Journal of Vocational Studies on Applied Research

# Optimization of Soxhlet Extraction Papaya Seed Oil (*Carica papaya L.*) with Petroleum Ether

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***Abstract** – Papaya seed oil is a high source of fatty acids, especially oleic acid, and palmitic acid. It has 71.60% oleic acid, 15.13% palmitic acid, and has a low cholesterol content so it can be useful as a food oil. This study aims to determine the effect of the ratio of ingredients, time, and particle size on the maximum extraction of papaya seed oil. Extraction of papaya seed oil was carried out by the soxhletation extraction method using petroleum ether solvent. The factorial experimental design of $2^3$ was used to determine the significant parameters for the resulting papaya seed oil: yield, density, fatty acid content, viscosity, and water content. The most influential process variable is particle size. The most optimal papaya seed oil extraction results were obtained at a particle size of 20 mesh, an extraction time of 180 minutes, and a ratio of ingredients to the dissolution of 1:9 (35 gram500 mL). That value obtains a yield of 57.029%.*

**Keywords:** *papaya seed oil; factorial design; extraction; optimization*

Received: February 01, 2023 Revised: June 05, 2023 Accepted: June 12, 2023

**Doi**: http://dx.doi.org/10.14710/jvsar.v5i1.17338
[How to cite this article: Andriana, R., and Broto, R.T.D.W. (2023). Optimization of Soxhlet Extraction Papaya Seed Oil (*Carica papaya L.*) with Petroleum Ether. *Journal of Vocational Studies on Applied Research, 5*(1), 17-22. doi: http://dx.doi.org/10.14710/jvsar.v5i1.17338]

## INTRODUCTION

Papaya (*Carica papaya L.*) is a fruit plant that has spread evenly in Indonesia. Papaya is a plant from the Caricaceae family. Papaya is a plant that spreads in Indonesia around the 17th century. The origin of this plant is Mexico and South America, then spread to the continents of Africa, Asia, and India. Papaya can be classified as a herbal plant that has a hollow stem and a tree height of up to 10 m (Setiaji et al., 2009).

Papaya seed oil is known to have a yellow color with a content of 71.60% oleic acid, 15.13% palmitic acid, 7.68% 3.60% linoleic acid, and contains other fatty acids that have certain levels of content (Warisno, 2003). The fatty acid content in papaya seeds varies depending on the type of fruit, which ranges from 25.41%-34.65%. The oil content in papaya seeds is relatively large when compared to sunflower seeds 22.23%, and coconut 54.74% (Sammarphet, 2006). Based on previous research, the most widely used extraction method is the soxhletation extraction method. In this study the method used is the soxhletation extraction method, this method is considered one of the most efficient methods by using a solvent that can be in direct contact with the material and runs continuously so that the solvent used to extract the material is always pure and the oil in the material can be reused.

In a study (Susilowati, N. and Primaswari, R., 2012) entitled extraction of candlenut seed oil (*Aleurites moluccana*) through extraction using a soxhlet, the research objective was to find out the most appropriate solvent for extracting oil from candlenut seeds using a soxhlet, where the solvent used is between others n-hexane, petroleum ether, and ethanol. In this study, the weight of candlenut seeds was used, namely 50 grams, and the weight of each solvent, namely 500 grams. The yield results were 38.72% n-hexane, 33.24% petroleum ether, and 18.36% ethanol. From these results, the best solvent for extracting oil from candlenut seeds is n-hexane, then petroleum ether. Whereas research (Achtami, 2017), aims to characterize the chemical components of papaya seeds using ingredients, namely papaya seed powder, alcohol, and distilled water. In this study, the solid-liquid method was used, and the results obtained were that papaya seeds contain FFA which can be used as a source of vegetable oil and cosmetic oil. The dominant fatty acids are stearic acid (saturated fatty acid) which is equal to 18.42%, and lauric acid (saturated fat) 11.58%. In a study (Soetjipto et al, 2018) entitled fatty acid profile and characterization of pumpkin seed oil (*Cucurbita moschata D.*), two methods were extracted for pumpkin seeds, namely the maceration method and the continuous method using soxhlet. In this study, the results obtained were that the maceration method obtained an oil yield of 32.54%, while the continuous or soxhlet method obtained an oil yield of 36.65%. In the study (Dewi, 2012), the characterization of oil from kidney bean seeds was carried out using the Soxhlet extraction method with two types of solvents namely n-hexane and petroleum ether, different oil results were obtained due to different types of solvents, but the differences were only slight. This makes it possible to use petroleum ether solvents in the extraction process due to the relatively cheap price compared to n-hexane solvents. In addition, petroleum ether is a non-polar solvent with high selectivity.

