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BEAUTY LEAF OIL REFINING AND CONVERSION INTO BIODIESEL

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Abstract*- The treating of crude oil is indispensable prior to biodiesel production because it can yield a high amount of triglycerides and to produce an acceptable quality of refined oil. Refining process was conducted on crude oil extracted from solvent extraction technique using n-hexane. Five replications each of 25 gms of crude oil were selected for refining as well as biodiesel conversion processes. The refining processes such as, degumming, neutralization as well as dewaxing processes were performed to remove all the gums (phospholipid compounds), free fatty acids and waxes from the crude oil, respectively, before carrying out the biodiesel production process. The results indicated that up to 81% of mass conversion efficiency was obtained from the refined oil in the refining process. It was also found that up to 90% of biodiesel and 8% of glycerin were produced in the transesterification process. While the entire processes (refining and transesterification) were considered, the conversion of beauty leaf tree (BLT) into biodiesel yielded up to 73% of mass conversion efficiency. The physico-chemical properties of refined oil as well as biodiesel were characterized according to the ASTM standards.*

Keywords: BLT crude oil, oil refining, transesterification, biodiesel, mass balance

1. INTRODUCTION

Vegetable oils are considered as one of the promising feedstock for biofuel production especially from non-edible oil feedstock because of their high energy, lower sulfur and aromatic content. These fuels are renewable, biodegradable, technically feasible, economically competitive, environmental friendly, non-toxic, portable, eco-friendly and easily available. Among various non-edible feedstocks, BLT (*Callophylum inophyllum*) is regarded as a prospective source of energy to produce biodiesel from the 2nd generation feedstock due to its potentiality as well as high oil yield content in the seed kernels. It can be grown on degraded land in Australia [1], in a variety of climatic conditions, easily cultivated and its fruit production rate is high as well as its seed kernels contain high oil yield. Furthermore, the BLT tree grows in free draining less fertile soils near the shorelines and it is grown in different clay soils both in Australia and different parts of southern and central Asia such as Sri Lanka and India [2]. BLT fruits are produced abundantly twice a year and seed kernels contain up to 65% of non-edible oil [3, 4] and higher than most of the general oil seed harvests; *Jatropha curcas*- 40%, *Pongamia pinnata* – 30%, oil palm – 60% [5]. BLT is native to Australia and is non-incursive; it does not contest with associate tress for nutrients [6].

Biodiesel is produced from vegetable oils via a transesterification technique which is the most popular technology [7, 8]. Transesterification is the most common method for biodiesel production due to its low cost and simplicity [9-14], and thereby, this method has been widely used to convert vegetable oil into biodiesel. As the BLT oil consists of large amount of free fatty acids (FFAs), these FFAs can be transesterified to alkyl ester in the presence of alcohol [15]. Base or alkaline catalyzed transesterification is the most general technique to produce biodiesel nowadays. Common alkaline catalysts are sodium hydroxide (NaOH), potassium hydroxide (KOH) and sodium methoxide ($NaOCH_3$) [16].

Therefore, the aim of this study is to accomplish a complete refining process of the BLT crude oil using the chemical technique and conducting the transesterification process for the production of biodiesel and conversion efficiency of the processes. The American Society for Testing Materials (ASTM) standards was used to characterize the physico-chemical properties of the resulting biodiesel.

2. MATERIALS AND METHOD

2.1 BLT Oil Extractions

Chemical (solvent) extraction using n-hexane as a solvent was used for oil extraction from the prepared seed kernels. Properly dried and treated BLT kernel samples were used to extract oil by this method.

In solvent extraction, the kernels were ground using a blender and coffee grinder machine to achieve a

consistency (of a finer size) for maximising particle surface area. Then n-hexane was added at a ratio of 2:1 (ml hexane: grams kernel) into conical flasks in which ground kernels were put. The kernel and n-hexane mixture samples were left to run for at least 16 hours in this condition. Then the oil mixtures were collected, filtered and decanted into aluminium foil containers for evaporating the solvent under a fume hood.

2.2. Oil Refining Processes

Vegetable oils must be properly pre-treated prior to the transesterification process for biodiesel production. Refining process undergoes a series of stages to remove undesirable constituents from the crude oil. Each replication of BLT oil was treated properly. Oil refining processes consists of degumming, neutralization (deacidification) as well as dewaxing. The degumming process removes unwanted phosphorous content, whereas neutralization and dewaxing processes removes free fatty acids (FFAs) and waxes, respectively.

2.2.1. *Degumming*

The purpose of the degumming process was to remove the gums i.e., phosphorous-based compounds, primarily lecithin and cephalin from the fresh crude oil before converting it into biodiesel. Firstly, the filtered oil was heated up to 60^0C for about 2 hours and then the heated (60^0C) deionized water (DI) was added to each replication of oil. Both acid and water degumming were conducted for crude oil. Acid degumming was performed by adding 4% (wt.) H_3PO_4 solution. The solution was heated up to 65^0C and left for 30 minutes. After centrifugation all the hydrated gums and the oil were separated, weighted and the results were recorded. The detail of the degumming process is given in Table 1.

