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Optimization of Papaya Seed Oil Production Process (*Carica papaya L.*) with Soxhlation Extraction Method using Factorial Design

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Abstract *– This study aims to optimize the extraction of papaya seed oil (Carica papaya L.) using a factorial design level 2. Papaya seeds are a significant component of the fruit, comprising approximately 14.3% of the total fruit weight. These seeds contain about 25% vegetable oil, predominantly composed of unsaturated fatty acids, which have the potential to be processed into consumable oil. The extraction method employed is Soxhlet extraction using a non-polar solvent, n-hexane. A factorial design level 2^3 was utilized to determine the most influential process variables. The variables were considered to the ratio of papaya seed mass to solvent volume (1:7 and 1:11), particle size (10 and 30 mesh), and extraction time (170 and 190 min). Optimization was conducted using the quicker method calculation, where the determination of the largest effect and the largest main effect played a crucial role. The analysis revealed that the ratio of papaya seed mass to solvent volume had the most significant main effect, with an effect value of 0.1217. From the analysis, it was found that the 1:7 ratio produced the lowest level of Free Fatty Acids (FFA) in the oil, at 0.5076%, which aligns with the Indonesian National Standard (SNI) requirement of 0.36-0.82% FFA content. Furthermore, an analysis of density, viscosity, and moisture content was conducted on the 1:7 ratio. The test results showed that the 1:7 ratio resulted in an oil density of 0.924 g/mL, moisture content of 0.07127%, and FFA content of 0.5076%. However, based on theoretical knowledge, an increase in FFA content leads to a decrease in oil quality. Additionally, higher ratios corresponded to increased moisture content and density. In conclusion, the papaya seed oil produced in this study complies with the Indonesian National Standard (SNI, 01-3555-1998) regarding FFA content (0.36-0.82%), moisture content (maximum 0.15%), and density (0.924-0.929 g/mL).*

Keywords: *carica papaya L.; papaya seed oil; soxhlation extraction; factorial design*

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INTRODUCTION

Papaya (*Carica papaya L.*) is a type of plant that is cultivated in the garden or yard (horticulture), currently papaya is a plant that is quite widely cultivated in Indonesia. The seed content in papaya fruit is about 14.3% of the whole papaya fruit. Simply put, papaya seeds can be utilized as a digestive medicine, skin, and as a source to obtain vegetable oil with a certain fatty acid content (Wulandari, 2017).

In papaya seeds, when calculated by dry weight, papaya seeds contain ±25% oil. When compared to sunflower seeds at 22.23% and soybeans at 19.63%, the oil content of papaya seeds is more so that it has the opportunity to be developed into raw materials for both vegetable oil and alternative fuels (Azhari et al., 2020). On the other hand, papaya seeds contain fatty acids, most of which are unsaturated fatty acids that can produce vegetable oil for public consumption.

Papaya seed oil is known to be yellow in color and contains 71.60% oleic acid. 15.13% palmitic acid; 7.68% linoleic acid; 3.81% citric acid and other fatty acids in relatively small or limited amounts. Besides containing fatty acids, papaya seeds also contain other chemical compounds such as phenols, alkaloids, and saponins (Andaka & Fajrah, 2020). Papaya seed oil is a type of vegetable oil. Vegetable oil is oil obtained from the extraction process of extracts from various parts of plants. Based on its use, vegetable oil is divided into two groups, namely, vegetable oil used in the food industry (edible oil) and in the non-food industry (non-edible) (Andaka & Fajrah, 2020).

Currently, the utilization of papaya seeds is usually only discarded after the fruit is consumed. Some researchers have previously conducted research on the utilization of papaya seeds as a source of vegetable oil. Papaya seeds, which have a clear membrane, if processed for oil will be very profitable. With the increasing demand for oil production, especially in food oil as a substitute for petroleum whose price is getting higher and higher, this is an opportunity to increase papaya seed oil production. Papaya seed oil has a great opportunity to be increased in volume, among others, by improving the production process system. Extraction processes that are usually carried out conventionally have difficulties in determining operating conditions. As a result, oil yield and oil quality are low and it is difficult to produce good quality oil in large quantities (Nofrin et al., 2012). Therefore, an appropriate extraction method is needed to produce good quantity and quality of papaya seed oil.

