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Extraction of Crude Sunflower Seed Oil as a Vegetable Oil Rich in Antioxidant using the Microwave Assisted Extraction (MAE) Method

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Abstract - *Oil is a basic necessity in every county, palm oil is an oil that is in high demand so the palm oil industry has an impact on world deforestation. One raw material that can be substituted is sunflower seeds, with a high antioxidant content obtained from vitamin E and selenium, amount 41.08 mg/100 gr. So we eill get a sunflower seeds oil product that is rich in antioxidants, which is beneficial for health. Extraction of sunflower seed oil was carried out using the Microwave Assisted Extraction (MAE) extraction method with N-Hexane solvent. This study will use the factorial design level 2^3 method to determine the most influential process variables to produce optimum operating conditions in making sunflower seed oil with Quicker Method analysis where the changing process. The variables extraction are temperature (T), time (W), and solvent volume (V), and the most influential process variables were obtained, amount extraction time of 9 minutes, temperature of 60°C, and solvent volume of 500 mL producing an oil yield of 53.6%, viscosity of 53.76 cSt, density of 0.919 g/cm³, water content of 0.23% b/b, FFA of 0.32% w/w, and IC50 value of 87.613 is classified as a strong antioxidant.*

Keywords *– Oil, Sunflower seed, Microwave assisted extraction, Antioxidant activity, Factorial design*

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1. Introduction

The palm oil industry was built to meet consumer needs, so palm oil raw materials is increasing in demand and resulting in deforestation. Another alternative to stopping deforestation is to replace the raw materials for making oil. There are four main vegetable oil crops in the world [1]. Palm oil, soybeans, rapeseed and sunflowers can supply around 85-90% of the world's vegetable oil.

Sunflowers with the latin name (Helianthus annuus) are in the Asteraceae family, have the flower part, namely the kernel or ovule, which contains lots of oil, phytochemicals, protein, and low crude fiber which is a source of zinc, copper, phosphorus, calcium, selenium, vitamin E. and B complex, and also antioxidant activity [2]. And the antioxidant content is obtained from vitamin E and selenium, for vitamin E it is found to be 41.08 mg/100 gr. Compared with soybean seeds and sesame seeds, the vitamin E content is only 3 mg/100 gr and compared to nuts, only 10.1 mg/100 gr [3].

Apart from containing high antioxidant activity, sunflower seed oil also contains more unsaturated fatty acids which can reduce cholesterol levels in the body. And with a high oleic content it is also very effective in preventing cardiovascular disease [4]. So sunflower seed oil is a healthy and good vegetable oil for consumption. However, previous research in making sunflower seed oil still uses an conventional extraction process which is takes a long time, so in this research used the Microwave Assisted Extraction (MAE) extraction method, which is a very effective and efficient extraction method. MAE it self is an extraction method that uses microwaves to extract compounds in samples, and is considered to have high potential because the extraction process requires less time and less solvent when compared to conventional methods [5]. This research was carried out using N-Hexane solvent, with fixed variables in the form are power of 400 Watts, evaporation time of 10 minutes, and rpm speed for evaporation of 60 rpm. Meanwhile, the independent variables are time (6 minute and 10 minute), temperature (55°C and 65°C) and solvent volume (450 ml and 550 ml). This research purpose to (1) determine the optimum operating conditions to obtain the best sunflower seed oil

(2) determine the extraction variables in the form of time, temperature and solvent volume (3) determine the water content, %FFA, and antioxidant activity values of sunflower seed oil.

2. Materials and Methods

2.1 Materials

The materials used are sunflower seeds, N-Hexane, Methanol, Ethanol, NaOH, Aquadest, PP Indicator, DPPH. The tools used in the research are MAE extraction tools, evaporators, ovens, UV-Vis spectrophotometers, measuring instruments, digital balances, beakers, glass stirrers, watch glasses, measuring flasks, dropper pipettes, volume pipettes, test tubes, pycnometer, erlenmeyer, measuring cup, porcelain cup, thermometer, filter paper, grinder.

2.2 Raw Material Preparation

The pretreatment stage is carried out by preparing 250 grams of sunflower seeds and cleaning them with water and washing them. After that, dry it in the open air without being exposed to sunlight for 2 weeks. If after 2 weeks a water content test is carried out, if the water content is below 10% then the next stage can be carried out, namely grinding and sizing using a 50 mesh sizer. However, if the water content is still above 10% then oven is carried out at a maximum temperature of 40°C.

