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Extraction and Characterization of Crop Oil from Seed Kernels of Feunkase (*Thevetia peruviana*) as Feedstock for Biodiesel Production

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ABSTRACTS

The seed kernel of Feunkase (*Thevetia peruviana*) was extracted with n-hexane and methanol mixture in a soxhlet extractor. The optimum condition for extraction of the crop oil from the seed kernel of Feunkase using conventional soxhlet technique was studied. Solvents used weren-hexane and n-hexane–methanol binary solvent. The parameters investigated effecting the oil yield involved various solvent polarities, extraction time, and temperature. Each experiment was conducted in 250 cm^3 soxhlet apparatus. The extracted oil was analyzed to examine the physicochemical characteristic, included: density, kinematic viscosity, acid, iodine, and saponification value, and water content. The optimum conditions were found after 4.0 h extraction time, extraction temperature of 70 ^oC, and n-hexane - methanol ratio of 50:50 (polarity index 3.30). The oil extract was found to be 46.84 ± 0.26%. The physicochemical properties of the extracted oil were density of 785 (kg/m^3), viscosity of 0.68 (mm^2/s), iodine value of 41.11 (g I_2/100 g oil), acid value of 0.09 (mg KOH/g oil), saponification value of 108.4 (mg KOH/g oil), and water content of 0.034 (%). These results revealed that the crop oil from seed kernel of Feunkase is one of the potential feed stocks for biodiesel production.

Keywords: biodiesel, crop oil, Feunkase, feedstock, thevetia peruviana
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1. Introduction

Feunkase, popularly known as *Thevetia peruviana* or yellow orleander, is a small tree that is grown as hedge and kept for its bright and attractive flowers basically used as an ornamental, evergreen plant in many tropical countries and belongs to the family of *Apocynaceae*. The Feunkase tree, which grows to about 2-6 m in height, can produce 400-800 fruits at a whole the year depending on the climatic conditions and age of the plant (Temitayo, 2017). It stars flowering after one and a half year planted and it blooms thrice a year. In a hectare, 3000 saplings can be planted and out of which 52.5 tons of seed can be collected (Balusamy and Marappan, 2007).

There has been reported (Ibiyemi *et al.*, 2002; Dhoot *et al.*, 2011) that Feunkase seed kernels has a high level of oil content (about 60-65%) and valuable protein content (30-37%), with fatty acid composition mainly (i) palmitic acid (C16:0), (ii) stearic acid (C18:0), (iii) oleic acid (C18:1) and linoleic acid (C18:2) (Ibiyemi *et al.*, 2002; ; Basumatary, 2013; Panchal *et al.*, 2017). Despite the fact that there is high level of oil in the seed, it remains non-edible because of the presence of toxic compounds which are mostly cardiac glycosides and their free aglycones such as thevetin, theveridoside, theveside, cerberin, peruvoside, perusitin, and digitoxigenin (Oluwaniyi and Ibiyemi, 2007; Kishan *et al.*, 2012; Godson and Udofia, 2015). It's non-edible and high oil content of the seed indicated potentiality of the plant as raw material for biodiesel production.

Crop oil from seed kernels of Feunkase can be extracted using several methods include solvent extraction, mechanical pressing, and supercritical fluid extraction. Mechanical pressing is commonly used but the oil yield from this method usually low and contains a high percentage of water. Highly purified oil is obtained from supercritical fluid extraction but it has high operating and investing costs (Temitayo, 2017). In this study, soxhlet extraction method was used to extract the crop oil from seed kernels of Feunkase. The choice of solvent type (polarity), extraction time, and temperature are the main factors determining the amount of extraction product (Markom *et al.*, 2007; Straccia *et al.*, 2012; Sicaire *et al.*, 2015). The inability to control these factors can lead to failure to achieve high yields and oil quality. The objective of this study was to determine the oil yield and quality. The effect of n-hexane–methanol solvent with different polarities, extraction time, and temperature on extracted oil was studied. In addition, the physicochemical characteristics of the oil produced from the soxhletation process determining the oil quality were also investigated.

2. Materials and Methods

2.1. Raw Material and Pre-treatment of Material

The seed kernels of Feunkase (Thevetia peruviana) were collected from Amarasi, Kupang District of East Nusa Tenggara Province, Indonesia. After collecting, the seeds were carefully separated from the shells and removed from unwanted ingredients, such as remnants of the outer shell, twigs, and fine pebbles. The seeds of Feunkase were opened manually to obtain their kernels. The kernels were grinded using a sieve plate and shaker grinder to reduce it into a size of range 0.5 – 0.75 mm and dried at 60 ^{o} C to a constant weight. The resulted powder was used for all experiments.

