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Optimization Extraction of Sunflower Seed Oil (*Helianthus Annus)* Using Factorial Design Experiment with Soxhlation Method

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Abstract – *Sunflower seed oil is one type of vegetable oil whose growth is still in its early stages in Indonesia. Due to a shortage of domestic availability, several edible oil enterprises in Indonesia still need to import a significant amount of sunflower seed oil. Rukmana (2004) states that the content of sunflower oil's content ranges from 23 to 45%. 11.7% oleic acid and 44-72% linoleic acid are both present in sunflower oil. The goal ofthis study was to identify the best process factors in light of the obtained oil yield. This study's used Fractional Design Experiment 2^3 with Quicker Method analysis. The results showed that the optimal operating conditions were an extraction time of 185 min, temperature of 60 ℃, and material-solvent ratio of (R) of 1:6, yielding an oil of 51.6%.*

Keywords: *sunflower, seed oil, composition, extraction*
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INTRODUCTION

Helianthus annuus, a species of sunflower plant that belongs to the Asteraceae family, is a native of Mexico and Peru in Latin America. Since 1970, sunflower research has been conducted in Indonesia. In addition to being attractive plants, sunflower plants now have a few other benefits. Sunflower seeds can be used as a raw material in the food industry to create a variety of products, including kwaci. Sunflower seeds can also be refined to make edible oil, which is very importantin the petroleum sector (Atjung, 1981). Sunflower seeds are dark green or grayish green in color (Anggraini, 2015).

Sunflower seed oil one type of vegetable oil that hasn't developed much in Indonesia (Guenther, 1990). Sunflower seeds typically contain 40%–45% oil, 14–18% protein, and 25%–30% skin, the majority of which is made up of inedible crude fiber, according to Le Clef & Kemper (2015). According to Rukmana (2004), sunflower seed oil contains 11.7% oleic acid and 44-72% linoleic acid. Furthermore, according to Rukmana, sunflower seed oil serves as acooking oil, a component of margarine, a raw material for the creation of cosmetics, and a source of protein at a concentration of 13–20%.

The extraction-destillation procedure utilizing ethanol and n-hexane solvents was used to produce sunflower seed oil in the research conducted by Pardian et al. (2017).

The experiment used ingredients that weighed 200 gr, 250 gr, and 300 gr. At ethanol concentrations of 75%, 85%, and 95%, the oil was extracted using the n-hexane solvent for 60, 90, 120, 150, and 180 min. The ideal results were obtained after 180 min and 200 gr of material. The research discovered that n-hexane out-performed ethanol in the extraction of sunflower seed oil.

Research on the quality differences between extracted and commercial sunflower seed oil was done by Katja (2012). Three experiments were performed using Soxhlet extraction with petroleum ether solvent over a four-hour period to extract sunflower seed oil. In this study, sunflower seed oil was extracted using

samples with an average weight of 43.28 gr. These samples had an average oil content of 34.45% and an average aggressive oil yield of 14.06 gr. To assess the quality of the oil, Katja (2012) measured its water content, free fatty acid content, and peroxide numbers, which were respectively 0.43%, 0.47%, and 5.22% in sunflower.

Seed extracted oil and 0.21%, 28%, and 4.89% in commercial sunflower seed oil. Using gas chromatography, Katja's (2012) research was able to confirm the fatty acid composition of sunflower seed oil. Linoleic acid was found to have the highest content in both the extracted oil and the commercial oil, with the extracted oil having a linoleic acid content of 36.96% and the commercial oil having a linoleic acid content of 67.86%. To determine the most important process variables and interaction variables in the extraction process and reach the ideal state, this research aims to optimize crude edible oil from sunflower seeds using factorial design experiment 2^3.

METHODOLOGY

Materials and Tools

The extraction of sunflower seed oil uses distilled water, n-hexane, and sunflower seeds as raw materials.

Figure 1. Soxhlation apparatus series

Chemicals were purchased at Multikimia Raya Store in Semarang. Soxhlet apparatus set was used in the extraction process. Figure 1 shows various soxhlation tools.

