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Preheated extracted jatropha oil using exhaust heat recovery

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ABSTRACT

The purpose of this research is to study the influence of extraction methods (solvent, soxhlet, screw and hydraulic) on the properties of jatropha oil. This study was established to use the exhaust heat recovery to preheat the extracted oil. The free fatty acid, and the properties of jatropha oils extracted using various methods were determined. The oil yields are 19, 11, 25 and 20% for screw, hydraulic, Soxhlet and solvent extraction processes, respectively. Free fatty acids were 2.7, 5, 17.6 and 21.1% for the oils extracted using the screw press, hydraulic, solvent and soxhlet methods, respectively. Linoleic, oleic, palmitic, and stearic fatty are the major fatty acids in the extracted oils. Measured calorific values of jatropha oil extracted following soxhlet, solvent, screw and hydraulic processes methods were 39135, 38808, 39128, 39201 and 38789 kJ/kg, respectively. For jatropha oil extracted by soxhlet, solvent, screw, hydraulic, and biodiesel methods, the evaluated cetane numbers were 37.53, 37.62, 37.83, 39.12, and 42.62, respectively. Effect of oil preheating on density and viscosity was investigated. As the temperature of the oil increased, the density and viscosity of the oil decrease.

Nomenclature

ASTM	American Society for Testing and Materials
C 16:0	Total number of carbon atoms to the number
	of carbon-carbon double-bonds
FFA	Free Fatty Acid, %
GC	Gas chromatography
HC1	Hydrochloric acid
NaOH	Sodium hydroxide
ρ	oil density, kg/m ³
μ	oil dynamic viscosity, Cp

1. Introduction

The researchers have been directed to find a suitable alternative fuel due to fuel crisis and global warming. Jatropha tree was cultivated in sewage bonds and desert. Extraction of jatropha oil is performed chemically or by mechanical presses. Biodiesel has become environmental-friendly and more attractive because it is obtained from renewable resources [1]. Jatropha oil was extracted using a variety of oil expellers.

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Sundhara and Komet expeller types are used. Chemical, mechanical, and enzymatic methods were used to extract jatropha oil. The mechanical press prototype has cast iron heavy parts and iron sheets. The mechanical press is driven by electricity power source. The oil is collected from the seeds by hand utilizing traditional methods in rural areas. A single screw mechanical expeller was used as Komet expeller type. Chemical methods are used for oil extraction like aqueous enzymatic treatment. Ultra sonication has been used as an effective method to extract jatropha oil, resulting in yield of 74% [2-5].

Soxhlet method named by Franz Von Soxhlet author is used as a reference conventional extraction method and other methods are compared to Soxhlet. Most extraction methods are similar to Soxhlet that they require a long time and large number of solvents. Most extraction processes are simple in performance. These methods are economical, so they have been favored to be used widely in industries and laboratories [6-7]. Two mechanical processes followed by solvent processes can be used in oil extraction from seeds. Soybean presented lower oil content, but sunflower seeds, palm and rapeseed produced higher oil content [8]. Processing of jatropha curcas seeds for oil production consumes roughly about 24% of the total internal energy in solvent extraction, but mechanical extraction consumes 66%.Seed pretreatment diminishes the energy saving prior to oil extraction. Mechanical extraction is a more efficient process in terms of energy usage. The solvent production of Jatropha oil is 79 % while the mechanical extraction yields 96 % [9]. Jatropha oil is used to make biodiesel [8, 9]. Properties of jatropha oil are similar to those of normal diesel, including density, flash point, kinematic viscosity, pour point and cloud point. Jatropha oil can be used in a conventional diesel engine without any modifications due to its properties [9].

In rural locations, the ram press generated small amounts of oil. The oil was generated more successfully using the strainer press, which is operated manually. On a big scale, the cylinder press produces oil from Jatropha seeds with an oil yield of over 89.4 % [10]. The performance of a screw press for oil production has been investigated using various machine capacities and seed moisture content. The oil yield, extraction efficiency, specific energy and operational cost have been studied. The highest value of production rate and extraction efficiency was 43 kg/hr and 81%, respectively [11]. Jatropha curcas seeds are the best source of oil feedstock for biodiesel synthesis since they are both sustainable and low-cost [12]. The behavior of oil extraction is highly dependent on how the seeds are prepared. The presence of seed shells result in the formation of a porous solid. Oil extraction required mechanical energy which is less than 5% of the total energy content of the oil[13].