In this research, optimization of papaya seed oil extraction will be carried out using the soxhletation extraction method and the level $2^3$ factorial design experimental design method to determine the most influential process variables, as well as obtain information regarding the interactions between variables to obtain optimum results. The use of petroleum ether solvent is a novelty in this research. Researchers generally use n-hexane solvents which are more expensive than petroleum ether solvents. With this novelty, optimal and more profitable results can be obtained based on economic value.

## METHODOLOGY

### Materials and Tools

The materials used in this study were 420 grams of papaya seeds (dry weight) obtained from a fruit seller in the market around Tembalang and 6000 mL of Petroleum Ether solvent purchased from Toko Agung Jaya Solo. The tools used are a series of soxhletation tools, a series of distillation apparatuses, sieves (10, 15, 20, 25, 30 mesh), an oven, grinder, filter paper, porcelain cup, desiccator, digital balance, Erlenmeyer, magnetic stirrer, pipette, measuring cup, stand coolers, electric heaters, volumetric flasks, and burettes.

### Papaya Seed Pretreatment

At this stage, the material that needs to be prepared is papaya seeds. The first step is to clean the papaya seeds by washing them and then drying them in the oven until they turn brown. Then grind the papaya seeds and sift according to the size of the variables, namely 10 and 30 mesh. Then store it in ziplock plastic.

### Extraction of Oil from Papaya Seeds

Prepare 35 grams of papaya seed oil measuring 10 mesh, then wrap it using filter paper, and put it in the soxhlet. Put 400 mL of Petroleum Ether into a three-neck flask, and extract for 170 minutes.

### Distillation of Extracted Oil

This stage begins with assembling a distillation apparatus, then inserting the extracted papaya seed oil into a three-neck flask, and distilling it at 60ºC for 60 minutes. Weigh the oil obtained and store it in a bottle for later analysis.

### Papaya Seed Oil Yield (% Yield)

Data analysis was used to determine the percentage of oil yield contained in papaya seeds. By weighing the initial mass of papaya seeds, then weighing the yield of papaya seed oil. Based on research (Rina et al., 2021), to calculate the yield percentage using Eq. (1):

$$Yield\ (\%) = \frac{oil\ yield\ weight}{material\ weight} \times 100\% \quad (1)$$

### Specific Gravity of Papaya Seed Oil (Density)

Density is done to get the density value of the resulting papaya seed oil. By weighing the empty pycnometer and recording it. Then fill the pycnometer with papaya seed oil up to the neck of the pycnometer, then close the pycnometer and make sure there are no air bubbles in the pycnometer. Then clean the outside of the pycnometer with a tissue or cloth until dry. And weigh the pycnometer along with the papaya seed oil in it. Based on research (Rina et al., 2021), to calculate density using Eq. (2):

$$\rho = \frac{m_2 - m_1}{v\ pycno} \quad (2)$$

Information:

ρ = Density of papaya seed oil (gr/mL)
m1 = Weight of empty pycnometer (gr)
m2 = Weight of pycnometer with sample (gr)
v pycno = Volume pycnometer (mL)