2.2. Chemical and Reagents

All chemical used in this work, such as n-hexane, iodine, methanol, potassium hydroxide, hydrochloric acid, glacial acetic acid, sodium thiosulfate, and potassium iodide, were of analytical reagent grade obtained from Cica-Merck (Kantako Chemical).

2.3. General Extraction Methods

A sample of 25 g of seed kernels of Feunkase was placed in the thimble of the soxhlet apparatus with 200 cm^3 extracting solvent (n-hexane-methanol mixture) in a 250 cm^3 round-bottomed flask and extraction was carried out at 70 ^{o} C for 4 h with different ratios of n-hexane to methanol (100: 0; 90:10; 80:20; 70:30; 60:40; and 50:50 v/v) to give the solvent polarity of 0.00, 0.66, 1.32, 1.98, 2.64 and 3.30, respectively. At the end of the extraction process, the solvent was removed by a vacuum evaporator at 40 °C for 1 h. Furthermore, the oil obtained was filtered and then mixed with 2 % of distilled water. Subsequently, the mixture was heated at 70 °C and stirred for about 30-60 minutes, added magnesium sulfate anhydrous and filtered to obtain the oil extract. The oil obtained after evaporation was weighed and the oil yield was calculated. All experiments were repeated at least twice and the final value was the average of all. The oil yield was calculated by using equation 1 (Panchal *et al*., 2013).

$$\text{Oil yield (\%)} = \frac{Weight\ of\ oil\ x\,100}{Initial\ weight\ of\ sample} \quad (1)$$

2.4. The Effect of Extraction Temperature and Time to Oil Yield

The effect of extraction temperature and time were investigated in order to obtain the optimum conditions for extraction to achieve maximum oil yield. Six different temperatures of 65, 70, 75, 80, 85 and 90 ^{o} C were investigated to obtain the optimum extraction temperature. Furthermore, the extractability of the oil was also investigated as a function of extraction time. The seed kernels powder of Feunkase was extracted for 3.0, 4.0, 4.5, 5.0, 5.5 and 6.0 h.

3. Results and Discussion

3.1. Effect of Solvent on Extract Yield

The successful separation of oil components from the Feunkase seed kernels using soxhletation technique highly depends on the level of solvent polarity and components separated. The polarity of the solvent is measured from the dipole moment. The n-hexane solvent was used because it has a high separation level and selectivity. n-Hexane including nonpolar solvents with a polarity index or Snyder index was 0.0 (zero), while methanol with Snyder index of 6.6 was a polar solvent (Markom *et al.*, 2007). The physical properties of the solvents used are listed in Table 1.

Table 1. Physical properties of n-Hexane and Methanol (Markom et al., 2007)

Physical Properties	Unit	Name of Solvent	
		n-Hexane	Methanol
Polarity (Snyder index)	-	0.0	6.6
Dipole moment	Debye	0.0	1.7
Boiling point	^{o}C	69	65
Dielectric constant	-	1.88	32.61
Cohesive energy density	J mol/cm^3	200.76	808.26
Viscosity	mPa	0.30	0.54
Surface tension	cal/molA^2	25.75	31.77

In this study, various combination of n-hexane and methanol mixture with polarities, 0.0 to 3.3, were tested for their extraction efficiency. The amount of extracted oil was calculated as percentage of total oil contained by the Feunkase seed kernels. The effect of the solvent polarity on oil yield was shown in Fig. 1.

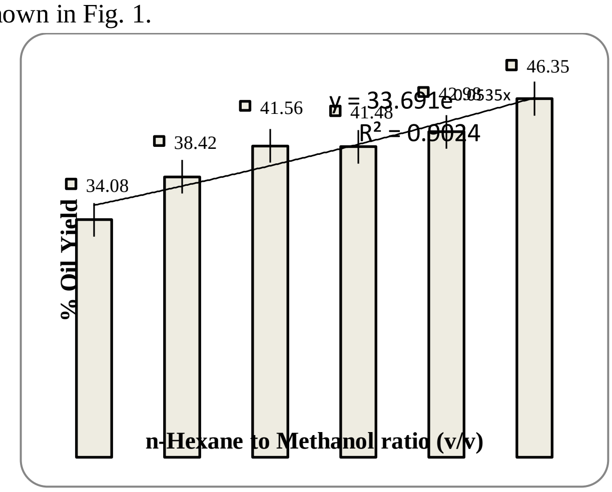

Fig. 1. Effect of biner solvent (n-hexane-methanol) on oil yield at 70 ^{o}C of extraction temperature and 4 h of extraction time

