The effect of Temperature, Residence Time and Particle Size on Extraction of Hevea Brasiliensis using n-Hexane as Solvent

by

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Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

JANUARY 2010

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CERTIFICATION OF APPROVAL

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Approved by,

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TRONOH, PERAK

JANUARY 2010

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MOHD SHAFIQ SUPENAH

ABSTRACT

Biodiesel is an alternative fuel produced from renewable vegetable oils, animal fats or recycled cooking oils by transesterification reaction. Biodiesel has drawn significant attention due to increasing environmental concern and diminishing petroleum reserves. However, using edible oils to produce biodiesel is not encouraged because it is competing resources with the food industry. Therefore, a possible solution to this issue is to produce biodiesel from non-edible oils such as Hevea Brasiliensis seeds. Production of Hevea Brasiliensis involve cultivation, oil extraction and transesterification. In conventional oil extraction, mechanical pressing is used. But, the residual oil left in the oil cake may be anywhere from 6% to 14% by mechanical pressing. The solvent extraction method recovers almost all the oils behind only 0.5% to 0.7% residual oil in the raw material. Due to the increasing demand and investment on Hevea Brasiliensis production. Thus, solvent extraction seems to be more economical for large scale production. N-hexane and water mixtures will be the solvent for the extraction. Using nhexane will give advantages since it can cut down the processing cost. However, due to the limited data on solvent extraction, the thermodynamics properties such as solidliquid equilibrium data, distribution coefficient, and enthalpy change and entropy change is studied. Therefore, this study will supports the production of biodiesel from Hevea Brasiliensis oil by using solvent extraction with n-hexane as the solvent.

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CHAPTER 1: INTRODUCTION

1.1 Background

Biodiesel is an alternative diesel fuel derived from the reaction of vegetable oils or lipids and alcohol with or without the presence of a catalyst. Despite the invention of the vegetable oil fuelled engine by Rudolf Diesel dated back in the 1900s, full exploration of vegetable oil based fuel such as biodiesel only came into light in the 1980s as a result of renewed interest in renewable energy sources for reducing greenhouse gas (GHG) emissions, and alleviating the depletion of fossil fuel reserves [1]. As a future prospective fuel, biodiesel has to compete economically with petroleum diesel fuels. One way of reducing the biodiesel production costs is to use the less expensive feedstock containing fatty acids such as inedible oils, animal fats, waste food oil and byproducts of the refining vegetables oils [2].

Conventional method for the production of biodiesel from Jatropha and other types of oil seeds involve cultivation, oil extraction, and esterification/transesterification. The requirement of these multiple processing stages constitute over 70% of the total production cost of biodiesel if refined oil is used as feedstock [3]. Oilseeds are extracted in two primary ways, by mechanical pressing, or squeezing, and with petroleum solvents. Prior to the 1940's, mechanical pressing was the primary method used. Mechanical extraction, however, had its limits in terms of oil recovery. Because pressing generates heat and high temperatures damage both the oil and meal, an oil content of the presscake below 5-6% was difficult to achieve. Solvent extraction was developed because it allows a more complete extraction at lower temperatures [4].

Chemically, biodiesel is a mixture of methyl esters with long-chain fatty acids and is typically made from nontoxic, biological resources such as vegetable oils [5], animal fats [6,7,8,9], or even used cooking oils (UFO) [10]. Vegetable oils are promising feedstocks for biodiesel production since they are renewable in nature, and can be produced on a large scale and environmentally friendly [11]. Vegetable oils include edible and non-edible oils. More than 95% of biodiesel production feedstocks comes from edible oils since they are