### FFA levels (Free Fatty Acid)

FFA analysis of papaya seed oil refers to (BSN, 2019) by preparing a neutral 95% ethanol solution, and a standard 0.1 N NaOH solution. After that, weigh 5 grams of the sample, put it in an Erlenmeyer, and add 50 mL of 95% ethanol. then drip with 3-5 drops of phenolphthalein indicator, and titrate with 0.1 N NaOH standard solution until a pink color is formed which lasts for 30 seconds. Record the volume of NaOH required for the titration, and calculate the free fatty acid content (FFA content) using Eq. (3):

$$FFA\ Levels = \frac{Vn \times Nn \times 282}{oil\ weight} \times 100\% \quad (3)$$

Information:

Vn = NaOH titration volume (mL)
Nn = NaOH Normality (mL)

### Viscosity

A viscosity test was carried out by cleaning the Ostwald viscometer. then water is put into the viscometer until it does not pass the mark. Place the deflated rubber ball on the capillary tube. Suck in the water by pressing the s point on the rubber ball to the upper mark. Then release the rubber ball while turning on the stopwatch. When the water reaches the lower mark, turn off the stopwatch. Then repeat these steps on the papaya seed oil. To calculate the viscosity can use Eq. (4):

$$\mu_x = \frac{t_x \times d_x}{t_0 \times d_0} \times \mu_0 \quad (4)$$

Information:

$\mu_x$ = Papaya Seed Oil Viscosity (cP)
$\mu_0$ = Water Viscosity (cP)
$t_x$ = Papaya Seed Oil Flow Time (s)
$t_0$ = Water Flow Time (s)
$d_x$ = Density of Papaya Seed Oil (gram/mL)
$d_0$ = Density of Papaya Seed Oil (gram/mL)

**Water Content Analysis**

This analysis was conducted to determine the percentage of water content contained in papaya seed oil. By weighing a porcelain cup that has been in the oven for 30 minutes at 105ºC, record the results. Then weigh 5 grams of papaya seed oil (sample weight) and put it in a porcelain cup, oven at 105ºC, for 1 hour, then cool in a desiccator to room temperature, then weigh it until constant weight, and record the results. Based on research (Rina et al., 2021), to calculate the water content using Eq. (5):

$$\%\ Water\ content = \frac{(w - w_0)w_1}{w_0} \times 100\% \quad (5)$$

Information:

W = Weight after in the oven (gr)
$W_0$ = Papaya seed oil sample weight (gr)
$W_1$ = Weight of cup + oil after baking (gr)

**RESULTS AND DISCUSSION**

Research on the extraction of papaya seed oil has been carried out and will be processed using a level $2^3$ factorial design experiment where there are 3 research variables, namely extraction time (170 minutes and 190 minutes), particle size (dried papaya seeds) (10 mesh and 30 mesh), the ratio of substance to solvent (1:10 g/g and 1:20 g/g). This study conducted 8 trials. This method is used to determine the effects of the variables, the most influential variables, and the optimum operating conditions. The use of symbols (+) and (-) indicates the type of the variable used in each experiment.

Table 1. Papaya Seed Oil Extraction Results (Factorial Design Level $2^3$)

| Run | Changed Variables | | | Interaction | | | | Yield | Density | FFA Levels | Viscosity | Water content |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | t | s | r | ts | tr | sr | tsr | | | | | |
| 1 | - | - | - | + | + | + | - | 49.51 | 0.925 | 0.564 | 1.320 | 0.092 |
| 2 | + | - | - | - | - | + | + | 35.69 | 0.926 | 0.564 | 1.355 | 0.045 |
| 3 | - | + | - | - | + | - | + | 54.14 | 0.927 | 0.677 | 1.531 | 0.098 |
| 4 | + | + | - | + | - | - | - | 55.03 | 0.925 | 0.620 | 1.685 | 0.085 |
| 5 | - | - | + | + | - | - | + | 46.11 | 0.929 | 0.564 | 1.489 | 0.082 |
| 6 | + | - | + | - | + | - | - | 42.57 | 0.927 | 0.564 | 1.476 | 0.071 |
| 7 | - | + | + | - | - | + | - | 53 | 0.928 | 0.395 | 1.564 | 0.073 |
| 8 | + | + | + | + | + | + | + | 59.43 | 0.926 | 0.677 | 1.869 | 0.067 |

