

Research Article

Simultaneous Optimisation of Oil and Resin Extraction from Nyamplung Seeds

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ABSTRACT: Nyamplung (*Calophyllum inophyllum* L.) is a plant with great potential as a fuel source that can be developed in Indonesia. Nyamplung seeds contain 60-75% oil and 10-30% resin. This study aims to optimise the extraction process of oil and resin from nyamplung seeds using hexane and methanol solvents under various operating conditions and to determine the optimal extraction conditions. In this process, extraction will be carried out using two types of solvents (hexane-methanol) with several operational variations, namely: (1) extraction temperature (35-45°C); (2) extraction time (4-6 hours); (3) stirring speed (200-600 rpm). The extraction process of oil and resin from nyamplung seeds was optimised using the Response Surface Methodology (RSM). The oil and resin obtained were then characterised through several analyses. The analyses for oil included yield, acid number, density, and viscosity, while the analyses for resin included yield and acid number. Based on the research results, simultaneous optimisation of the extraction process conditions for oil and resin from nyamplung seeds using a mixture of hexane and methanol solvents yielded an oil yield of 52.43% and a resin yield of 9.56% at a temperature of 40°C, extraction time of 5 hours, and stirring speed of 500 rpm. Optimisation aimed at maximising oil yield resulted in a yield of 55.33% at a temperature of 45°C, extraction time of 6 hours, and stirring speed of 600 rpm. Resin yield was obtained at 10.91% at a temperature of 36.35°C, extraction time of 4 hours, and stirring speed of 591.78 rpm. The quality of the optimised oil simultaneously includes an acid number of 10.51 mgKOH/g, density of 0.94 g/ml, and viscosity of 4.47 cP, while the resin has an acid number of 142.79 mgKOH/g.

Keywords: *calophyllum*, oil, optimisation, resin, response surface methodology

1. INTRODUCTION

Population growth accompanied by improved community welfare has led to an increase in household needs. This has led to a rise in the demand for fuel oil. Globally, fuel oil is the largest energy source compared to other energy sources. The demand for fuel oil increases with population growth and technological development, but fossil fuel reserves are depleted due to their non-renewable nature. Energy consumption has grown so much that Indonesia can no longer meet its own energy needs. Indonesia's oil reserves have declined from 5.9 billion barrels in 1995 to 3.7 billion barrels by the end of 2015. With current oil production levels and no new oil reserves discovered, it is estimated that Indonesia's oil reserves will be depleted within the next 11 years [1]. This estimate is supported by frequent fuel shortages in several regions of Indonesia.

Indonesia's dependence on petroleum needs to be reduced and even eliminated. This problem can be overcome by developing alternative renewable energy sources, such as vegetable oil. Given the abundance of oil-producing plants in Indonesia that can be used as raw materials for biodiesel, biodiesel is an alternative renewable energy source that can be used as a substitute for diesel fuel. One of the most promising plants for use as a raw material for biodiesel is the nyamplung plant (*Calophyllum inophyllum* L.).

Oil from nyamplung seeds has great potential to be developed as a raw material for several products, as its use does not compete with food production. One of the most developed applications is as a raw material for biofuel, as the dried seeds contain 60-75% oil [2]. Nyamplung seeds contain a fairly high resin content (10 – 30%), which enhances the diversity of products derived from nyamplung seeds and increases their value. In the health sector, Nyamplung seed resin can be used as a medicinal ingredient due to its various compounds that can be utilised as anti-HIV agents [3]. In the industrial sector, resin can be used as a coating material [4].

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In Indonesia, the natural potential of nyamplung seeds is not yet known with certainty, but initial estimates suggest that the potential area is 480,000 ha, of which 255–300 ha is nyamplung forest. The fruit production potential of natural stands varies depending on location, such as Ciamis (60–110 kg/tree/year), Banyuwangi (220 kg/tree/year), Purworejo (70 – 150 kg/tree/year), and Papua (130 kg/tree/year) [5]. The productivity of nyamplung seeds is very high, ranging from 40–150 kg/tree/year or approximately 20 tons/ha/year. This indicates that nyamplung seed productivity is higher than that of other crops such as *jatropha*, which is approximately 5 tons/ha/year, and oil palm, which is approximately 6 tons/ha/year [6].

