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Optimization of Soxhlet Extraction of Candlenut Oil *(Aleurites moluccana (L.) willd)* Using Factorial Experimental Design Level 2^3

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Abstract – *Candlenut (Aleurites moluccana (L.) wild) is a plant of the Euphorbiceae family. Candlenut oil is obtained by extracting the oil content in candlenut seeds. Soxhlation is an effective method for solvent extraction, ensuring continuous contact between the solvent and the material to extract the oil efficiently while maintaining the purity of the solvent. In this study, optimization of candlenut oil extraction was carried out using the factorial experimental design level* 2^3*. Using this method, it can be determined that the extraction time is the most influential variable in the candlenut oil extraction process. The results showed that the optimum operating conditions were an extraction time of 185 min, a candlenut seed powder size of 20 mesh, and a ratio of material-solvent ratio of 1:6 g/g, resulting in an oil yield of 43.2%. The analysis of candlenut oil conducted at an extraction time of 185 min revealed the following results: a refractive index value of 1.4736, a moisture content of 0.08% bb/b, which complies with the standard SNI 01-4462-1998 for candlenut oil, a viscosity of 15.47 Cp, and a density of 0.869 g/mL. The analysis of the free fatty acid number of 2.4%bb/b and saponification number of 181.55 mg KOH/g is not consistent with SNI 01-4462-1998 candlenut oil.*

Keywords: *candlenut seeds; extraction; vegetable oil*

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INTRODUCTION

Candlenut oil has seen a significant increase in consumer demand over time. Farmers are given the chance to fulfill market demand as a result. By enhancing the processing system, candlenut oil has the potential to grow both in volume and as a source of vegetable oil, for instance. Even in Indonesia, traditional methods are frequently used to process candlenut oil, which makes it challenging for producers of the oil to identify optimal operating conditions. As a result, candlenut oil production has poor quality and yield (Sariyusda, 2017). Therefore, a proper extraction process is crucial to produce candlenut oil of high quality and in large quantities (Nofrin et al., 2012).

Soxhlation is a useful technique for solvent extraction procedures because it keeps the solvent in constant contact with the material, maximizing the amount of oil that can be extracted while maintaining the solvent's purity. In addition, the solvent can be recovered and reused (Voigt, 1994). Solvent selection also has a strong influence on the extraction process. High selectivity, low solvent boiling point, polarity, low toxicity, insolubility in water, inertness or the lack of reaction with other components, low toxicity, and an affordable price are some examples of factors that affect solvent selection in general. N-hexane, the lightest solvent type with a dielectric constant of 1.89, which includes non-polar solvents, is the solvent that can extract the oil content present in grains, according to Guenther (1987).

In the study conducted by Yanuar et al., (2017) on the extraction - distillation method of sunflower seed oil extraction with n-hexane solvent and ethanol solvent as a comparison, the results of the extraction process using n-hexane obtained a yield% of 43.89 - 81.61%, acid number 0.927 - 1.659, peroxide number 1.560 - 7.80, saponification number 122.851-124.352, FFA% of 1.826 - 2.494, iodine number of 55.429 - 100.961, and oil density of 0.867 - 0.907. Meanwhile, the ethanol solvent extraction process produced a yield of 78.75-91.25 percent, an acid number of 0.009 -

1.226, a peroxide number of 46.8 - 81.9, a saponification number of 112.843 - 119.849, an amount of FFA% of 1.826 - 2.494, an amount of iodine of 55.429 - 100.961, and an oil density of 0.867 - 0.907. Based on this study, it can be inferred that the use of n-hexane solvent yields the best results compared to ethanol solvent in terms of quality. However, ethanol solvent is superior when considered in terms of quantity. In the study conducted by Erna et al., (2017), candlenut seed oil was subjected to supercritical CO_2 extraction, and process optimization was conducted using Taguchi orthogonal array. The physicochemical properties of the oil were evaluated under varying pressure variables (25, 30, 35 MPa), temperature variables (40°C, 50°C, 60°C), and material particle sizes (0.8 mm, 0.55 mm, 0.3 mm). The best results were obtained at a pressure of 35 MPa, a temperature of 60°C, and a material particle size of 0.8 mm. The yield is 61.4%. In contrast, Fuad's research (2019) examined the effects of temperature and extraction time on the kinetics of soxhlation extraction of candlenut oil using methanol as the solvent. The oil content was found to be 32.89% with an ideal extraction time of 80 min. In this study's factorial experimental design level 2^3, the most important process variables and the interactions between them were identified to optimize the extraction of candlenut seed oil.