Fixed Variable Extraction

Solvent: n-Hexane 99%

Fixed Variable of Solvent Evaporation

Oven Temperature : 70℃
Distillation Temperature : 70℃
Time of Frying : 60 min
Time of Distillation : 60 min

Variable Changed

Extraction Time (T) :190 min (+) 170 min (-)
Extraction Temperature (S) :65℃ (+), 55℃ (-)
Ratio of material and solvent (R) :1 : 8 g/g (+)
1 : 4 g/g (-)

Extraction Procedure

To conduct this experiment, n- hexane was used as the solvent for soxhletation extraction. A 2^3-level factorial design experiment was used to process this research under various extraction conditions. This experiment's goal was to identify the effects of the process variables used and to obtain an accurate optimal state by considering some of their interactions. Three process variables were used in this study: the weight ratio of the materials to the mass ratio of the sample and solvent (R), the extraction time (T), and the temperature (S).

RESULTS AND DISCUSSION

The experimental results of this study were processed using a factorial design, with up to 8 repetitions, as shown in Table 1. The lower level's variable extraction time (-) 170 min results in a lower oil yield value than the upper level's (+) 190 min. The oil yield has a value that corresponds to the sign of the variable level of the extraction process duration, as demonstrated in Table 1's findings of data processing with a factorial experimental design. To maximize this research using the Quicker Method, it is essential to be aware of the important factors and concentrate on computing the primary effects and their interactions on the yield generated.

Table 1. Extraction Result of Sunflower Seed Crude Oil

Run	Variable Changes		Interaction					Yield (%)	Refractive Index	Density	Viscosity
	R	T	S	RT	RS	TS	RTS				
1	-	-	-	+	+	+	-	30.50	1.391	0.912	53.39
2	+	-	-	-	-	+	+	32.00	1.406	0.913	55.45
3	-	+	-	-	+	-	+	36.33	1.462	0.919	53.96
4	+	+	-	+	-	-	-	41.00	1.413	0.912	56.97
5	-	-	+	+	-	-	+	31.16	1.375	0.881	55.95
6	+	-	+	-	+	-	-	31.83	1.384	0.910	56.21
7	-	+	+	-	-	+	-	34.00	1.390	0.890	56.39
8	+	+	+	+	+	+	+	44.33	1.468	0.912	57.02

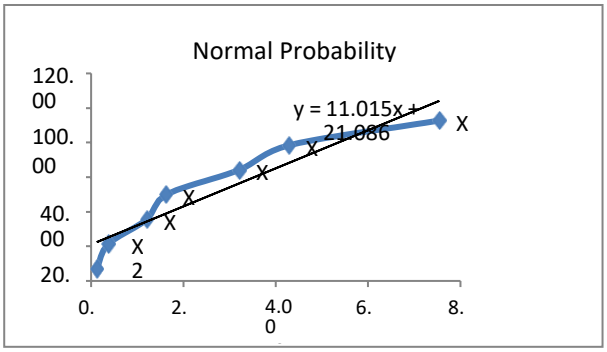

Figure 2. Normal Probability Plot for 2^3

A normal probability curve between the P-value and the result, with an R^2 value of 0.8835, is shown in Figure 2. According to Figure 2, 88.35% of the independent factors have the potential to affect the dependent variable. As a result, the extraction time is determined to be the factor that has the greatest impact on the yield of sunflower seed oil during production. Sunflower seed oil yield is also influenced by the interaction between the solvent-to-material ratio and the extraction time. With the help of this research, we can immediately improve the procedure by adjusting the extraction time (T) to get the best sunflower seed oil output.

Oil extraction optimization results of the oil yield are shown in Table 2 and show that it keeps rising during extractions that last between 170 and 185 min. The oil yield value decreases at an extraction time of 190 min. The graph of sunflower seed oil extractionoptimization in Figure 4 illustrates this issue.

Table 2. Optimization Results of Sunflower Seed Oil Crude Extraction

Time (minute)	Temperature (°C)	Ratio solute:solvent (g/g)	Yield (%)
170			31
175			33,8
180	60	1:6	39
185			51,6
190			50