mainly produced in many regions and the properties of biodiesel produced from these oils are much suitable to be used as diesel fuel substitute [12]. However, it may cause some problems such as the competition with the edible oil market, which increases both the cost of edible oils and biodiesel [13]. Moreover, it will cause deforestation in some countries because more and more forests have been felled for plantation purposes. In order to overcome these disadvantages, many researchers are interested in non-edible oils which are not suitable for human consumption because of the presence of some toxic components in the oils. Furthermore, non edible oil crops can be grown in waste lands that are not suitable for food crops and the cost of cultivation is much lower because these crops can still sustain reasonably high yield without intensive care [14,12]. However, most non-edible oils contain high free fatty acids. Thus they may require multiple chemical steps or alternate approaches to produce biodiesel, which will increase the production cost, and may lower the ester yield of biodiesel below the standards [15,11,16]. Animal fats contain higher saturated fatty acids and normally exist in solid form at room temperature that may cause problems in the production process. Its cost is also higher than vegetable oils [17]. UFO is not suitable for human consumption but is a feedstock for biodiesel production. Its usage significantly reduces the cost of biodiesel production. However, the quality of UFO may cause concern because its physical and chemical properties depend on the contents of fresh cooking oil and UFO may contain lots of undesired impurity, such as water, free fatty acids [17,12,28,29]. Since the cost of raw materials accounts about 60–80% of the total cost of biodiesel production, choosing a right feedstock is very important [17,12]. Refer to figure 1.1 below to show the cost breakdown for production of biodiesel.



Figure 1.1 : General cost breakdown for production of biodiesel [17]

Since early 1980s, Malaysian government had realized the importance of developing biofuel and in particular, biodiesel in the long term. Being the world's largest producer and exporter of palm oil, it was imperative that Malaysia emerged as one of the pioneers in the palm biodiesel industry. This was achieved through the aggressive stance by Malaysian Palm Oil Board (MPOB) when the project of developing palm biodiesel was initiated at laboratory scale in 1982 [20].

In Malaysia, biodiesel production is synonymous to palm oil. To date, all of the established biodiesel production chains in Malaysia are using palm oil as primary feedstock. As a matter of fact, the thriving plantation of palm oil is the main factor which drives Malaysia towards developing biodiesel production and technology. Therefore, the availability and accessibility of palm oil supply are crucial in determining the potential growth of biodiesel production in Malaysia. Comparing to palm oil biodiesel industry, biodiesel produced from rubber seed is still in its nascent state in Malaysia even though considerable interest has been shown lately by both the government and private sectors. The potential of using rubber seed as feedstock for biodiesel production has received much attention worldwide [21,22] and even acknowledged by Goldman Sachs, U.S. leading investment banking and securities firm [23].

1.2 Objectives

Due to less attention being paid in solvent extraction process in extracting oil from seeds, limited data are reported on solvent extraction of Hevea Brasiliensis seeds. Therefore, it has become the main objective of the research to study the effect manipulating variable of oil extraction from Hevea Brasiliensis using n-hexane as solvent. The objectives are stated below:

- To determine the effect of temperature, size of particle and residence time on oil extraction.
- To determine the efficiency of oil extraction using batch extraction.

1.3 Scope of the research

Hevea Brasiliensis seeds or known as rubber seeds are chosen in the research. The seed kernel will be undergone various experiments to determine the total oil content (batch extraction) and moisture content(standard method). As stated in the objective, the variable parameters of the oil extraction are size of particles, temperature and residence time.

1.4 Significance of the research

Rubber seeds contain considerable amount of oil that can be extracted. The nonedible oil remains underutilized being side products of rubber plantation. The seeds are abundant over the wide rubber plantation in Malaysia and the oil can be converted to alkyl esters which can be used as biodiesel. However, the industry and the technology for biodiesel production from that feedstock are not well established. Rubber seed oil, typical non-edible high FFA oil is considered as the potential feedstock for biodiesel production in this study. The solvent extraction method recovers almost all the oils and leaves behind only 0.5% to 0.7% residual oil in the raw material. By using n-hexane as the solvent, solvent extraction can be more economical ways compare to mechanical extraction being used nowadays as n-hexane gives advantages since it can be obtained from renewable, and agricultural cultivated sources.

CHAPTER 2: LITERATURE REVIEW

2.1 Characterization of rubber seed oil

The rubber tree (*Hevea brasiliensis*) is widely used as a source of natural rubber, and its seeds have been found to be rich in oil. At the present time, the production of rubber seed oil (RSO) shows a huge increase in both quantity and quality in Malaysia [23]. Rubber trees yield 3-seeded ellipsoidal capsule, each carpel with one seed. Rubber seeds are ellipsoidal, variable in size, 2.5-3 cm long, mottled brown, lustrous, weighing 2–4 g each. Capsules are spread over the ground. These are collected and kernels are separated by breaking 2the capsules [20]. Although there are variations in the oil content of the seed from different countries, the average oil yield have been reported to be 40% [18]. Rubber seed oil is yellow, semi-drying oil [24]. The oil does not contain any unusual fatty acids, and its rich source of essential fatty acids $C_{18:2}$ and $C_{18:3}$ that make up 52 % of its total fatty acid composition [25]. The fatty acid composition and the important properties of rubber seed oil in comparison with other oils is given in table 2.1.