2.2.2. *Neutralization*

The neutralization process was conducted to remove the FFAs before biodiesel production. Accordingly the sodium hydroxide (NaOH) aqueous solution (8% wt.) was mixed with the degummed oil of each replication. The solution was mixed slowly 30 minutes to allow soap formation, and the mixture was separated by centrifugation. After that the oil was washed twice with 35^0C warm tap water to make sure that all traces of soap were eliminated. Finally, the oil, gum and soap solution were separated by centrifugation. The amount of oil as well as recovered gum and soap were recorded. The detail of the neutralization process is shown in Table 1.

2.2.3. Dewaxing

The vegetable oil also contains large amount of waxes which form cloud when refrigerated or brought to lower temperatures. Therefore, the waxes have to be removed before biodiesel production. This was done through adding 5% (wt.) sodium hydroxide aqueous solution and 5% (wt.) de-ionised (DI) water, respectively, to the neutralize oil of each replication. The mixture was placed in a chiller at 5^0C and agitated for about 4 hours. The soapy water wetted the waxes and helped to change from oil to water phase. Finally, the oil, wax and soap were separated by centrifugation and their weights were recorded. The detail of the dewaxing process is shown in Table 1.

3. BIODIESEL CONVERSION THROUGH TRANSESTERIFICATION PROCESS

The BLT refined oil was converted into biodiesel via transesterification process. To carry on the transesterification process sodium methoxide solution was prepared first by adding NaOH solution (1% wt.) and methanol (11.12% wt.) in a covered conical flask. The transesterification process was then conducted by adding sodium methoxide solution into the heated refined oil. The reaction temperature was maintained less than 65^0C and allowed for half an hour. After completing the reaction, the mixture was poured in the separatory funnel to separate the biodiesel and glycerin. Finally, the glycerin was removed and the biodiesel was washed with warm DI before further processing.

3.1 Characterization of BLT Oil and Biodiesel

Physico-chemical properties of BLT crude and refining oil were determined. The properties of all 5 replications of oil sample were characterized according to the ASTM standards. Physico-chemical properties of BLT crude and refined oil are shown in Table 2.

Biodiesel obtained through transesterification was characterized in accordance with ASTM standards. Physico-chemical properties of BLT biodiesel are also shown in Table 2.

3.2 Mass Balance

The mass balance for the refining oil and the biodiesel were determined in accordance with the oil input and the weights of the products. The mass balance for a refining and transesterification processes are shown in Figs. 1 and 2, respectively. The efficiency of BLT crude oil conversion into refined oil and then produce biodiesel and by-products (glycerin) namely "% Mass conversion" was estimated using equations 1 and 2, respectively.

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

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

Where, W_{prod} is the weight of products, kg

$W_{crude\ oil}$ is the initial weight of BLT crude oil, kg

The mass balance for the refining process, transesterification process and the overall mass balance of the whole processes (refining and transesterification) are presented in Figs 1, 2 and 3, respectively.

Table 1: Oil Refining Processes

No. of Test Samples	Replication 1	Replication 2	Replication 3	Replication 4	Replication 5
Amount of test (oil) samples (gm)	25	25	25	25	25
Degumming Process	Acid Degumming	Acid Degumming	Acid Degumming	Acid Degumming	Water degumming
1. Heat up the oil sample to 60^0 C for 2 hours	√	√	√	√	√
2. Adding de-ionised (DI) heated (60^0C) water (4% wt.) (g)	1	1	1	1	1
3. Adding phosphoric (H_3PO4) acid solution (4% wt.) (g)	1	1	1	1	--------
4. Heat up the solution at about 65^0C for about 30 minutes.	√	√	√	√	√
5. Centrifugation if necessary to separate the gums and oil	√	√	√	√	√
Neutralization Process	Degummed oil	Degummed oil	Degummed oil	Degummed oil	Degummed oil
1. Add sodium hydroxide (NaOH) solution (8% wt.) (g)	2	2	2	2	2
2. Mix slowly and allow 30 minutes for soap formation	√	√	√	√	√
3. Wash with 35^0C warm tap water	√	√	√	√	√
4. Centrifugation to separate the mixture	√	√	√	√	√
5. Wash with 35^0C warm tap water	√	√	√	√	√
6. Centrifugation	√	√	√	√	√
7. Discard water (containing soap)	√	√	√	√	√
8. Separation of gum, soap and oil	√	√	√	√	√
9. Soap formation (g)	1.1	1.26	1.11	1.14	1.21
10. Gum formation (g)	2.23	2.31	2.21	2.19	2.14
Dewaxing process	Neutralized oil	Neutralized oil	Neutralized oil	Neutralized oil	Neutralized oil
1. Adding sodium hydroxide solution (5% wt.) (g)	1.25	1.25	1.25	1.25	1.25
2. Adding de-ionised (DI) water (5% wt.) and mix slowly (g)	1.25	1.25	1.25	1.25	1.25
3. Placing in a chiller at 5^0C for 4 hours	√	√	√	√	√
4. Centrifugation at 5^0C	√	√	√	√	√

5. Separation of waxes and oil	√	√	√	√	√
6. Wax formation (g)	0.83	1.07	0.79	0.89	1.02
Recovered soap (g)	1.1	1.26	1.11	1.14	1.21
Recovered gum (g)	2.23	2.31	2.21	2.19	2.14
Recovered wax (g)	0.83	1.07	0.79	0.89	1.02
Oil yielded (g)	20.46	20.32	20.13	20.93	19.21
% of oil recovery	81.84	81.28	80.52	83.72	76.84
Average % of oil recovery	80.84				

Table 2: Physico-chemical properties of BLT crude and refined oil, and biodiesel.