Operating characteristics such as twin screw design, screw rotation speed, and press temperature all have an impact on the oil yield. The maximum oil output was recorded as 71% [14]. Using a hydraulic press, the effect of method parameters on the oil recovery was examined. The compression speed was 0.05–2.5 MPa/s, the applied pressure was 5–25 MPa, the pressing temperature was 25–105 °C, the pressing time was 1–30 minutes, and the time of preheating was 0–30 minutes. The amount of recovered oil increased as the temperature and the pressing duration increased. When Jatropha seeds were pressed at 15 MPa and 90°C for 10 minutes, the best oil recovery was obtained [15, 16]. A seed pressing vessel of 60 mm diameter and 80 mm height was used to crush the jatropha seeds [17]. Prior to dry heating, the seeds should be crushed with a hammer mill to particles less than 2 mm [18].

The influence of different production procedures on Jatropha oil properties such as fatty acid concentration, oil yield, and oil properties was investigated in this research. The produced oil was from non-edible jatropha seeds because of the higher price of edible oils. Using pure vegetable oils directly in diesel engine is restricted due to the higher density and viscosity of diesel oil. This paper used exhaust heat recovery as an economic way to reduce the oil density and viscosity to be near to that of diesel oil. The oil was extracted from jatropha seeds using screw, hydraulic, solvent and Soxhlet methods. Exhaust gases were used as a waste energy to heat water which used to heat the fuel. Free fatty acid contents and GC analysis were investigated for the different extraction processes. The density, viscosity, heating value, cetane number, and flash point of the produced oil were all investigated. The effect of preheating the oil on the density and viscosity of biodiesel and extracted oil by screw, hydraulic, soxhlet, and solvent was studied.

2. Methodology

The seeds were cleaned to separate foreign materials. Jatropha seeds have fragile shell which can be removed by an impact huller. Seeds were broken to reduce the particle size before processing. The seeds were manually removed from the stems and kernels and dried. The shells were removed and the seeds were cracked. Grinding machines were used to mill the kernels. There are four different processes for oil extraction from seeds: soxhlet extraction, screw, hydraulic press extraction and solvent based extraction.

1.1. Soxhlet extraction

Because the solvent has a limited solubility and the contaminant is insoluble in it, soxhlet extraction is performed. Figure 1 shows the designed soxhlet extraction device. The powdered jatropha seeds were placed in a filter paper and soaked in the soxhlet apparatus. The petroleum ether solvent was vaporized using a magnetic heat stirrer, which was then condensed using a reflux condenser. The grinded seeds were inserted in the magnetic heat stirrer with petroleum ether in a conical flask. To achieve petroleum ether purity, this process took four hours and was performed more than five times. A rotary evaporator is used to produce the oil from the mixture. The rotary evaporator's motor speed, temperature, and pressure are 115 rpm, 40 °C, and 2 bar, respectively. The oil yield is about 25% [1, 2].



Fig. 1: Soxhlet extraction process

1.2. Screw press extraction

The screw press is made from a helical screw on a conical shaft that rotates inside a cylindrical barrel. Near the discharge end of the assembly revolves as the shaft rotates, moving the seed from the hopper to the intake, where it is pushed. The oil is released through perforations in the barrel bottom, and the cake is charged at the barrel's open end. The screw is put into its housing, which is then held in place by the base. To feed the seeds to the rotating screw, the base and the housing contain holes on top of each other. The oil is collected from beneath the housing through a hole. As shown in Fig. 2, the current designed screw press is made up of three basic components: the base, the housing, and the screw. It is a continuous production method because it extracts a significant amount of oil from the seeds, about 19% by weight [3-5].



Fig. 2: Schematic diagram of the screw press

2.3 Hydraulic extraction

The designed hydraulic press acts as a pump that has humble mechanical force acting over a small cross-sectional area as shown in Fig. 3. The other part is a press having a larger area that produces a large mechanical force correspondingly. The pressure on a restricted fluid is transferred without being diminished and acts with equal force on equal areas at 90° to the wall of container wall. When the press is pushed internal, the incompressible oil is displaced, and the volume displaced by the small press is equal to the displaced volume by the large press. This causes a difference in the displacement which is proportional to the ratio of the press head area. A sieve plate covered with fine mesh was used. The pressing chamber has a controlled temperature from 30 to 100°C with a fixed diameter.