Table 2.1 : Properties of	f rubber seed oil ir	comparison	with the oth	ner oils	s[20	ן
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Property Fatty acid composition (%)	Rubber seed	Sunflower	Rapeseed	Cotton seed	Soybean
(i) Palmitic acid C _{16:0}	10.2	6.8	3.49	11.67	11.75
(ii) Stearic acid C _{18:0}	8.7	3.26	0.85	0.89	3.15
(iii) Oleic acid C _{18:1}	24.6	16.93	64.4	13.27	23.26
(iv) Linoleic acid C _{18:2}	39.6	73.73	22.3	57.51	55.53
(v) Linolenic acid C _{18:3}	16.3	0	8.23	0	6.31
Specific gravity	0.91	0.918	0.914	0.912	0.92
Viscosity (mm2/s) at 40°C	66.2	58	39.5	50	65
Flash point (°C)	198	220	280	210	230
Calorific value (MJ/kg)	37.5	39.5	37.6	39.6	39.6
Acid value	34	0.15	1.14	0.11	0.2

Cetane number (CN) is widely used as diesel fuel quality parameter related to the ignition delay time and combustion quality. Higher the cetane number is, better it is in its ignition properties [6]. An adequate cetane number is required for good engine performance. High cetane numbers help ensure good cold start properties and minimize the formation of white smoke [31]. It is well known that biodiesel cetane number depends on the feedstock used for its production. The longer the fatty acid carbon chains and the more saturated the molecules, the higher the cetane number [9]. High cetane numbers were observed for esters of saturated fatty acids such as palmitic ($C_{16:0}$) and stearic ($C_{18:0}$) acids [32]. The presence of these monounsaturated compounds gave a high cetane number to the biodiesels. Those which were richer in unsaturated ester of linoleic acid ($C_{18:2}$), presents a cetane number in the medium range [9].

There are many uses of the rubber seed oil, such as its uses in the manufacturing of laundry soap, paints, varnishes, deoxidized oil. The RSO is suitable for the production of fat liquor for the tanning of leather industry and in the preparation grease. The rubber seed oil is used for the production factice and epoxidized. RSO is used in formulations for anticorrosive coatings and adhesives [24]. The rubber seed oil is used as a plasticizer and the fatty acid component of the activator in natural rubber and styrene butadiene rubber [26].

2.2 Vegetable Oil Extraction

Oil is extracted from seeds, beans, and nuts for use as cooking or salad oil; as an ingredient in paint, cosmetics, and soap; and even as fuel. There are several methods to extract vegetable oils from oil bearing seeds. Of which, mechanical and solvent techniques are the most commonly used. Historically, such oils have been extracted by wrapping seeds in cloth, and then using devices operated by stones and levers to exert pressure on them. Because most press or expeller processes overheat the meal and leave too much of the high value oil in the seed cakes, methods of extracting the oil with solvents were developed. Seeds (like soybeans) with low oil content are processed by solvent methods [30]. Each one has its own advantages and disadvantages. In mechanical extraction, the capital involved is less than that for solvent extraction since it requires less machinery, material and operation cost. Mechanical extraction involves pressing of the seeds to obtain the oil [54]. Solvent extraction of vegetable oils, which recovers more oil than earlier methods and leaves more usable meal, begins to be economically attractive where large quantities of seed can be processed (at least 200 tons per day for continuous-feed processes [30].

The solvent extraction method recovers almost all the oils and leaves behind only 0.5% to 0.7% residual oil in the raw material. In the case of mechanical pressing the residual oil left in the oil cake may be anywhere from 6% to 14%. Because of the high percentage of recovered oil, solvent extraction has become the most popular method of extraction of oils and fats. In practice, both techniques are employed in a combined method and used by the industries dealing with vegetable oil extraction. Like pressing, solvent extraction can be done with equipment that processes the oilseed in batches, or with equipment that processes it continuously. A continuous extractor is not considered economically practical unless it processes at least 200 tons per day [30].