In this study, the method used was liquid extraction, which is considered one of the most effective and commonly used methods. It involves using a solvent that can directly contact the material and operate continuously, ensuring the solvent used for extraction is always pure. Additionally, the oil within the material can be reused. In the extraction process, commonly used solvents include n-hexane and ethanol. However, previous research has demonstrated that n-hexane solvent can produce oil with better quality and quantity. Generally, the selection of solvents is influenced by factors such as high selectivity, boiling point, polarity, low toxicity, insolubility in air, and inertness, preventing reactions with other substances (Guenther, 1987).

Therefore, it can be concluded that qualitatively, papaya seed oil is better produced using n-hexane solvent as it can yield good oil quality and a large quantity. In this study, innovation was carried out by optimizing the Soxhlet extraction of papaya seed oil using a factorial design experiment with a level 2^3 to determine the most influential process variables, understand the interactions between variables, and determine the optimal results in the extraction process to obtain papaya seed oil.

RESEARCH METHOD

Materials and Tools

The materials used in this study were 280 gr of dried papaya seeds obtained from Banyumanik Market and 4 liters of n-hexane solvent obtained from PT. Multikimia Raya. The equipment used included a Soxhlet extraction apparatus, a distillation apparatus, a chopper, an oven, sieves (10 and 30 mesh), Erlenmeyer flasks, an analytical balance, measuring cylinders, volumetric pipettes, an electric heater, clamps and stands, a magnetic stirrer, measuring flasks, glass funnels, porcelain crucibles, a desiccator, burettes, and filter paper.

Preparation Materials

The research procedure for the material preparation phase in this study referred to a previous study conducted by Zamrodah (2018), following a series of steps. The process started with washing the papaya seeds thoroughly and drying them under sunlight for approximately 1 day. Subsequently, the dried papaya seeds were further dried in an oven at a temperature of 100°C for 1 hour. Once the papaya seeds were dried, they were crushed using a chopper and sieved through 10 and 30 mesh size screens. If there were still papaya seeds that did not meet the 10/30 mesh size requirement, they were crushed again using a chopper and sieved once more.

Extract Papaya Seeds

The procedure for the Soxhlet extraction of papaya seeds in this study referred to Zamrodah's research (2018). It began with the preparation of 35 gr of 10-mesh-sized papaya seeds. The dried papaya seeds were then wrapped in filter paper and placed into the assembled Soxhlet apparatus. Next, 400 mL of n-hexane solvent was added to a three-necked flask. The material was extracted for 170 min at a temperature of 70°C, corresponding to the specified variable. The extracted results were collected in an Erlenmeyer flask and further experiments were conducted for subsequent variables.

Table 1. Research Variables

Variable	Lowe Limit (-)	Upper Limit (+)
Ratio of papaya seed mass to solvent volume	1:7	1:11
Particle Size	10 mesh	30 mesh
Extraction Time	170 min	190 min

Figure 1. Soxhlation Apparatus Tools

Distillation

Separation of the solution is by distillation method. Separation is done to produce a solution that is free of solvents. The purpose of this separation process is to obtain a purer sample to produce vegetable oil. This distillation process refers to previous research, namely Zamrodah (2018) with the stages of the process starting with assembling the distillation device, putting the papaya seed oil that has been extracted into a three-neck flask, then inserting a vacuum pressure source into the channel before the Erlenmeyer, heating at 70℃ for 1 hour, then distillation is stopped when there is no dripping solvent. Weigh and measure the volume of the resulting oil yield.