The press pressure is up to 100 MPa. The piston was lowered on the top of the seeds. The seeds were laid in the press chamber then the seeds were pressed by the piston. The pressure was extended linearly at the desired speed until it reached to the final pressure. The yield out of this extraction process is about 11% [19, 20].



Fig. 3: The hydraulic press Schematic diagram.

2.4 Solvent extraction

Solvent extraction can be done by passing the solvent through the solid mass of seed. The extractors fall into three classes: first, percolation in which the solvent flows by gravity through a bed of solids, second, immersion, in which the solids are immersed in the solvent, third, extractors which are the combination of the two types. The method produces concentrated solution due to the separation of insoluble solid such as pigments from a soluble material. To saturate the crushed seed and extract the oils, chemical organic solvents such as petroleum ether and n-hexane are utilized. The solvent is concentrated and reclaimed after the extraction procedure is completed. Solvent extraction steps are as follows: decorticating seeds by removing the shells, grinding seeds into powder, measuring 40 gram of jatropha seed powder into a beaker, pouring the heated jatropha powder into a round bottomed flask, adding 80 ml solvent to the round bottomed flask, stirring and shaking the mixture for 10 minutes, leaving the mixture to settle for 12 hrs to separate cake from solvent with oil, placing the round bottomed flask in the distillation column (rotary evaporator) at temperature between 65-69 °C to remove hexane and finally measuring the mass of the oil. The solvent extraction steps are shown in Fig. 4. The oil yield is about 20% [21, 22].



(a) petroleum ether and Jatropha powder (b) First filtration (c) Second filtration (clear oil)

Fig. 4: Sequence of solvent extraction process

2.5 Biodiesel production process

The purpose of acid catalyst-based esterification was to convert Free Fatty Acids (FFA) in the oil into esters, lowering FFA level below 1%. The oil was initially mixed with methanol and concentrated sulfuric acid in a reaction flask (1 % H₂SO₄ by weight). The reaction was carried out for one hour at 60°C with a 400 rpm agitation rate. Methanol was utilized in a 9:1 molar ratio with oil. The blend was left to settle for 2 hrs after the reaction. The top layer was removed, and the acid value of the reaction products was determined. The second phase of base catalyst based transesterification was carried out with the lowest FFA. Methanol (CH₃OH) with a purity of 99 % was employed, as well as sodium hydroxide (NaOH). The solid particles were filtered out of the oil. The oil was heated to 90°C for 30 minutes to remove any water that could cause saponification [5, 6]. The amount of methanol used was 20% of the mass of the jatropha oil by mass following the oil to methanol molar ratio 6:1. In a separate vessel, methanol was combined with 0.8 g of NaOH and stirred continuously until all of the NaOH blending with the methanol, resulting in Sodium Methoxide [7, 8]. Jatropha oil was heated to 65°C and blended with Sodium Methoxide for 90 minutes. The methyl esters were separated from the glycerin during the transesterification process [9, 10]. The mixture was placed into a separator funnel and left for at least 12 hours. The methyl ester floated on the top of the separating funnel, while the denser glycerin was in the bottom [11, 12]. After mixing the biodiesel, it was rinsed with warm water to eliminate any unreacted alcohol, oil, or catalyst. Then, it was left to settle for 24 hours under gravity until the bottom phase produced a pH value of 7 [15, 16].