The extracted oil components increase with increasing methanol composition or solvent polarity (Fig. 1). Higher polarity of the binary solvents (n-hexane: methanol) higher oil yield of the results, in which the polarity changes from 0.0 to 3.3 generated the yields equaled to 36 % higher than the initial yields. These results indicated that extraction process highly depends on the ability to control the solvent polarity The data also shows that soxhlet extraction using a single solvent of n-hexane or methanol is difficult to obtain highly oil yields because the soxhlet method does not select a single compound of the multi-component compounds existing within the plant material which has complex interactions. The boiling point of the solvent, surface tension, and viscosity could decrease at a lower temperature; therefore, the solvent can more easily reach the active sites of the matrix. Methanol with a higher surface tension and viscosity than that of n-hexane usually not desirable in extraction because it's solvent absorption could be inhibited at the active sites of the material matrix, however, it's dielectric constant and cohesive energy density are markedly higher than the n-hexane, the methanol molecules can strongly bond to polar oil components in the matrix (Markom et al., 2007). Therefore, the ratio of n-hexane to methanol could be arranged to be 50 : 50 (polarity 3.3) which gave the highest oil yield and then it was considered the best solvent choice and to be studied further.

The chemical composition contained within the oil consisted of triglycerides (as the main component), free fatty acids (FFA), mono and diglycerides, and other components such as phosphor glycerides, vitamins, and minerals. The difference in solvent polarity produces oil containing different composition between triglyceride and FFA.

The solubility of the oil components depends on solvent polarity which involved the contribution of the electrostatic forces, formation of hydrogen bonds, and molecular conformation. The tendency of the solvent molecules to donate their electrons (nucleophilic) and/or accept electrons (electrophilic) during interactions within the macromolecules of the host matrix in soxhlet system determines the final oil components of the extracts.

3.2. Effect of Extraction Temperature and Time

Temperature affects the rates of extraction and oil yields because temperature could influence the kinetic energy of solvent and the collision frequency between solvent and target molecules. The effects of the temperature in extraction calculated from the amount of oil yield using n-hexane and methanol mixture solvent are shown in Fig. 2.

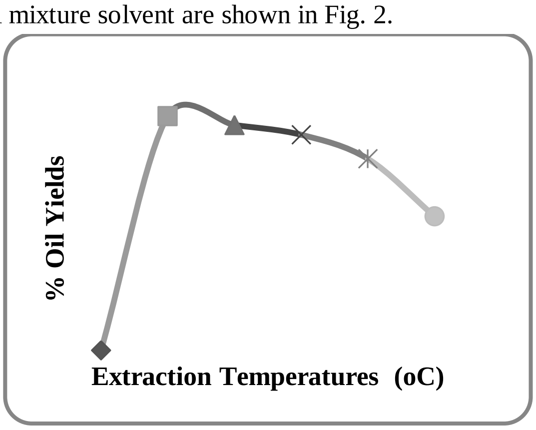

Fig. 2 The effect of temperatures on oil yield at the reaction condition of n-hexane to methanol ratio of 50:50, extraction time 4 h

The tendency of increasing oil yield occurs in the range of temperature from 65 to 70 ^{o}C. In this range, extracted oil was increased by 43 %. However, extraction at 75-90 ^{o} C, the oil yield decreased by 1.2 to 12.9 % (Fig. 2). These facts conclusively explained that an increase of temperature reduced the oil viscosity and therefore increase mobility of oils in cellular walls. Based on these facts, the 70 ^{o} C was the adequate temperature to give the highest yields of crop oil from the seed kernels of the Feunkase.

Instead of the temperature, the extraction time is one the important factors influencing the yields. An appropriate timing of extraction directly affects to the oil yield, due to the maximum interaction between the solvent and the oil molecules when the contact time and residence time of the solvent in the extractor were optimum. Fig. 3 showed the total amount of oil extracted from the seed kernels of Feunkase at different extraction time.

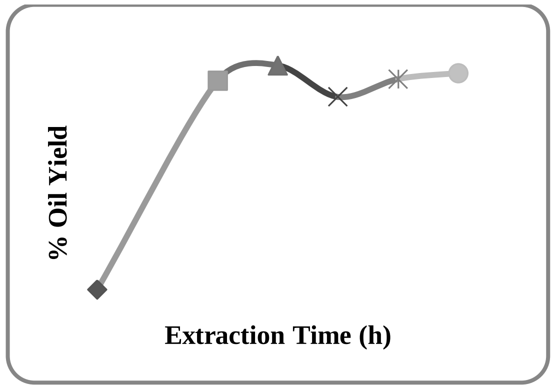

Fig. 3.Effect of extraction time on oil yield. Condition: n-hexane to methanol ratio of 50:50, 70 ^{o}C

Fig. 3 shows the total amount of oil extracted from seed kernels of Feunkase at different extraction time. It was observed that the oil yield increase by 12.6 % when the extraction time was increased from 3 hours to 4 hours, but within the extraction time range of 4 hours to 6 hours the extracted oil product did not change significantly. Hence, the extraction time of 4 h was chosen as optimum time for Feunkase seed kernels oil yield.