Commercial grade hexane had been used for long time as the solvent of choice since it's relatively less toxic and flammable and can be easily recovered when compared with other types of solvents [55]. Researchers investigated the kinetics of solvent extraction using various types of alcohols such as n-hexane and iso-propanol. Hydrocarbon solvents like n-hexane and Iso-hexane were also investigated. Aqueous acid solutions were found to give better results when used in conjunction with solvents. Sulfuric acid, hydrochloric acid and phosphoric acid are examples of these acids that can be used [56].

2.3 Leaching

Extraction is a term used for an operation in which a constituent of a liquid is transferred to another liquid (solvent). The term solid-liquid extraction is restricted to those situations in which a solid phase is present and includes those operations frequently referred to as leaching, lixiviation and washing [40]. The principle for the solid-liquid extraction is that the soluble compounds of a solid matter, existing of an inert matrix and the active agent, are extracted by a solvent. The extract can be included in the extraction matter in solid or liquid form. It can be included in cells like oil in oil seeds or as fine dispersion on the solid matter like caffeine in coffee [41]. Leaching process can be considered in three parts which are diffusion of the solvent through the pores of the solid, the diffused solvent dissolves the solutes (i.e. transfer the solute to the liquid phase), and transfer of the solution from porous solid to the main bulk of the solution.

Solid-liquid extraction is carried out in single and multiple stages. A stage is an item of equipment in which the solid and liquid phases are brought into contact, maintained in contact for a period of time and then physically separated from each other. During the contact, mass transfer of components between the phases takes place and they approach a state of equilibrium.

Two different methods of leaching will be approached in the studies which are leaching by using Soxhlet extractor and by using conical flask which will be dipped into water bath. In leaching by the soxhlet apparatus multiple contacts of solids with the fresh solvent is performed at each stage of operation. This is equivalent to multiple cocurrent system, as described in the following diagram in figure 2.1.



Figure 2.1 : Multiple co-current system

To achieve the fastest and most complete solid extraction possible, the solvent must be provided with large exchange surfaces and short diffusion paths. This can be done by pulverizing the solid to be extracted. However, an excessively small grain size can cause agglutination and make it more difficult for the solvent to permeate. In considering factors that affect extraction, different parameters will be set in conducting the batch extraction experiment (by using conical flask). They are the particle size of the milled seed kernel, the temperature, the length of the experiment (which is extraction), and the solvent-to-solid ratio.

2.4 The effect of temperature

In Figure 2.2 as shown below the data for the extraction of soybean flakes with commercial hexane at three temperatures are plotted to show the effect of temperature on the extraction rate curves obtained. From the resulting curve families, cross plots were made showing the relation of temperature and "Time to 1%" (5, 10), defined as the extraction time in minutes required to reduce the oil seed to a dry-basis oil content of 1.0%. This is a criterion of extraction rate.



Figure 2.2 : Rate of extraction of soybean flakes at three temperatures.

In the literature, explanations for the effect of temperature on extraction are based on the viscosity of the solvent and the solubility of some of the components of the crude oil (3,8).

2.5 The effect of particle size

Figure 2.3 below presents extraction data for flakes of various thicknesses made from soybeans, cottonseed, peanuts, and flaxseed. The data for each lot of material are recorded in a single series, in which the only variable is flake thickness. This is a log-log plot of percentage of residual oil content, on a moisture-free basis, against extraction time in minutes (10). The curves approximate straight lines and can be easily interpolated and extrapolated.



Figure 2.3 : Relation of extraction time and residual oil.

The time to reach a residual oil of 1.0% is taken as an index of extraction rate. This time, termed "time to 1%," will be used frequently in the discussion of extraction data. It will be noted that the time to 1% increases in a regular manner with increased flake thickness although the degree varies for different oil seeds.