2.6 Fuel preheated system

Fuel supply system allows two separate passes for preheated fuel and unheated fuel. These two separate passes allow for the use of preheated and unheated fuels. The fuel system design consists of two fuel tanks for preheated fuel and unheated fuel. The heat exchanger is a double pipe counterflow heat exchanger, in which the fluids enter from opposite ends. Heat exchanger components were shown in Table 1. Because it can transport most of the heat per unit mass, the counter current configuration is the most efficient. The water was warmed by the heat rejected from the exhaust heat recovery. The rejected heat depends on the applied load using diesel oil. The exhaust temperature ranges from 135 to 320 °C from zero to full load. The exhaust gas temperature was adjusted at no load for diesel oil. The water coil circulated around the exhaust muffler. The hot water transferred the heat to the jatropha oil to preheate the oil. The water exit temperature reaches up to 95 °C. The outlet exit temperature of water depends on the applied load. Control valve was used to control the mass of hot water that enters the heat exchanger. Temperature thermocouples were used to show the exit temperatures of water and oil. The heat exchanger schematic diagram is shown in Fig. 5.

Table 1: Specifications of the oil preheating heat exchanger

No.	Helical coil exhaust- water heat exchanger	Specifications
1	Heat exchanger material	Copper
2	Length of water coil	20 m
3	Water coil diameter	6.35 mm
4	Water coil height	60 cm
5	Flow direction	$\downarrow\uparrow$
6	Heating fluid	Exhaust gases
7	Cooling fluid	Water
8	Exhaust temperature	Between 125 to 320 °C
9	Water inlet temperature	25 °C
10	Water outlet temperature	up to 95 °C
	Parallel tube water- fuel heat exchanger	
2	Length of heat exchanger pipe	80 cm
3	Outer water pipe diameter	50.8 mm
4	Inner fuel pipe diameter	6.35 mm
5	Flow direction	$\downarrow\uparrow$
6	Heat source	Hot water
7	Cooling fluid	Fuel
8	Water inlet temperature	up to 95 °C
9	Fuel inlet temperature	25 °C
10	Fuel outlet temperature	up to 85 °C



- 6. Control valve
- 12. Fuel tube in13. Graduated glass cylinder

Fig. 5: Schematic diagram of fuel preheated system by exhaust.

3. Results and discussions

3.1 Free Fatty acid percentage

As seen in Fig. 6, the extraction process affects the color of the oil. The color extracted oil becomes darker as the extraction temperature and time increase. The oxidation that occurs throughout the extraction process is responsible for the dark color. Soxhlet extracted jatropha oil is darker than solvent extracted, hydraulic-extracted, and screw-extracted jatropha oil. The proportion of fatty acids is seen in Table 2. Screw press has lowest FFA content (2.7 %) and the lightest color. The FFA content of the oil extracted using soxhlet method is 21.1 % and its color is darkest. Oil color and FFA content are affected by extraction temperature which is influenced by the biodiesel synthesis process. The proportion of FFA in the oil is determined by titration. Four grams of NaOH were dissolved in one litre of distilled water (0.1 normality NaOH (4 gm per liter of water) solution. The end point is determined using the phenolphthalein indicator. One mL of jatropha oil was dissolved in isopropyl alcohol of 10 ml. The blend was then gently warmed and stirred until all of the oil was dissolved, and the mixture turned transparent. Two drops of phenolphthalein were added. Drops of NaOH solution were added to the oil alcohol phenolphthalein solution one at a time and stirred frequently until the solution became pink for 10 seconds. These formulas [23] were used to estimate the FFA % from the titration parameters:

$$FFA\% = (28.2 \text{ v n}) / \text{w}$$
 (1)

Where:

v = titration solution volume,

n = NaOH solution normality (0.025), and

w = The oil sample weight in grams (1ml = 0.92 g).

FFA% = 0.766 v

Table 2: Fatty acid concentrations of oil extraction processes

No.	Process of extraction	NaOH, ml	FFA, %	Color
1	Screw press	3.5	2.7	Lighter
2	Hydraulic	6.5	5.0	Light
3	Solvent	23.0	17.6	Dark
4	Soxhlet	27.5	21.1	Darker



Fig. 6: Oil color of extracted oils.