2.3 Sunflower Seed Oil Extraction

Sunflower seed powder is weighed according to the variable 50 grams using a beaker, then prepare the N-Hexane solvent and mix it in the beaker. If it has been mixed, put it in the MAE, then set the power to 400 watts and the temperature and time according to the variables used. The extraction process was carried out 8 times.

Table 1. Extraction Variables

No.	Variables	Lower Limit (-)	Upper Limit (+)
1.	Time	6 minute	10 minute
2.	Temperature	55°C	65°C
3.	Volume solvent	450 mL	550 mL

Figure 1. Microwave Assisted Extraction equipment circuit

2.4 Evaporation

After the extraction process is carried out, evaporation is carried out to separate the solvent contained in sunflower seed oil. Evaporation was carried out using an evaporator at a speed of 60 rpm for 10 minutes. The evaporation process is stopped if there is no extract dripping, then the oil obtained is weighed to calculate the % yield.

Figure 2. Evaporation equipment circuit

2.5 Water Content

Water content analysis is based on SNI for oils and fats (SNI 01-3555-1998), sunflower seed oil is analyzed by heating a porcelain cup to a temperature of 105°C for 60 minutes which has been previously washed and then cooled in a desiccator for 30 minutes. Then weigh the porcelain cup and record the weight. Then weigh the oil sample of ± 5 grams in a porcelain cup whose constant weight is known, then put it in the oven at 105°C for 60 minutes then put it in a desiccator for 30 minutes, the procedure is repeated until the weight is stable. Moisture content can be calculated by the formula below:

$$Kadar\ Air(\%) = \frac{(W+W_0)-W_1}{M_0} \quad (1)$$

2.6 Free Fatty Acid Analysis

Analysis of free fatty acid content in sunflower seed oil extract was carried out by using 0.1 N NaOH solution and standardization. After that, weigh the sample as much as 5 grams into erlenmeyer and add 50 mL ethanol PA. Then drop 3-5 drops of phenolphthalein (PP) indicator solution and titrate with 0.1 N NaOH standard solution until the sample turns pink. Then record the volume of NaOH used and the free fatty acid content in the sample is calculated using the following equation:

$$FFA = \frac{mL\ Na0H\ x\ N\ Na0H\ x\ 40}{W}\ x\ 100\% \quad (2)$$

W= Sample weight (g)

2.7 Antioxidant Activity Anlysis

Stages of testing the antioxidant activity of sunflower seed oil using the DPPH method [6]. Antioxidant activity testing was carried out by modifying 1 mL of sunflower seed oil extract solution which was then mixed with 3 mL of DPPH solution which was previously made by reacting 96 mL of methanol with 4 mL of DPPH. The mixture was incubated in the dark for 30 minutes, then the absorbance was measured at a wavelength of 520 nm using a UV-Vis spectrophotometer. In this test, a mixed solution of 1 mL of methanol and 3 mL of DPPH was used as a blank solution. Antioxidant activity is measured using the following equation:

$$\%\ inhibisi = \frac{(A\ blanko - A\ ekstrak)}{A\ blanko} x\ 100\% \qquad (3)$$

3. Results and Discussion

This research on the extraction of crude sunflower seed oil used a factorial design level 2^3 experimental design with different extraction conditions. This experimental design was carried out to determine the effects on the process variables used and the optimum conditions obtained were more precise because they included interaction factors. This research uses three process variables, namely extraction time (W), solvent volume (V), and extraction temperature (T). To carry out optimization in this research, it is necessary to know the process variables that are very influential. These process variables are obtained through quicker method analysis, namely by calculating the main effects and interactions with the resulting yield. The results of calculating influential variables can be seen in table 2, table 3, and figure 3.

Table 2. Results of Main Effect and Interaction Calculation on Yield

Effect	Total
I_1, T	5.39
I_2, W	9.01
I_3, V	0.72
I_{12}, TW	4.27
I_{13}, TV	1.58
I_{23}, WV	0.28
I_{123}, TWV	2.20

Table 2 shows the results that extraction time is the variable that most influences the process of extracting oil from sunflower seeds when viewed from the calculation of the quicker method which produces the largest number of effects.