Method	Oil type	Property	Replication No./Sample No.				
			1	2	3	4	5
n-hexane	Crude	Density@ 28^0C (g/mL)	0.9011	0.8969	0.9012	0.9036	0.9057
		Viscosity@ 40^0C (poise)	0.5372	0.5426	0.5339	0.5412	0.5333
		Acid value (mg KOH/g oil)	32.65	32.87	32.71	32.58	31.96
		pH value	4.82	4.65	4.46	4.58	4.49
n-hexane	Refined	Density@ 28^0C (g/mL)	0.8655	0.8716	0.8682	0.8728	0.8631
		Viscosity@ 40^0C (poise)	0.4054	0.4103	0.4278	0.4156	0.4034
		Acid value (mg KOH/g oil)	3.37	3.31	4.14	3.2	3.09
		pH value	7.63	7.99	7.31	7.24	7.38
n-hexane	Biodiesel	Density@ 28^0C (g/mL)	0.8571	0.8548	0.8514	0.8523	0.8483
		Viscosity@ 40^0C (poise)	0.0448	0.0441	0.0453	0.0445	0.0458
		Acid value (mg KOH/g oil)	0.45	0.56	0.51	0.63	0.43
		pH value	9.55	9.4	9.48	9.57	9.64

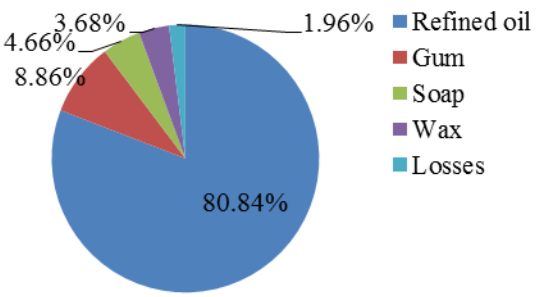

Fig. 1: Mass balance for a refining process

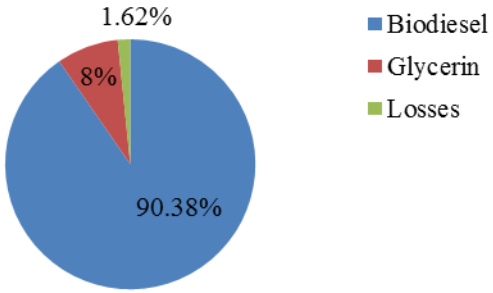

Fig. 2: Mass balance of transesterification process

4. RESULTS AND DISCUSSION

The BLT oil extracted from chemical method was refined through degumming, neutralization as well as dewaxing processes. The degumming process was conducted for both acid degumming and water degumming. About 81% of conversion efficiency was obtained through refining process including 8.86 % gum, 4.66% soap, 3.68% wax and 1.96% % other losses as shown in Fig. 1. Large amount of soap by-product in neutralization process indicated high FFA composition, which reduced the yield of biodiesel conversion [17].

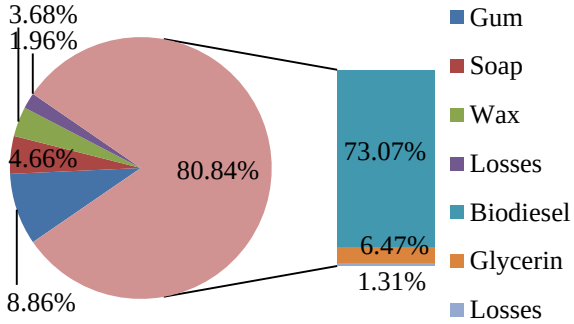

Fig. 3: Mass balance of the complete (refining and transesterification) processes

As shown in Fig. 2, the biodiesel conversion efficiency of 90 % was yielded in transesterification technique including 8% glycerin and 2% other losses. Moreover, 73% of conversion efficiency with 6.47% glycerin and 1.31% other losses were obtained while the entire processes (refining and transesterification) were considered as shown in Fig. 3. The ASTM characterization was performed to confirm that the biofuel would meet the specification of standard fuels.

5. CONCLUSION

In this study, BLT oil purifying process and then conversion of refined oil into biodiesel through transesterification method was successfully performed. The results indicated that after the treating process the good quality of the BLT oil was obtained. The properties of the BLT crude and refined oil, and BLT biodiesel were determined in accordance with the ASTM standards and the results showed that the BLT biodiesel has met the ASTM standards of fuel. Approximately 81% and 90% conversion efficiencies were obtained through refining and transesterification processes, respectively. The overall conversion efficiency of 73% was obtained while the entire process (refining and transesterification) was considered.

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