3.2 Gas chromatography analysis of Jatropha oil

The methylation technique was used to prepare oil samples for gas chromatography. During the methylation process, 2 grams of sodium hydroxide was dissolved in 100 ml methanol to produce methanolic sodium hydroxide. The blend was agitated for 2 minutes to get clear. A 0.2 g oil sample was mixed with methanolic sodium hydroxide of 6 ml. The mixture was then simmered for 10 minutes. The mixed solution is of 20 ml methanol and 30 ml concentrated HCL was refluxed for another 10 minutes. Then 10 mL of hexane was added and the mixture was refluxed for 2 minutes before cooling. Distilled water of 10 mL was added to the mixture and then put into a separating funnel. Calcium chloride was used to collect and dry the upper layer. The material was then used for GC analysis. For GC analysis, a Hewlett Packard model 5890 was utilized. which included a flame ionization detector (FID), an oven, and a fused silica capillary column DB wax (60 m \times 0.32 mm). The retention indices of the isolated fatty acids methyl esters components were computed using fatty acids methyl esters standards (C4-C22) (Sigma Aldrich Co.) as references. The saturation and poly unsaturation of the vegetable oil should be less. Jatropha oil has higher monounsaturated fats than other vegetable oils, making it a viable option for biodiesel manufacturing. Oleic, palmitic, linoleic and stearic fatty acids are the most abundant in jatropha oil. The unsaturated fatty acid content of jatropha oil is greater than other vegetable oils. In the presence of air, jatropha biodiesel is unstable and readily oxidized. The oxidation stability of jatropha biodiesel increased in the following order (in terms of the method used to prepare it), according to their linoleic acid concentrations: soxhlet, screw, hydraulic, then solvent, as shown in Table 3 and Fig. 7. Increases of linoleic concentrations lead to the poor oxidation stability and this affect on the biodiesel yield. Because of the palmitic acid concentration, the freezing point and flow characteristics of jatropha oil decrease in ascending order from solvent, screw, soxhlet, to hydraulic. This leads to the poor flow characteristic at low temperatures. Jatropha oil can be classified as oleic–linoleic oil [24].

Table 3: Composition of main fatty acids in jatropha oil of extraction processes (* related to the total number of carbon atoms to the number of carbon-carbon double-bonds).

No.	Fatty acid (mass fraction)	Hydraulic	Screw press	Soxhlet	Solvent
1	Palmitic (*C16:0)	17.69	25.26	23.83	28.91
2	Stearic (*C18:0)	3.16	2.11	0.04	1.86
3	Oleic (*C18:1)	22.64	17.09	0.47	0.05
4	Linoleic (*C18:2)	51.63	48.15	15.63	65.98



(c): Screw press oil extraction.



(d): Soxhlet oil extraction.

Fig. 7: GC spectra for different oil extraction methods.

3.3 Impact of extraction technique on oil properties

According to Table 4, the evaluated lower heating values of jatropha oil extracted using soxhlet, solvent, screw and hydraulic methods, and biodiesel were 39135, 38808, 39128, 39201, and 38789 kJ/kg, respectively. The output power improves as the heating value of the fuel increases. For jatropha oil extracted by screw, soxhlet, solvent, hydraulic, and biodiesel, the measured cetane numbers were 37.83, 37.53, 37.62, 39.12, and 42.62, respectively. The high Cetane numbers produces shorter ignition delay and lower engine performance. Screw, hydraulic, soxhlet, and solvent extractions of jatropha oil have flash points of 142, 146, 140, and 138 °C, respectively. Because the flash points of the extracted oil by various production procedures are greater than those of diesel oil, storage and handling of these oils pose a lower risk than diesel fuel, as shown in Table 4. The higher value of lower heating value and higher flash point are favorable. Cetane number near to diesel oil is favorable. Hydraulic extraction process produces the favorable lower heating value, Cetane number and higher flash point about other extraction processes because the higher contents of Oleic and Linoleic acids [22-24].

Table 4: Properties of prepared jatropha oil using different extraction

No.	Production process	Flash point, °C	Cetane number	Lower Heating value, kJ/kg	Yield, %
Test Method		ASTM D- 92	ASTM D-13	ASTM D-240	
1	Screw press	142	37.83	39128	19
2	Hydraulic	146	39.12	39201	11
3	Soxhlet	140	37.53	39135	25
4	Solvent	138	37.62	38808	20
5	Biodiesel	121	42.62	38789	
6	Diesel oil	75	45	42000	