Table 1. Determination of Influential Variables

%P	Effect
7.14	0.28
21.43	0.72
35.71	1.58
50.00	2.20
64.29	4.27
78.57	5.39
92.86	9.01

From the results of Table 3, it shows that yield has the most significant influence, namely with a value of 9.01

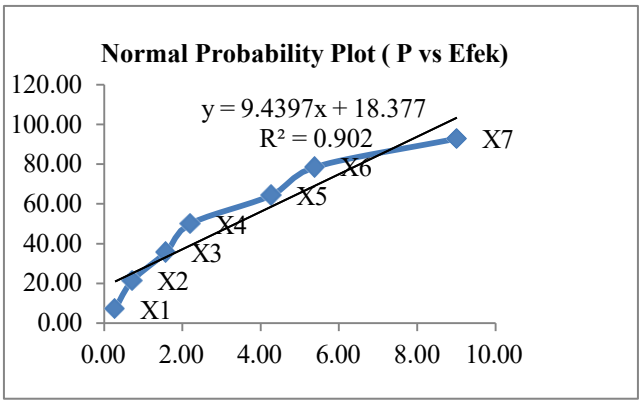

Figure 1. *Normal Probability Plot (P vs Effect)*

Meanwhile, Figure 3 shows that x7, the point resulting from calculating the effect of extraction time with a percent probability is away from density. From the results of this analysis, process optimization can be directly carried out by varying the extraction time variable (W) to determine the yield of sunflower seed oil.

3.1 Optimization of Crude Sunflower Seed Oil Extraction

From the variance analysis that has been carried out, it can be seen that the process variable that influences the sunflower seed oil extraction research process is extraction time, so that for this optimization process the variables T (temperature) and V (solvent volume) become fixed variables and the variable W (extraction time).) becomes a changing variable. The results of the optimization that has been carried out can be seen in Table 4.

Table 4 shows that the extraction time of 6 minutes to 10 minutes produces an increasing oil yield and decreases in yield at an extraction time of 10 minutes. A graphic depiction of the results of optimization of sunflower seed oil extraction can be seen in Figure 4.

Table 4. Sunflower Seed Oil Yield in the Optimization Process

Extraction Time (minute)	Temperature (°C)	Solvent Volume (mL)	Yield (%)
6	60	500	32.62
7			35.2
8			41.37
9			53.21
10			51

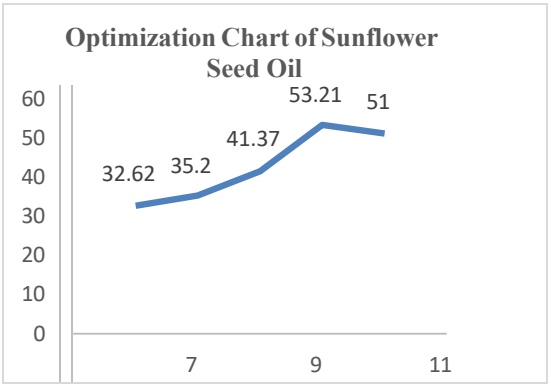

Figure 4. Optimization Chart of Sunflower Seed Oil

Based on the oil yield produced as presented in graph Figure 4, it can be seen that an extraction time (W) of 9 minutes is the best result with a yield of 53.21% with other optimum operating conditions being 60oC for operating temperature and 500 mL solvent volume. The longer extraction time will make it easier for solvent penetration in the sample. The solubility of sunflower oil occurs slowly in proportion to the length of extraction time. In general, the longer the extraction time, the more optimal the extraction results obtained because the more frequent solvent circulation (reflux) occurs. However, this only lasts for a certain time, because once the oil in the sample has been completely extracted and the solvent is in a saturated state, the next extraction process no longer affects the extraction results and is better stopped.

3.2 Water Content Analysis

Water content analysis obtained a result of 0.23%. This result meets SNI standards, namely a maximum of 0.3%. When compared with the water content in sunflower seed oil from previous research conducted [7], it was 0.43%, and research conducted [8] was 0.28%. So from these results it can be concluded that the water content of the extracted sunflower seed oil is quite good.

The results of this water content are in accordance with the statement that the higher the temperature and drying air flow speed, the faster the drying process, the higher the drying air temperature, the greater the mass of liquid that is evaporated from the material. The material's ability to release water from the surface will also cause the temperature to increase and the drying process to take longer [9]. So the water content gets lower.