Figure 2. Set of Distillation Equipment

Viscosity

The way to measure and calculate viscosity is by using an *Ostwald Viscometer*, suction ball, stopwatch, glass funnel and measuring cup. First, put clean water into a measuring cup then put it into the *Ostwald Viscometer* while turning on the stopwatch then when the water reaches the limit mark turn off the stopwatch. Then do this for the papaya seed oil produced. Finally, calculate the viscosity value with the following formula:

$$\eta = \frac{t \times \rho}{t_0 \times \rho_0} \times \eta_0 \qquad (1)$$

Information:

η = Papaya Seed Oil Viscosity (cP)
η_0 = Water Viscosity (cP)
t = Flow Time of Papaya Seed Oil (s)
t_0 = Water Flow Time (s)
ρ = Density of Papaya Seed Oil (gram/mL)
ρ_0 = Density of Water (gram/mL)

Density

Density is calculated to determine the density of the papaya seed oil produced. To know the density, the first step is to clean the pycnometer by rinsing it with acetone and diethyl ether. Dry the empty pycnometer and weigh its mass. Put the sample into the pycnometer until it reaches the mark. Let stand for 30 min then clean the neck of the pycnometer with filter paper and leave at room temperature. Then remove and weigh. Do the procedure with blank water as well. Then to calculate the density:

$$\text{Density } (\rho) = \frac{W_1 - W_0}{\text{Volume of Pycnometer}} \qquad (2)$$

Information

W_1 = Weight of pycnometer with sample (gr)
W_0 = Empty pycnometer weight (gr)

Water Content

Analysis of water content in papaya seed oil by washing the porcelain cup using distilled water, then drying the porcelain cup in the oven at 105°C for ± 1 hour. After that, cool in a desiccator for 30 min, then weigh and record the weight. Do the treatment on the porcelain cup until you gain a constant weight. Then, weigh 5 gr of sample in a porcelain cup and heat it in an oven at 105°C for ± 1 hour. After that, re-cool in a desiccator for 30 min and measure the porcelain cup containing the sample until a constant weight is obtained. Then calculate the water content with the following formula:

$$\text{Water Content} = \frac{w_0 - w_1}{w_0} \times 100\% \qquad (3)$$

Information:

w_0= Weight of the cup with oil before it is in the oven
w_1= Weight of the cup with oil after it's been in the oven

The quality requirement for maximum water content of papaya seed oil is 0.15% w/w.

Free Fatty Acid

Crude analysis of free acids in papaya seed oil can be done by preparing 0.1 N NaOH solution and standardizing it. After that, measure 2-5 gr of sample into an Erlenmeyer and add 50 mL of 95% ethanol. Then, add 3-5 drops of phenolphthalein (PP) indicator and titrate with 0.1 N NaOH standard solution until the color changes to pink. Note the volume of NaOH titration results and finally, calculating the free fatty acid content in the sample using the following formula:

$$\text{FFA Content} = \frac{\text{V NaOH x N NaOH x 282}}{\text{Weight of Papaya Seed Oils}} \times 100\% \qquad (4)$$

Quality requirements for free fatty acids (FFA) of papaya seed oil, namely 0.36-0.82% w/w.

RESULTS AND DISCUSSION

Design Experiment is one form of experiment that can control all external variables that can affect the course of the experiment. In the design experiment, each tested variable is determined at several values, and all possible combinations of variables are tried. This research uses a factorial design method 2^3, where each variable is taken at only 2 levels, and 3 variables are changed, namely the mass ratio to the solvent (1:7 and 1:11), the particle size of the material (10 and 30 mesh), and the extraction time (170 and 190 min). Therefore, 8 treatments are obtained, and their effects will be analyzed. The use of symbols (+) and (-) is used to indicate the upper and lower limits of each variable in each experiment. The quality of papaya seed oil produced will be compared with the Indonesian National Standard (SNI) 01-3555-1998 and previous research conducted by Cahyaningtyas et al. (2017).

