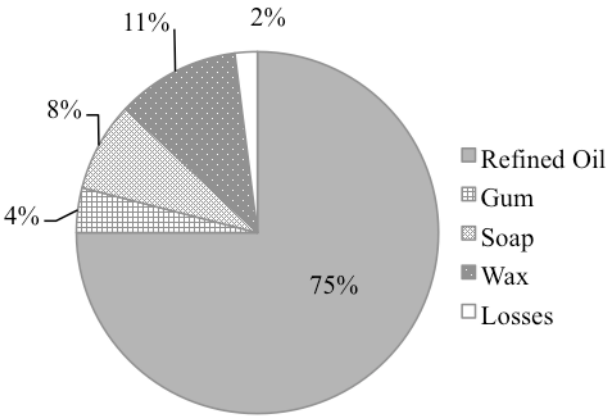


**Fig. 2. Mass balance for a refining process.**

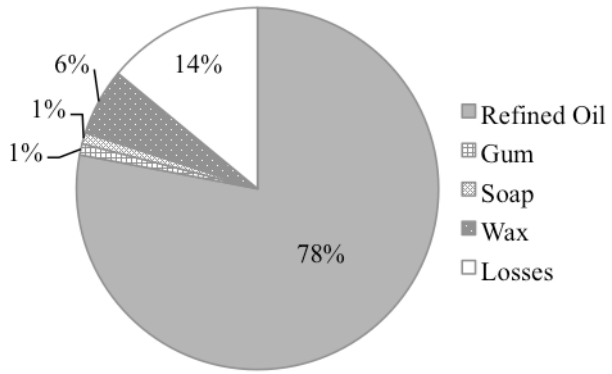


**Fig. 3. Energy balance for a refining process.**

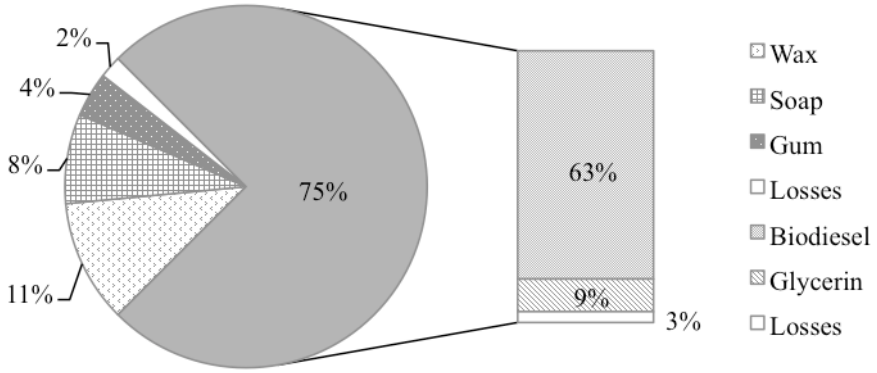


**Fig. 4. Mass balance for the complete process.**

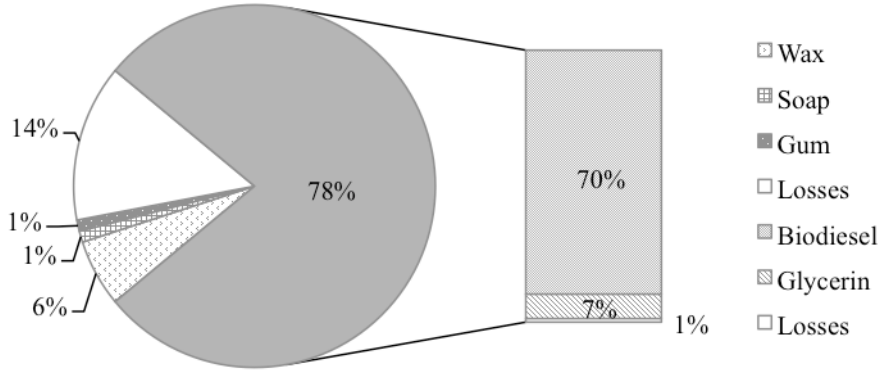


**Fig. 5. Energy distribution for the complete process.**

## 4. CONCLUSION

In this study, the Jatropha oil refining process and transesterification process of refined oil into biodiesel were successfully conducted and yielded a mass conversion efficiency of 75% and energy conversion efficiency of 78%. The higher energy conversion efficiency is a result of higher heating value of the final refined oil product. It was found that the quality of the Jatropha oil was improved after the refining process. The properties of Jatropha biodiesel were determined according to the ASTM standards and the results showed that the Jatropha biodiesel had met the ASTM standard of fuel except for the kinematic viscosity which was slightly elevated. Future research and development can be done on the improvement of Jatropha biodiesel in order to meet the standard specification of the fuel.

The transesterification mass and energy conversion efficiencies were found to be 83% and 90%, respectively. Likewise, this is a consequence of improved heating value of the final biodiesel product. The combined mass and energy conversion efficiencies for both the refining and transesterification process yielded overall efficiencies of 63% and 70%, respectively. Biodiesel producers will have to be aware of mass and energy losses at each step of the process and there should be improvements in the heating value of the product at each step even if mass losses are encountered.

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## NOMENCLATURE

| | |
|---|---|
| ASTM | American Society for Testing and Materials |
| BETA Lab | Bio-Energy Testing and Analysis Laboratory |
| KOH | Potassium Hydroxide |
| NaOH | Sodium Hydroxide |

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