METHODOLOGY

Materials and Tools

Candlenut seeds, n-hexane, and distilled water are components used in the extraction of candlenut seed oil. Chemicals were purchased at Multikimia Raya Store in Semarang. To separate the oil using solvents, a series of distillation tools are used, while a series of soxhlation tools that operate at 70°C are used in the extraction process. Figure 1 depict a Soxhlet apparatus.

Fixed Variable Extraction

1. Extraction Temperature : 70°C
2. Solvent : n-Hexane 99%

Fixed Variable Solvent Evaporation

1. Oven Temperature : 70°C
2. Distillation Temperature : 70°C
3. Time of frying : 60 min
4. Distillation Time : 60 min

Variable Changes

1. Extraction Time (T)
   190 min (+)
   170 min (-)
2. Particle Size (S)
   30 *mesh* (+)
   10 *mesh* (-)
3. Material to Solvent Ratio (R)
   1 : 8 g/g (+)
   1 : 4 g/g (-)

Figure 1. Soxhlet apparatus series

Candlenut Seed Extraction Procedure

First, assemble the Soxhlet extraction device, which consists of a three-neck flask, Soxhlet sleeve, and condenser. Secure the device using clamps and stands. After that, add the ground candlenut seeds following the variables specified in procedure Table 1.

Table 1. Candlenut Seed Extraction Procedure

Candlenut	Mesh	N-hexane	Time	Temp
50 gr	10(-)	1 : 4	170 mins	70°C
	30(+)	1 : 8	190 mins	

The next step involves using a variety of distillation apparatus at 70°C to separate the candlenut seed oil from the n-hexane solvent. The remaining solvent was then evaporated from the resulting oil for 60 min in an oven set at 70°C. The resulting candlenut seed oil was then weighed and kept at a low temperature in a bottle for analysis.

RESULTS AND DISCUSSION

There were eight runs in this study, with various treatment applied to each run. The oil yield produced by using extraction time variables at the lower level (-) 170 min yields less oil than the upper level (+) 190 min, as shown in Table 1's results of data processing with a factorial experimental design, where the oil yield has a value that corresponds to the sign of the variable level of the extraction process time. By using the quicker method to optimize this research, it is necessary to know the process variables that have a significant effect on the research and to focus on the calculation of the main effect and interaction on the resulting yield.

Table 2. Extraction Results of Candlenut Seed Oil

Run	Variable Changes			Interaction				X1	X2	X3	X4	X5
	R	**T**	**S**	**RT**	**RS**	**TS**	**RTS**					
1	-	-	-	+	+	+	-	0,4	1,507	15,55	0,85	24,6
2	+	-	-	-	-	+	+	0,8	1,496	15,32	0,86	26,4
3	-	+	-	-	+	-	+	1,0	1,472	15,37	0,86	33,0
4	+	+	-	+	-	-	-	1,0	1,470	15,62	0,85	39,0
5	-	-	+	+	-	-	+	0,6	1,475	15,54	0,85	27,0
6	+	-	+	-	+	-	-	0,6	1,478	15,77	0,88	28,2
7	-	+	+	-	-	+	-	0,6	1,475	15,32	0,87	31,2
8	+	+	+	+	+	+	+	0,8	1,474	15,48	0,84	43,0

X1 = FFA Content X4 = Density
X2 = Refractive Index X5 = Yield
X3 = Viscosity

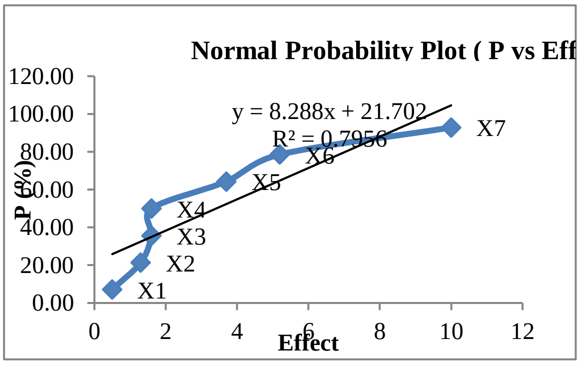

Figure 2. Normal Probability Plot for 2^3

Figure 2 shows a normal probability plot between the P-value and the effect in which a regression (R^2) value of 0.7956. Based on Figure 2, it can be concluded that 79.56% of the independent variables can influence the dependent variable. Therefore, in the production of candlenut oil, the extraction time is identified as the most influential variable on the %yield of candlenut oil. Additionally, the interaction between the extraction time and the solvent-to-material ratio also affects the %yield of candlenut oil. From this analysis, we can directly optimize the process by varying the extraction time (T) to determine the optimal yield of candlenut oil.