3.4 Influence of preheating temperature on extracted oil density

Figure 8 depicts the differences in density of jatropha oil at various preheating temperatures. The findings show that the temperature at which oil is preheated has a substantial impact on density. At a temperature of 20 °C, the determined values of oil density by ASTM D-1298 are 916, 914, 808 and 906 kg/m³, respectively for hydraulic, screw, Soxhlet and solvent methods related to just 825 kg/m³ for diesel oil. The density of the oil decreases as the temperature of the oil increases. The oil density can be formulated as linear relationship ($\rho = -0.5247$ T + 923.79), ($\rho = -0.5760$ T + 923.73), ($\rho = -0.5277$ T + 920.38), ($\rho = -0.5146$ T + 915.69) and ($\rho = -0.6072$ T + 885.84) with oil temperature for hydraulic, screw, Soxhlet, solvent methods and biodiesel, respectively.Jatropha oil density variation at different temperatures was referenced to diesel oil (825 kg/m³) [22, 23].

3.5 Impact of preheating temperature on oil viscosity

Jatropha oil has a substantially higher viscosity than diesel oil as shown in Fig. 9. The measured values of viscosities by ASTM D-445 for jatropha oil at 40°C are 4.1, 4.4, 3.9 and 4.3 Cp for screw, hydraulic, solvent and Soxhlet, respectively as compared to only 1.2 Cp for diesel oil at the same temperature. The jatropha oil viscosity at different temperatures was compared to diesel oil in Fig. 9. The viscosity decreases when the temperature of the oil rises. Preheating bio oil fuel lines is an approach for dealing with the problems caused by the greater oil viscosity. The curve-fitting equation of the experimental data for the viscosity of jatropha oils using power law function as ($\mu = 84.624 \text{ T}^{-0.809}$), ($\mu = 76.152 \text{ T}^{-0.795}$), ($\mu = 76.562 \text{ T}^{-0.806}$), ($\mu = 69.746 \text{ T}^{-0.788}$) and ($\mu = 5.4451 \text{ T}^{-0.366}$), respectively for hydraulic, screw, Soxhlet, solvent processes and biodiesel as confirmed by references [22, 24].







Fig. 9: The oil viscosity at different oil temperatures.

4. Conclusions

The major aim of this research is to demonstrate the oil yield, free fatty acids (FFA), physical and chemical properties of jatropha seed oil extracted using various methods (i.e. screwbased, hydraulic, solvent-based, and soxhlet-based). The main conclusions are summarized as follows:

- The oil yields are 19, 11, 25 and 20% for screw, hydraulic, Soxhlet and solvent extraction processes, respectively.
- For the oils extracted using screw press, hydraulic, solvent, and soxhlet methods, free fatty acids are 2.7, 5, 17.6, and 21.1%, respectively. Palmitic Oleic, linoleic and stearic fatty acids are the most abundant in jatropha oil.
- Hydraulic extraction process produces the favorable lower heating value, Cetane number and higher flash point about other extraction processes because the higher contents of Oleic and Linoleic acids.
- The lower heating values of jatropha oil extracted using soxhlet, solvent, screw and hydraulic methods, and biodiesel were 39135, 38808, 39128, 39201, and 38789 kJ/kg, respectively. For jatropha oil extracted by screw, soxhlet, solvent, hydraulic, and biodiesel, the measured cetane numbers were 37.83, 37.53, 37.62, 39.12, and 42.62, respectively. Screw, hydraulic, soxhlet, and solvent extractions of jatropha oil have flash points of 142, 146, 140, and 138 °C, respectively.
- The oil viscosity and density decrease with increasing oil temperature. The oil density can be formulated as linear relationship ($\rho = -0.5247 \text{ T} + 923.79$), ($\rho = -0.5760 \text{ T} + 923.73$), ($\rho = -0.5277 \text{ T} + 920.38$), ($\rho = -0.5146 \text{ T} + 915.69$) and ($\rho = -0.6072 \text{ T} + 885.84$) with oil temperature for hydraulic, screw, Soxhlet, solvent methods and biodiesel, respectively.

• The viscosity of jatropha oils was correlated as ($\mu = 84.624$ T^{-0.809}), ($\mu = 76.152$ T^{-0.795}), ($\mu = 76.562$ T^{-0.806}), ($\mu = 69.746$ T^{-0.788}) and ($\mu = 5.4451$ T^{-0.366}), respectively for hydraulic, screw, Soxhlet, solvent processes and biodiesel

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