Table 5. Water Content Optimization Results of Sunflower Seed Oil

Analysis	Research Optimization	Previous Research	Water Content (%)
	0.23%	SNI	0.3%
		Quality of Commercial Sunflower Oil and Oil Extracted from Sunflower Seeds (Helianthus annuus L.)	0.43%
		Optimization Extraction of Sunflower Seed Oil (Helianthus Annus) Using Factorial Design Experiment with Soxhlation Method.	0.28%

3.3 Free Fatty Acid Analysis

The results for free fatty acid levels in this study were 0.32% w/w, which did not meet the SNI standard, namely 0.3% w/w. The free fatty acid content obtained in previous research conducted [7] was 0.47% w/w, and carried out by [8] was 4% w/w. However, these results still do not comply with SNI requirements. This could be caused by the storage time before analysis being long enough, so that the water content in the oil could increase again and is also thought to be caused by the high concentration of free fatty acids in the oil. Differences in results can be caused by the type of extraction carried out and also the solvent used. In the two previous studies, soxhletation extraction was used, where the results were 0.47% w/w using petroleum ether solvent for a time of 4 hours, while the results of 0.4% w/w used N-Hexane solvent for a time of 3 hours.

Table 6. Optimization Results of Free Fatty Acid of Sunflower Seed Oil

Analysis	Research Optimization	Previous Research	Water Content (%)
	0,32%	SNI	0,3%
		Quality of Commercial Sunflower Oil and Oil Extracted from Sunflower Seeds (Helianthus annuus L.)	0,47%
		Optimization Extraction of Sunflower Seed Oil (Helianthus Annus) Using Factorial Design Experiment with Soxhlation Method.	0,4%

3.4 Antioxidant Activity Analysis

From the graph we get the equation y = 8.6174x – 11.455, and the R2 value = 0.9914, the results of antioxidant activity testing show an IC50 value of 87.613 µg/mL which is obtained from the formula x = (y-b)/a, a value = 8, 6174 and the value b = 11.455 which will then be calculated to get the IC50 value. And the IC50 value < 50 µg/mL the antioxidant power obtained is very strong, if the IC50 is 50-100 µg/mL the antioxidant power is strong, then when the IC50 is 101-150 µg/mL the power moderate antioxidant and weak IC50 > 150 µg/mL [10]. And it can be concluded that the results obtained in this research on the crude extraction of sunflower seed oil showed that the antioxidants obtained were in the range of 50-100 µg/mL, which means that the antioxidant activity contained in sunflower seed oil is strong.

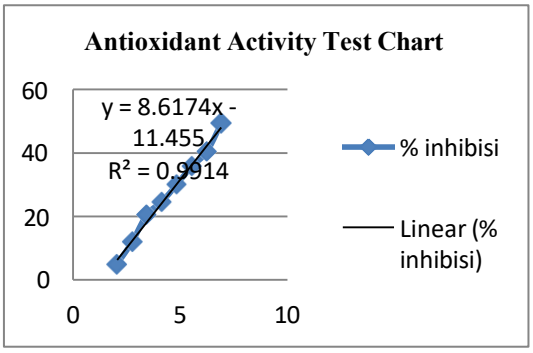

Figure 5. Antioxidant Activity Chart of Sunflower Seed Oil

4. Conclusion

From the results of research on the extraction of crude sunflower seed oil which was carried out using the MAE extraction method using the solvent N-Hexane, , and the fixed variables were extraction power of 400 Watts, evaporation time of 10 minutes at a speed of 60 rpm and evaporation temperature of 68°C. In this research, the three-variable factorial design method was used, and by using a faster method to calculate the main effect and variable interaction effect with a total of 8 experiments and the most influential process variables were obtained, namely time 9 minutes, temperature 60°C, and solvent volume 500 mL effect which is obtained for a yield value of 53.6%, a viscosity of 53.76 cSt, a density of 0.919 g/cm3, a water content of 0.23% w/w, an FFA of 0.32% w/w, and an antioxidant activity value of IC50 amounting to 87.613 µg/mL. All the results were in accordance, but the FFA levels were not suitable, due to the storage mass being long enough to cause the water content to rise again so that the results of the FFA levels were also high.

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