In this research, 8 experiments were carried out, with each experiment being given a different treatment according to the experimental design that had been made before. The percentage of oil yield produced is based on Table 1, namely the longer the extraction time, the higher the percentage of oil yield produced, because the longer the extraction time, the contact that occurs between the materials and the dissolution lasts a long time, so that the oil content in the papaya seeds will be extracted a lot (Azhari et al., 2020). In previous studies, the best results were also obtained at an extraction time of 180 minutes. The oil yield increased as the volume of solvent used increased. The more volume of solvent used, the greater the ability of the solvent to extract the oil contained in papaya seeds (Wulandari, 2017). In the process variable particle size, where at the lower level the particle size (-) 10 mesh produces less oil yield than the upper level (+) 30 mesh. The smaller the sample size, the greater the yield of oil produced (Andaka, 2020). To be able to know the optimal operating conditions need to be optimized.

Before optimizing, it is necessary to know the most influential process variables in the research using the quicker method, namely by taking into account the main effect and interaction with the resulting oil yield.

Based on the calculation of the quicker method, the greatest effect is produced at 11.9285. And the most influential variable is produced, namely particle size. These results can be seen in Table 2 and Table 3.

Table 2. Main Effect Calculation Results and Interaction on Yield

| Effect | Amount |
|---|---|
| $I_1$,t | -2.514 |
| $I_2$,s | 11.928 |
| $I_3$,r | 1.686 |
| $I_{12}$,ts | 6.171 |
| $I_{13}$,tr | 3.957 |
| $I_{23}$,sr | -0.057 |

Table 3. Determination of Influential Variables

| P(%) | Efek |
|---|---|
| 7.14 | 0.0571 |
| 21.43 | 1.1857 |
| 35.71 | 1.6857 |
| 50.00 | 2.5142 |
| 64.29 | 3.9571 |
| 78.57 | 6.1714 |
| 92.86 | 11.9285 |

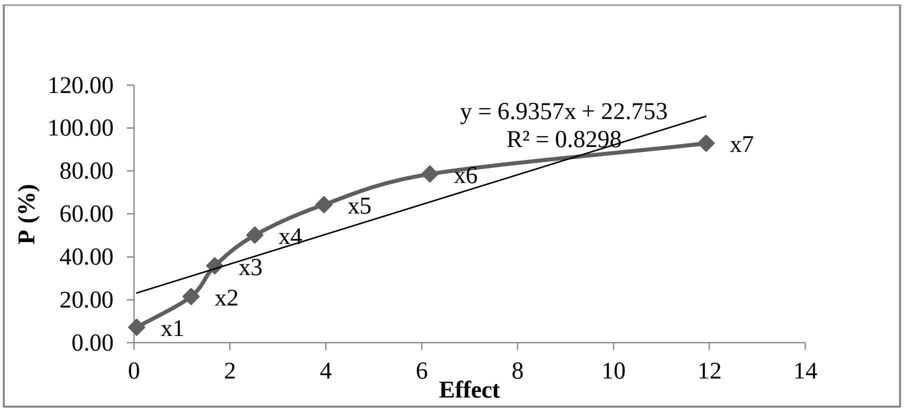


Figure 1. Normal Probability Plot for $2^3$

Whereas in Figure 1 it shows that X7, where the point of the calculation of the effect of particle size with percent probability is away from the density. From the results obtained, process optimization can be carried out with variations in particle size (s) to determine the yield of papaya seed oil. Optimasi yang dilakukan yaitu pada ukuran partikel 10 mesh, 20 mesh, 25 mesh, dan 30 mesh. And with extraction operating conditions time of 180 minutes, and the ratio of material to solvent 1 : 9 g/g.