CHAPTER 3: METHODOLOGY

3.1 Materials

3.1.1 Hevea Brasiliensis seeds

The seeds of rubber tree are ellipsoidal shapes with varying brown shells covering kernels. They are also varies in sizes. Rubber seeds contain considerable amount of oil that can be extracted. Rubber seeds are only utilized to regrow a new plant of rubber tree. Refer to figure 3.1 and figure 3.2 for picture.



Figure 3.1 : Rubber seeds



Figure 3.2 : Schematic diagram of rubber seed

3.1.2 Anhydrous N-hexane

Its aqueous solution with different water content will be needed.

3.2 Analytical Methods

3.2.1 Hevea Brasiliensis seeds

3.2.1.1 Rubber seed processing

Physical properties of rubber seeds that have been collected should be taken such as the weight and its length and the average should be recorded as rubber seeds are varies in sizes. Some of the rubber seeds then will be processed by taken off its shell and obtain the kernel. The kernel physical properties also should be recorded. Several batches of kernel will be dried in an oven at 105°C for 30 minutes and is done until the weight is constant. Next, the kernel should be crushed into smaller pieces and being stored in an air tight container to preserve its properties. The kernel will be crushed into several sizes which are 250 μ , 500 μ , 1000 μ , and 2000 μ -4000 μ .

3.2.1.2 Moisture Content

The moisture content is determined by establishing the loss in the weight of the sample when heated under rigidly controlled conditions of temperature, time and atmosphere, sample weight, and equipment specification. The percentage of moisture in the analysis sample is calculated as follows:

Percentage of moisture = $\underline{A} - \underline{B} \times 100$ B - C

Where, A: mass of container and wet sample, g;

B: mass of container and dry sample, g;

C: mass of container, g.

3.2.1.3 Leaching

The batch solid-liquid extraction will be conducted by using conical flask immersed in water bath. The 200ml conical flask will be used. There will be four different parameters that will be set in order to obtain the data from the leaching experiment. They are the particle size of the milled seed kernel, the temperature, the time of extraction, and lastly the solvent-to-solid ratio. 50 ml of the solvent will be used for each experiment and parameters while the solvent chosen is n-hexane. The data obtained from this experiment will be compared to the commercially grade solvent, n-hexane that undergo the experiment with the same parameters.

In the first stage of the experiment, by fixing the temperature at 25°C, the extraction will be conducted by using different particles size of the milled seed kernel in one hour time of experiment. The seed kernels that are milled earlier available in four different size which are in 4-2mm, 1mm, 0.5mm, and 0.25mm diameter. As mentioned earlier, the extraction is conducted in 25°C, therefore, in the next stage; the experiment will be conducted by regulating the temperature of the water bath. The suggested temperatures for the leaching are 25°C, 30°C, 35°C, and 40°C. It is understood that the solubility of the material which is being extracted will increase with temperature to give a higher rate of extraction.

The other parameter which will be regulated in the leaching process is the length of the experiment. The experiment to be conducted in one hour, six hours, 12hours, and 24 hours of time.

After finished with extraction, the kernel will be filtrated by using vacuum filtrator to separate between the solid and the solvent. Next, the solvent obtained will be evaporated by using rotary evaporator to separate between the oil and the n-hexane at around 79°C which is higher than the boiling point of n-hexane. Meanwhile, the n-hexane can be recovered by condensation which can be done on the same equipment. Should the oil obtained from this experiment is more than the Soxhlet extracted, the solvent will be undergone the evaporation once again to remove moisture that maybe present.



Figure 4.7 : Oil extracted at different temperature at different particle size



4.2 Temperature Effect



At particle size 2-4mm, it shows the oil extracted increase as the temperature increase. Theoretically, as the temperature increase the kinetic rate increase thus contribute to a faster reaction to extract more oil. But, it shows significantly drop at temperature 55°C due to some limitation by the solvent properties.

4.3 Time Effect



Figure 4.9 : Oil extracted at 40C at 2-4 mm particle size at different residence time

The residence time did affect oil extraction in such a way, the longer the time the higher amount of oil extracted. As show in figure 4.9, the oil extracted most at 6 hr. This happen because the longer the residence time the longer the seed being contact with the solvent.