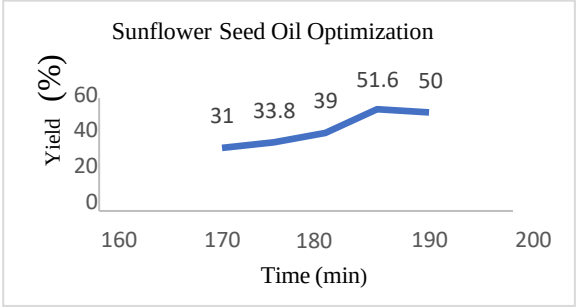

Figure 3. Sunflower Seed Oil Optimization

According to the method, the best oil yield was produced at 185 min at 51.6% extraction time, with other ideal operating conditions being 60°C for the operating temperature and 1:6 for the ratio of material mass to solvent mass. A higher oil yield will be obtained from a longer extraction time (T), which will facilitate the interaction of the solvent with the sample. As extraction time increases, sunflower seed oil's solubility gradually decreases. Due to continuous circulation (reflux), optimal extraction results will typically be obtained with longer extraction times. This is only true for a short period of time, similar to the yield in the fifth experiment, because once the oil content in the sample has been extracted, further extraction does notaffect the extraction outcomes.

Analysis of the Results of Crude Sunflower Seed Oil Extraction

Moisture Content Analysis

Analysis of the water content obtained results of 0.28%. This result has met the SNI 01-3720-1995, which is a maximum of 0.3%. When compared to the water content found in sunflower seed oil from earlier research conducted by Katja (2012), it can be inferred from these results that the water content in sunflower seed oil from this extraction is quite good.

The water content discovered in this researched can be said to be in accordance with the statement put forward by Winarno (1988) that the higher the temperature and the longer the heating, thefaster the evaporation will occur, so that the water contained in the material is lower. The sample in this research was subjected to a further heating process, namely the oven process, to remove the solvent and water that was included in the sample so that a low water content was obtained.

Free Fatty Acid Analysis

The free fatty acid content test results in this study were 0.4% b/b, which did not meet the SNI standards. A free fatty acid content of 0.47% b/b was obtained in Katja's (2012) research through extraction by soxhlation using petrolium ether solvent and an extraction time of 4 hours, though these outcomes also fell short of SNI requirements. Given the lengthy period of storage before analysis, which increased theoil's water content, it is predicted that the high concentration of free fatty acids in the oil is the resultof this. Oils and fats can be converted into free fatty acids and glycerol when there is water present (Ketaren, 1986).

Saponification Number Analysis

The saponification number test results of this experiment amounted to 175.31 mg KOH/gr which

is lower than the results of previous research by Simanjuntak (2015) of 189.50 and still below the SNI standard for sunflower seed oil. The high content of free fatty acids in the oil, which causes the high content of unsaturated fatty acids in the test sample, is assumed to be the cause of the low value of the saponification number. Harun, N. (2006) found that oils rich in unsaturated fatty acids have a high saponification number.

The sample's unsaturated fatty acids lead to the formation of carbonyl groups, which can eventually react with alkali, by oxidizing the unsaturated bonds in the unsaturated bonds. The amount of saponification depends on the molecular weight of the oil, according to Ketaren (1986). The resulting saponification number value for oils with a high molecular weight will be lower. On the other hand, oil with a lower molecular weight will have a higher saponification factor.

Viscosity, Density, and Refractive Index Analysis

Viscosity, density, and refractive index testing results were obtained at 56.42 cSt, 0.922 g/cm^3, and 1.461, respectively, at the 180-minute time variable. The viscosity, density, and refractive index values in earlier research by Sutrisno et al. (2021) were 57.30 cSt, 0.94 g/cm^3, and 1.46, respectively. This value, as determined by both prior research and the author's own research, is in accordance with the Gulf Standard, which was carried out by the G.C.C. Standardization Organization (GSO 1929/2012), regarding the physical and chemical characteristics of edible vegetable oils. Sunflower seed oil has a density of 0.918–0.923 g/mL and a refractive index of 1.461–1.468.

CONCLUSION

Based on the results of processing and discussion, it is concluded that the use of the factorial design method with two levels on three process variables with the quicker method in this study obtained a yield value at 185 min, which is the optimum yield of 51.6% with an operating temperature of 60℃ and a ratio of material mass to solvent mass of 1:6. When the chemical properties of the optimized oil were tested after 185 min, the results showed that the free fatty acid value was 0.4% b/b, which is below the SNI standard for sunflower seed oil, and that the saponification number was175.31 mg KOH/gr, which is also below the SNI standard for sunflower seed oil. As for the percentageof water content, the result is 0.28%.

In order to obtain a better analysis of sunflower seed oil, it is necessary to pay attention to the oil storage container and oil storage time, therefore, further research is needed to obtain the results of FFA% and saponification number to reach the SNI 01-3720-1995 for sunflower seed oil.

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