**Optimization of Papaya Seed Oil Extraction**

Based on the analysis that has been done, it can be concluded that the process variable that has the most influence on papaya seed oil extraction research is particle size (papaya seed size), so in the optimization process variables, t (extraction time) and r (ratio of material to solvent) will be used. and the variable s (particle size) as the variable changes. The optimization results that have been carried out are presented in Table 4.

Table 4. Normal Probability Plot for $2^3$

| Extraction Time (minute) | Seed Size (*mesh*) | The ratio of Material to Solvent (g/g) | Yield (%) |
|---|---|---|---|
| 180 | 10 | 1 : 9 ( 35 gr : 500 mL) | 47.057 |
| | 15 | | 52.257 |
| | 20 | | 57.029 |
| | 25 | | 51.800 |
| | 30 | | 46.114 |

In Table 4 it can be seen that there was an increase in the yield of papaya seed oil extraction at seed sizes of 10 mesh to 20 mesh, then decreased at seed sizes of 25 mesh to 30 mesh. These results can be seen in the optimization graph of papaya seed oil extraction in Figure 2.

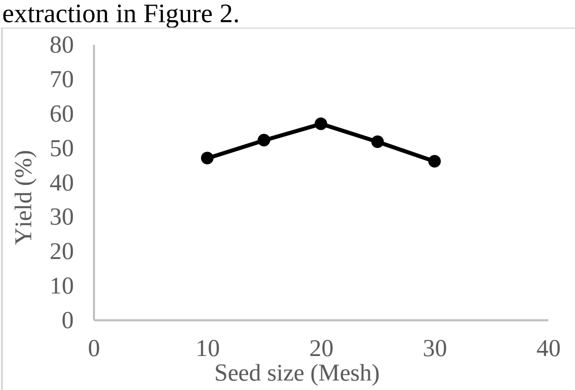


Figure 2. Optimization Graph of Papaya Seed Oil Extraction Results

Based on the yield of papaya seed oil produced, it can be seen that the optimum conditions were achieved for the variable changing the size of the papaya seeds by 20 mesh, namely a yield of 57.029% with optimum operating conditions of 180 minutes extraction time and a ratio of material to solvent 1 : 9 g/g. This is because the larger the mesh size, the smaller the seed size, and has a large surface area so that it can improve the performance of the extraction process, and the extract yields obtained are increasing. In the extraction of papaya seed oil, the appropriate or most optimal size of papaya seeds will make the extraction process run faster. In this study the optimal mesh size was 20 mesh, then the extraction yield decreased for larger mesh sizes, namely 25 mesh to 30 mesh. This can happen because the material is too fine, and the space between cells will be narrower, making it difficult for the solvent to enter into the powder.

**Analysis of Papaya Seed Oil Extraction Result**

Analysis of the oil extracted from papaya seeds, namely density test, FFA content, and water content. This is to find out whether the oil extracted from papaya seeds is in accordance with the existing SNI.

**Density**

A density test was carried out on samples at optimum operating conditions, namely particle size of 20 mesh, extraction time of 180 minutes, and the ratio of material to solvent 1 : 9 g/g. The test results can be seen in Table 5.

Table 5. Papaya Seed Oil Density Test Results

| Testing | Extracted Oil | Prior Research Oils | SNI Papaya Seed Oil |
|---|---|---|---|
| Density (gr/mL) | 0.926 | 0.90 | 0.924-0.929 |

The results in the table above show that the density of extracted papaya seed oil is close to that obtained by previous studies, and is in accordance with the Indonesian National Standard (SNI), which is between 0.924-0.929 gr/mL. In previous studies, the results obtained a density of 0.90427 g/mL (Andaka, 2020). With this density test can be known the purity of papaya seed oil. The more components contained in the oil, the higher the heavy fraction. Which means the specific gravity of the oil will be greater (Azhari et al., (2020).