Particle			Oil
Size	Temperature	Time	percent
2-4	40	3	42
1.00	40	3	40.2
0.50	40	3	80.2
0.25	40	3	82

Table 4.3 : Batch extraction at 40°C for 3hr at different particle size



Figure 4.3 : Oil extracted at 40°C at different particle size

Overall, the result trend shown similar as other temperature. At temperature 40 °C, it shows slightly drop in oil percent from particle size 2-4 mm to 1 mm as shown in table 4.3 and figure 4.3. The drop is because the oil sample mixed with water, thus, it gives higher oil percent at 2-4 mm. Practically, the mixing occur because the ratour vapour sample bowl hard to dry up due to the small physical appearance.

Particle			Oil
Size	Temperature	Time	percent
2-4	45	3	47.6
1.00	45	3	72.4
0.50	45	3	69.2
0.25	45	3	82.6

Table 4.4 : Batch extraction at 45°C for 3hr at different particle size



Figure 4.4 : Oil extracted at 45°C at different particle size

Refer to table 4.4 and figure 4.4, the data at 45°C shows tremendous increment as compare to previous temperature. The error clearly indicate at particle size 1 mm. The exact trend of oil percent should be increase as the particle size getting smaller.

Particle Size	Temperature	Time	Oil percent
2-4	50	3	50.2
1.00	50	3	73
0.50	50	3	79.6
0.25	50	3	84.6

Table 4.5 : Batch extraction at 50°C for 3hr at different particle size



Figure 4.5 : Oil extracted at 50°C at different particle size

The oil extracted by using n-hexane best at temperature 50°C as show in table 4.5 and figure 4.5. The amount of oil produced is higher as compare to other oil produced at different temperature. The same experiment has been done by using different solvent which is ethanol. The optimum temperature at 45°C. Thus, solvent did really affect the best operating temperature to obtain best result.

Particle Size	Temperature	Time	Oil percent
2-4	55	3	44.2
1.00	55	3	70.2
0.50	55	3	74.2
0.25	55	3	83.2

Table 4.6 : Batch extraction at 55°C for 3hr at different particle size



Figure 4.6 : Oil extracted at 55°C at different particle size

As a conclusion for the viewed data, it shows the batch extraction of rubber seed it suitable to be conducted at temperature 50 C. However, there is an error occur to some of the data due to the water contaminated. Figure 4.7 shows final graph that comparing experiment on different particle size at different temperature.



Figure 4.7 : Oil extracted at different temperature at different particle size



4.2 Temperature Effect

Figure 4.8 : Oil extracted at different temperature at 2-4 mm particle size

At particle size 2-4mm, it shows the oil extracted increase as the temperature increase. Theoretically, as the temperature increase the kinetic rate increase thus contribute to a faster reaction to extract more oil. But, it shows significantly drop at temperature 55°C due to some limitation by the solvent properties.

4.3 Time Effect



Figure 4.9 : Oil extracted at 40C at 2-4 mm particle size at different residence time

The residence time did affect oil extraction in such a way, the longer the time the higher amount of oil extracted. As show in figure 4.9, the oil extracted most at 6 hr. This happen because the longer the residence time the longer the seed being contact with the solvent.

CHAPTER 5 CONCLUSIONS & RECOMMENDATIONS

5.1 Conclusion

There are numerous properties that has to be identify in order to extract rubber oil for large scale production. The effect of temperature, residence time and particle size are part of the properties that contribute to produce rubber oil as an alternative biodiesel.

The temperature did affect oil extracted from rubber seed. The oil percent increase as the temperature increase. This is because higher kinetic rate contribute higher chemical reaction thus more oil can be extracted. However, the amount of oil extracted is decrease significantly at temperature 55°C. This is because the solvent has its optimum condition that need to be operated.

The residence time plays an important effect in oil extraction. The oil extracted seems to be higher as the residence time increase. The long time more time for the seed for being contacted with solvent.

The oil extracted most at small particle size. Small particle size has a large surface area. Thus, more area can be contacted with the solvent. For future large scale production are advised to use small particle size to produce more oil to be contacted.

5.2 Recommendation

For future improvement, further analysis should be done on other properties such as thermodynamic properties. By combining both the effect and thermodynamic properties in the studies, it will help in developing a large scale rubber oil production for biodiesel as a future energy source. Finally, an environmentally energy source use can be developed after all the factors have been taken into consideration.

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