3.3. Physical and Chemical Properties of Extracted Oil

Before the transesterification process to convert extracted oil to methyl ester, it was analyzed to determine its physical and chemical properties. The results of analysis were needed to determine the process stages that would be used in the production of methyl ester and determining the required of the methanol and catalysts requirements. The parameters analyzed include: density, viscosity, acid value, iodine value, saponification value and water content. The physicochemical properties of the extracted oil at the optimum conditions are shown in Table 2.

Table 2. Physicochemical properties of crop oil from seed kernels of Feunkase

Properties	Unit	Value	SNI Specification
Density	kg/m^3	785	850 – 890
Viskosity	mm^2/s	0.68	2.3 – 6.0
Iodine value	g I_2/100 g oil	41.11	115 max
Acid value	mg KOH/g	0.09	0.8 max
Saponification value	mg KOH/g	108.41	500 max
Water content	%	0.034	0.05 max

Density and viscosity are physical characteristics that have been observed for extracted oil. Density is a measure of mass per unit volume of material or substance. Based on the analysis result, it was found that the density of extracted oil before being processed into biodiesel was 785 kg/m^3. This value was lower than the value of SNI (850 - 890 kg / m^3). Oil density is influenced by the composition of fatty acids and the purity of raw materials. Density would be decreased with increase the lenght of carbon chain or decrease in the number of double bonds in fatty acids. Viscosity is closely related to the ability of the material to flow. The thicker a liquid, the greater the force needed to make it's flow at a certain speed. The viscosity as a measure of oil resistance to fluid obtained in this works was 0.68 mm^2/s indicating that the viscosity of oil from Feunkase seed kernels fulfilled the standard of SNI. This results agreed with the findings (Joeliangsih *et al*., 2008) that the unsaturated fatty acids composition of the Feunkase seed kernels oil would contributed most to the viscosity value. The more the number of double bonds with the same length of carbon chain, the smaller the viscosity.

The saponification value can be used to identify the type of oil or fat. It is determined by the molecular weight of the constituent fatty acids. The saponification value obtained in this work was 108.41 mg KOH/g of oil. The low saponification value of the extracted oil indicated that the triglyceride has along carbon chain. The longer the carbon chain of fatty acid, the less fatty acid content in the oil. Oils composed by long-chain fatty acids, higher molecular weight smaller saponification value, on the contrary, lower molecular weight higher saponification value.

The acid value indicated the level of free fatty acids in the oil. The acid value is equivalent to the amount of milligram KOH required to neutralize free fatty acids in the oil. The presence of free fatty acids in biofuel products can lead to the formation of ash at the time of combustion, clogging the filter with precipitate and corroding the diesel engine. The acid value can be an indicator of damage occurring to the oil, due to oxidation activity. The results of present study showed that the acid value in extracted oil was 0.09 mg KOH/g oil, this value met the SNI for biodiesel products which is a maximum of 0.8 mg KOH/g oil. The lower the acid values better the quality.

The iodvalue indicated the degree of unsaturation or the number of double bonds contained in the oil. The iodvalue obtained in this study was 41.11 g I_2/100 g of oil. This value was fullfiled the standard of SNI. This result indicated that the amount of iodine that is bound to the double bond is relatively small low the degree of unsaturation of fatty acids. The numbers of mono-unsaturated fatty acid compounds in oil make them easy to react with oxygen during combustion and forming chains with very large molecular weights. The iodine value has a direct relationship with the viscosity and cetane numbers. The higher the iodvalue, the lower the cetane number and vice versa. Decreasing viscosity and cetane numbers will result in increasing the oil unsaturation. The most optimal iodine value for raw material of biodiesel was 70-100 g I_2/100 g oil.

The water content in the extracted oil is one of the important factors that must be considered before it further processed into biodiesel products. Water content in oil will worsen the quality of oil because it will accelerate the hydrolysis of triglycerides to form glycerol and fatty acids. The more hydrolysis reactions that occur, the more free fatty acids formed, higher the acid value. Water content in raw materials of biodiesel (methyl esters) maximum was 1 %. The water content of oil extracted from seed kernels of Feunkase using n-hexane and methanol solvent (50:50) was 0.034 %. This value is relatively low and meets the SNI specified quality standards.

4. Conclusion

Feunkase seed kernels oil extacted by using n-hexane-methanol binary solvent potentially be utilized as feedstock for biodiesel production. Thus, it could reduce dependency on using conventional and edible oil feedstock. The optimum conditions were obtained at n-hexane to methanol ratio of 50:50 (polarity 3.30), extraction temperature of 70 ^{o} C and extraction time of 4.0 h. The optimized oil yield was found to be 46.84 ± 0.26 % (weight basis). It had been observed that solvent polarity and extraction time were the most important parameters affecting the oil yield.

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Conflict of interest: Non declare