Table 2. Data on Papaya Seed Oil Extraction Results (Factorial Design Level 2^3)

Run	Variables Changed			Interaction				Yields	Density	Viscosity	FFA Content	Water Content
	Q	**S**	**R**	**TS**	**TR**	**SR**	**TSR**					
1	-	-	-	+	+	+	-	39.57	0.927	2.58	0.564	0.056
2	+	-	-	-	-	+	+	57.40	0.924	3.75	0.6768	0.045
3	-	+	-	-	+	-	+	49.51	0.929	1.81	0.564	0.069
4	+	+	-	+	-	-	-	48.26	0.924	5.10	0.6768	0.044
5	-	-	+	+	-	-	+	41.63	0.928	4.24	0.7332	0.046
6	+	-	+	-	+	-	-	59.11	0.925	2.90	0.7332	0.053
7	-	+	+	-	-	+	-	67.34	0.928	1.68	0.7896	0.059
8	+	+	+	+	+	+	+	43.17	0.928	2.18	0.7332	0.053

This experiment was conducted with 8 running, with each run having a different treatment. Table 2 shows that the FFA content of the oil produced has a value that corresponds to the sign of the level of process variables, namely the ratio of materials and solvents where the large ratio or marked (+) has a high FFA content while the small ratio or marked (-) has a smaller FFA content than the large ratio. To optimize this research, it is necessary to know the most influential process variables in the research by means of the quicker method, namely by taking into account the main effect and interaction on the resulting FFA content. The calculation results for the most influential variables are shown in tables 3 and 4 and figure 3.

Table 3. Main Effect and Interaction Calculation Results for FFA Levels

Effect	Amount
I_1, T	0.0421
I_2, S	0.0143
I_3, R	0.1217
I_{12}, TS	-0.0139
I_{13}, TR	-0.0703
I_{23}, SR	0.0139
I_{123}, TSR	-0.0143

Table 3 shows that from the result of the three variables studied, the ratio is the most influential variable in the process of oil extraction from dried papaya seeds. When viewed from the calculation of a faster method that produces the greatest amount of influence.

Table 4. Determination of Influential Variables

P (%)	Effect
7.14	-0.0703
21.43	-0.0143
35.71	-0.0139
50.00	0.0139
64,29	0.0143
78.57	0.0421
92.86	0.1271

Table 4 shows the determination of the influential variable at 0.1271 which indicates the most influential variable is the ratio of ingredients and solvents.

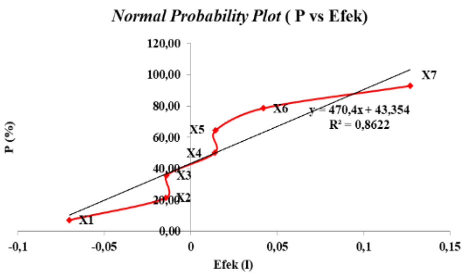

Figure 3. Normal Probability Plot for 2^3

In Figure 3 shows that X7 is the result of the calculation of the effect of extraction time with a percent probability of moving away from density. From the results of this analysis, process optimization was carried out by varying the ratio of materials and solvents (R) to determine the FFA content of papaya seed oil and tested for the best results and then compared with previous research by Cahyaningtyas et al. (2017), Wulandari (2017) and the Indonesian National Standard (SNI).

Optimization of Papaya Seed Oil Extraction

From the analysis of the table above, it can be concluded that the most influential process variable in this study is the ratio of materials and solvents, so the optimization process uses the time variable (T) and particle size (S) as fixed variables and the variable ratio of materials and solvents (R) as a change's variable. The results of the optimization carried out can be seen in table 5.