Optimization of Candlenut Seed Oil Extraction

According to Table 2, an increase in oil yield occurs between 170 and 185 min of extraction time, and a decrease occurs at 190 min. This can be seen in the candlenut seed oil extraction optimization graph in Figure 3.

Based on the yield of candlenut seed oil produced, it can be seen that the oil yield is getting higher as the extraction time increases. With the ideal operating conditions of a seed size of 20 mesh and a material-to-solvent ratio of 1:6 g/g, it is clear from Table 2 and the graph in Figure 3 that the extraction time of 185 min produced the highest oil yield, which is 43.2%. So it can be concluded that the longer the extraction time given will increase the circulation of solvents (reflux), causing the oil components in candlenut seeds to be dissolved into the solvent.

Table 3. Optimization Results of Candlenut Oil Extraction

Extraction Time (mins)	Particle Size (*mesh*)	Material Solvent Ratio (g/g)	Yield (%)
170			23.8
175			27.6
180	20	1 : 6	32.8
185			43.2
190			42.6

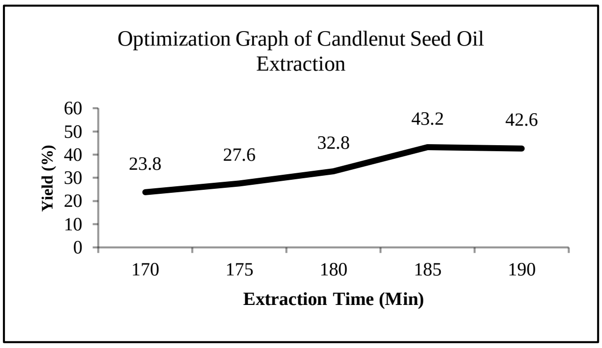

Figure 3. Optimization Graph of Candlenut Seed Oil Extraction Results

This method is based on the mass transfer of solid ingredients into the solvent, where the initial mass transfer takes place at the interface layer. The active substance will dissolve outside the cell after the organic solvent breaks through the cell wall and enters the cavity where it is present. Once equilibrium is reached in terms of the concentration of active substances inside and outside the cell, the concentrated solution will continue to diffuse out of the cell. As a result, lengthening the extraction process will not result in a higher yield of candlenut seed oil. It decreased in the fifth experiment because the oil in the sample had been extracted, as shown in Table 2 and Figure 3.

Candlenut Oil Extraction Analysis Results

Refractive Index Analysis

Refractive index is one of the factors that determines how light behaves differently when it travels through air in a bright medium. The refractive index is measured using a refractometer. The refractive index of candlenut oil produced in the study is 1.4753, and the refractive index of candlenut seed oil in the research of Nababan et al., (2018) is 1.4736. This is in accordance with the SNI of Candlenut Seed Oil. Refractive index testing, according to Formo (1979), is one method for determining the purity of oil. The length of the carbon chain and the quantity of double bonds in fatty acids will both increase the refractive index of the oil. The density of oil has an impact on its refractive index. The weight fraction within an oil determines the density of the oil. This implies that there is a connection between density and the refractive index. The density of the oil increases when there is a higher concentration of heavier components. This, in turn, causes the refractive index of the oil to increase. The reason behind this is that when light passes through the oil, it is refracted at an angle closer to the normal line.

Viscosity Analysis

A liquid's viscosity is the measure of the frictional force acting on it and expresses how viscous a liquid is. Viscosity in fluids is caused by the friction within the liquid layers, whereby a greater amount of friction results in higher viscosity. Conversely, lower friction leads to lower viscosity. Pressure, temperature, and concentration are the variables that determine the viscosity value. In this study, 15.47 Cp was used to measure viscosity.

The viscosity value of candlenut oil at 100°C is 14.86 Cp, according to Sudik et al. (2013). The viscosity measurement in this study was performed at room temperature because viscosity is inversely proportional to temperature, and the resulting value increased. The obtained viscosity value cannot be verified to see if it satisfies the requirements because there is no viscosity quality standard established for candlenut oil. The reference material or standard used to assess the viscosity of candlenut oil is derived from earlier investigations and is a benchmark for assessing the produced oil's viscosity.