**FFA Levels ( Free Fatty Acid)**

In the FFA content test carried out on samples at optimum operating conditions, namely particle size of 20 mesh, extraction time of 180 minutes, and the ratio of material to solvent 1: 9 g/g, the test results are presented in Table 6.

Table 6. Papaya Seed Oil FFA Levels Test Results

| Testing | Extracted Oil | Prior Research Oils | SNI Papaya Seed Oil |
|---|---|---|---|
| FFA Levels (%w/w) | 0.58 | 0.47 | 0.36-0.82 |

Free fatty acid testing is an important parameter to determine oil quality. The level of free fatty acids contained in vegetable oil can be one of the parameters determining the quality of the oil. The amount of free fatty acids in the oil is indicated by the value of the acid number (Cahyaningtyas et al., 2017). A high acid number indicates that the free fatty acids present in vegetable oil are also high so the quality of the oil is even lower, this is because the free fatty acids produced due to hydrolysis can reduce the quality of the oil. Free fatty acids can increase due to repeated heating even at a constant temperature (Andaka, 2020). If you look at the table above, the oil from this study is in accordance with the SNI for papaya seed oil, which is 0.58, where the SNI for free fatty acids for papaya seed oil is between 0. 36-0.82. The results of free fatty acid levels in this study were carried out under operating conditions, namely an extraction time of 180 minutes, a distillation time of 1 hour, a particle size of 20 mesh, and a ratio of material to solvent of 1: 9 g/g, with a weight of dry papaya seed material of 35 grams and 500 mL of solvent.

**Water Content**

In the water content test carried out on samples at optimum operating conditions, namely particle size of 20 mesh, extraction time of 180 minutes, and the ratio of material to solvent 1: 9 g/g, the test results are presented in Table 7.

Table 7. Papaya Seed Oil Water Content Test Results

| Testing | Extracted Oil | Prior Research Oils | SNI Papaya Seed Oil |
|---|---|---|---|
| Water content (%w/w) | 0.077 | 0.088 ± 0.05 | Maks. 0.15 |

Based on Table 7 it is known that the water content of papaya seed oil is 0.077%w/w, this result is in accordance with SNI papaya seed oil, which is a maximum of 0.15%w/w. The water content in the oil is one of the parameters determining the quality of the oil. The higher the water content in the oil, the lower the quality of the oil because water is one of the hydrolysis catalysts in oil (Cahyaningtyas et al., 2017). In this study, the decrease in water content was caused by repeated heating, namely in the distillation and oven processes to remove solvents which could cause the oil to be easily damaged and the water content in the samples included so that the water content would decrease. In addition, the higher the temperature and the longer the heating will accelerate evaporation so that the water contained in the oil will be less (Andaka, 2020). The water content obtained in this and previous studies is in accordance with SNI. In this study, it was found that the quality of papaya seed oil was better because of less water content.

**CONCLUSION**

This study uses factorial design analysis and the quicker method to calculate the effect. From the experiments conducted, it was found that the most influential process variable for optimizing the papaya seed oil extraction process was the particle size variable (s) with an effect value of 11,928.

From the optimization carried out on the particle size variable, the optimum yield value was obtained at 57.029%, namely at a particle size of 20 mesh, an extraction time of 180 minutes, and a ratio of material to solvent of 1:9 (35 grams : 500 mL). The resulting density is 0.926 gr/mL according to SNI, namely 0.924 - 0.929. The resulting FFA (Free Fatty Acid) content is 0.58% according to SNI, namely 0.36% - 0.82%. And the resulting water content is 0.077% according to SNI, which is a maximum of 0.15%.

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