Table 5. Optimization Results of Papaya Seed Oil Extraction

Ratio of Material to Solvent (g/g)	Seed Size (*mesh*)	Extraction Time (Minute)	FFA content (%)
1:7	20	180	0.5076
1:8			0.564
1:9			0.6204
1:10			0.6768
1:11			0.6204

Table 5 shows that the bigger the ratio, the higher the FFA content. However, based on theory, the higher the FFA content, the lower the oil quality. We can see this in the optimization graph of papaya seed oil extraction in figure 4.

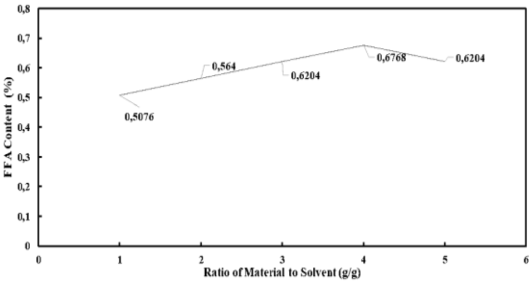

Figure 4. Graph of Optimization of Papaya Seed Oil Extraction Results

Based on the FFA (Free Fatty Acid) content of the papaya seed oil produced, it can be observed that the higher the ratio of material to solvent, the higher the resulting FFA content. This can be seen in Table 5 and the graph in Figure 4, where the 1:10 material ratio shows the highest FFA content, which is 0.6768, under the optimum operating conditions of 20 mesh seed size and 180 min of extraction time. This is because using a larger amount of solvent on the dry papaya seeds will yield a higher yield and can affect the FFA value, as shown in the graph above, where a higher material-to-solvent ratio corresponds to a higher FFA content. Increasing the volume of solvent used enhances its ability to extract the oil contained in the papaya seeds. Additionally, a larger volume of solvent also prolongs the extraction time between the material and solvent. Thus, it can be concluded that an increasing material ratio leads to a higher FFA value, as evident in Table 5 and Figure 4, except for the fifth experiment where a decrease in FFA occurred as the oil in the sample was fully extracted. Moving forward, the best research result was obtained from the 1:7 material ratio, which had the lowest FFA content of 0.5076%. This result will be further analyzed for density, viscosity, and water content and then compared with previous research by Cahyaningtyas et al. (2017), Wulandari (2017) and the Indonesian National Standard (SNI).

Analysis of Papaya Seed Oil Extraction Results

Analysis of papaya seed oil extraction results will be tested based on SNI standards for papaya seed oil as vegetable oil which includes density, moisture content and FFA content. This analysis is carried out on the optimization results that have the lowest FFA content, because the lower the FFA content, the higher the quality of papaya seed oil.

Figure 5. Samples of Papaya Seed Oil

Density

Density is a parameter used to determine the components contained in oil. The more components contained in the oil, the higher the weight fraction. This means that the specific gravity of the oil will be greater. This specific gravity test is useful for determining oil purity. From the observation of the specific gravity of oil extracted from dried papaya seeds, it showed in table 6 with the research oil, oil from previous research and SNI papaya seed oil as a comparison.

Table 6. Density Result of Papaya Seed Extract Oil

Testing	Soxhletation Extracted Oil	Oil Results of Previous Research	SNI Papaya Seed Oil
Density	0.924	0.910	0.924-0.929

Based on table 6, it can be concluded that the specific gravity value produced is 0.924 which meets the requirements, namely in accordance with SNI, which is between 0.924-0.929 % and also based on the research of Cahyaningtyas, et al. (2017) which is close to the specific gravity value of 0.910 gr/mL.

Water Content

The results of the observation of the moisture content of the oil extracted from dried papaya seeds can be seen in table 7 with the research oil, the oil from previous research and SNI papaya seed oil as a comparison. Moisture content in oil is one of the parameters that determine the quality of oil. Where, the higher the water content, the lower the quality of the oil because water is one of the catalysts for hydrolysis in oil. We can see in table 7 that the water content of the research results is in accordance with the maximum limit of the SNI standard for papaya seed oil and in previous research conducted by Cahyaningtyas, et al. (2017) produced high water content and exceeded the SNI value limit of 1.06%. In this study, the water content can be influenced by the amount of heating carried out starting from the extraction process to the distillation process. However, this can make the oil easily damaged and the water content in the sample is carried away and can also cause the water content to decrease. This is in accordance with the theory of Swern (1982) that a material with high water content will produce oil with high water content as well, so it will be easily hydrolyzed. This is also the same as that expressed by Winarno (1988), the higher the temperature and the longer the heating, the faster the evaporation will occur, so the water in the material will be lower.