Density Analysis

Density is one of the variables used to determine the specific gravity of the components in a sample. The weight fraction increases as the number of components in the oil increases. This indicates that the oil has a higher specific gravity. According to Yusnita et al. (2001), this specific gravity test can be used to determine the purity of oil. The density of candlenut oil, which is 0.869 gr/mL, was determined from the study's findings. The density range for candlenut seed oil, according to Ketaren (2008), is 0.924–0.929 gr/mL. However, it is impossible to say whether candlenut oil density satisfies the requirements because there is no quality standard. The standards or literature from earlier studies are used as a reference to calculate the density of the oil that was obtained.

Moisture Content Analysis

The amount of water in the oil is one of the factors affecting its quality. A high water content in the oil will lower the quality of the oil produced because water is a catalyst for oil hydrolysis (Ketaren, 2008). This study produced a moisture content of 0.08% b/b, which is in accordance with the SNI of candlenut seed oil when compared to the moisture content of oil from previous research, which is 0.18% b/b. The reduction of solvents that may contribute to oil degradation and the removal of water content in the sample are achieved through repeated heating, particularly in the distillation and oven processes. This process leads to a decrease in the water content. According to Swern (1982), hydrolysis will occur easily in materials with a high water content, resulting in the production of oil with a high water content as well. Winarno (1988) asserted the same phenomenon, saying that the faster evaporation happens, the less water there is in the material, and the longer the heating lasts at a particular temperature.

Saponification Number Analysis

Ketaren (2008) asserts that the saponification number is inversely correlated with the oil's molecular weight; the lower the oil's molecular weight, and vice versa, the higher the oil's molecular weight, the lower the saponification number. In this study, the saponification number was less significant and stood at 181.55 mg KOH/g.

In contrast to the findings of this study, which produced a saponification number value of 184.45 mg KOH/g, which is in accordance with the SNI standard for candlenut seed oil, Chynintya and Paramita (2016)'s research produced a higher value using the screw press method at a temperature of 70oC and 90 min of preheating. When considering the cooking time factor, Yusnita et al. (2001) found that the tendency to reduce the saponification number appeared to be greater the longer the cooking time. This is related to the chemical processes that take place, such as oxidation and polymerization, which can lead to the breakdown of fatty acids and produce compounds that are more complex and have a high molecular weight. Additionally, short-chain fatty acids are thought to evaporate as a contributing factor. The level of oil degradation can be determined by the high value of the saponification number.

Free Fatty Acid Analysis

When compared to oil in previous studies, the value of free fatty acids in this study is higher, at 2.4%

bb/b. This difference in value may be due to the operating conditions. With an extraction temperature of 80oC and a maceration extraction method using n-hexane solvent for 60 min, Nababan et al., (2018) were able to produce free fatty acid content of 1.03%, which satisfies the SNI quality standard for candlenut seed oil with a maximum value of 1.50%.

In the meantime, the oil used in this study is repeatedly heated, specifically during the distillation and oven processes, to get rid of solvents that can make oil degrade. Additionally, the oil from this study underwent a storage process because it wasn't immediately analyzed, which led to physical and chemical changes in the oil that could have been brought on by oxidation or hydrolysis. It can be inferred that the free fatty acids in candlenut seed oil also rise as the heating temperature does. It is believed that the oil present in candlenuts undergoes an oxidation reaction when heat is applied.

CONCLUSION

Through the optimization process, which involved varying the extraction time of candlenut seed oil, a significant correlation was identified between the extraction time (T) and the resulting oil yield. This correlation was determined through eight repetitions of the research, utilizing a faster method for calculating effects and interactions, as well as a factorial experimental design at level 2^3. With a seed size of 20 mesh, a material to solvent ratio of 1:6 g/g (50 g in 500 mL), and an optimal extraction time of 185 min, a yield of 43.2% was achieved in this study.

In accordance with SNI 01-4462-1998 for candlenut seed oil, an analysis of candlenut seed oil at an extraction time of 185 min yields a refractive index value of 1.4736, a moisture content of 0.08% bb/b, a viscosity of 15.47 Cp, and a density of 0.869 gr/mL. Additionally, the analysis of the free fatty acid number is 2.4%bb/b, and the number for saponification is 181.55 mg KOH/gr, which is not in accordance with SNI 01-4462-1998 candlenut seed oil.

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