Table 7. Results of Water Content from Papaya Seed Extract

Testing	Soxhlation Extracted Oil	Oil Results of Previous Research	SNI Papaya Seed Oil
Water Content (%)	0.07127	1.06	max. 0.15

Free Fatty Acid

The results of the observation of free fatty acids contained in papaya seed oil extracted are shown in table 8 with the research oil and SNI papaya seed oil

Table 8. Results of Oil-Free Fatty Acid Test from Papaya Seed Extract as a comparison.

Testing	Soxhlation Extracted Oil	Oil Results of Previous Research	SNI Papaya Seed Oil
Free Fatty Acids (FFA) (% w/w)	0.5076	0.3762	0.36-0.82

Free fatty acid testing is an important parameter for determining oil quality. The amount of free fatty acids in the oil is indicated by the acid number value. A high acid number indicates that the free fatty acids contained in vegetable oil are also high so that the quality of the oil will be lower, this is because the free fatty acids produced due to hydrolysis can reduce oil quality. Free fatty acids can increase due to repeated heating even at a constant temperature, when viewed in table 8 the oil from the results of this study is in accordance with SNI standards where the resulting fatty acid content is 0.5076, in research conducted by Wulandari (2017) produced FFA levels of 0.3762 and from both studies the papaya seed oil produced is in accordance with SNI which is between 0.36 - 0.82.

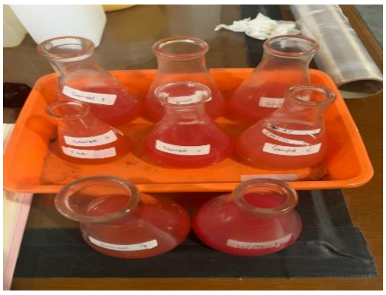

Figure 6. Titration Results of Papaya Seed Oil for Determination of FFA Levels

The results of FFA content in this study were carried out under operating conditions with an extraction time of 180 min, a distillation time of 1 hour with a particle size of 20 mesh and a ratio of material and solvent 1: 7, namely 400 ml of solvent and 35 gr of dried papaya seed material.

CONCLUSION

From the research results, data processing was carried out using a factorial design 23 and then optimization was performed. The optimization was done using the quicker method calculation, where the determination of the largest effect and the largest main effect were determining factors. The analysis results showed that the variable of papaya seed mass to solvent volume ratio is an influential process variable with the largest main effect, with an effect value of 0.1217.

Based on these results, additional optimization was conducted by varying the ratio to 1:7, 1:8, 1:9, 1:10, and 1:11. From this analysis, it was found that the 1:7 ratio variable resulted in oil with the lowest Free Fatty Acid (FFA) content, which is 0.5076%, in accordance with the SNI (0.36-0.82) standard. Furthermore, from the 1:7 ratio variable, density, viscosity, and water content analysis were conducted. The test results showed that the 1:7 ratio produced oil with a density of 0.924 g/mL, water content of 0.07127%, and FFA content of 0.5076%.

The research results indicated that as the ratio of papaya seed mass to solvent volume increased, the FFA content tended to increase. However, according to theory, an increase in FFA content will decrease the oil quality. Additionally, higher ratios resulted in higher water content and density. In conclusion, the papaya seed oil produced in this research meets the SNI (01-3555-1998) standard with FFA content (0.36-0.82), water content (maximum 0.15), and density (0.924-0.929).

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