To produce biofuel, the initial step is to extract oil from oil-bearing materials. Oil extraction can be performed using several methods, such as rendering, mechanical extraction, and chemical extraction [7]. The most commonly used method for extracting nyamplung oil is mechanical extraction or pressing. The oil produced from this method is black and thick. This is because the mechanical extraction process (pressing) is less selective in extracting the substances contained in nyamplung seeds. Unlike chemical extraction, this process uses solvents that are more selective compared to mechanical extraction [8].

2. RESEARCH METHODOLOGY

This research will take several stages, namely, drying the nyamplung fruit until the water content reaches 10-15%. Next, the fruit is peeled to separate the shell from the seeds. To reduce the size, the seeds are then ground using a blender. Then experiment with the extraction of oil and resin simultaneously. Then perform separation, which separates the hexane fraction from the methanol fraction. The hexane fraction was evaporated to evaporate hexane and obtain oil, and the methanol fraction was evaporated to evaporate methanol and obtain resin. Further characterisation of the physicochemical properties of oil and resin.

2.1 Materials and Tools

The main tool used for oil and resin extraction is a three-neck flask with a capacity of 2 L equipped with a condenser, stirring motor, thermometer and heater. The following is a series of tools:

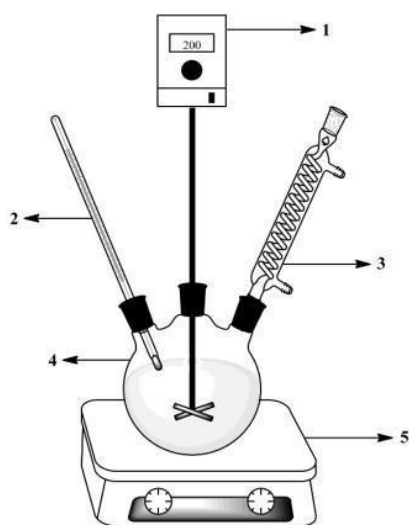


Figure 1 Experiment set-up

Tool Description:

1. Stirring motor
2. Thermometer
3. Condenser
4. Triple-neck flask
5. Hot plate

The materials needed are nyamplung fruit that has been dried using sunlight for 10 days, methanol (98% purity) and hexane (98% purity), as well as other solutions for characterisation tests of the physicochemical properties of oil and resin.

2.2 Research Procedures

2.2.1 Preparation of Tools and Materials

For the simultaneous extraction of oil and resin from nyamplung fruit raw materials that have been sun-dried for 10 days, the first thing to do is to assemble the tools that have been provided in the laboratory. Second, the shell of the nyamplung fruit is manually separated from the seeds. Seeds with a moisture content of about 10-15% were ground to reduce their size using an electric grinder (blender) for 15 minutes.

2.2.2 Extraction

Raw materials that have gone through the preparation stage are put into the reactor, then methanol and hexane are added. The ratio of material to methanol (b/v) used was 1:2; and the ratio of material to hexane (b/v) used was 1:5. The extraction process was carried out in a reactor (triple neck flask) with a capacity of 2 L equipped with a stirrer, heater and condenser, under temperature conditions of 35 – 45°C; reaction time of 4 – 6 hours, and stirrer speed of 200 – 600 rpm.

2.2.3 Separation

After the extraction process is complete, the mixture is cooled to room temperature, then the filtrate is separated from the pulp using a vacuum filter. After that, proceed to the separation process, where the filtrate is separated by putting it into a separating flask and left overnight so that the filtrate forms two layers. The top layer is the hexane fraction and the bottom layer is the methanol fraction. Both fractions are then evaporated to separate the oil from hexane and resin from methanol.

2.2.4 Characterization

The oil and resin obtained are then characterized through several analyses. The analysis for oil is yield, acid number, density and viscosity, while the analysis for resin is yield, and acid number.

2.2.5 Experiment Design

The experiment was designed so that the data generated could be processed using Response Surface Methodology (RSM). In the study, three independent variables were determined, namely temperature (X_1), time (X_2) and stirrer speed (X_3).

Table 1 Process center condition variables

Variable	Code	Units	Taraf		
			-1	0	+1
Temperature	X_1	°C	35	40	45
Time	X_2	Hours	4	5	6
Stirrer speed	X_3	rpm	200	400	600

2.3 Experimental Variations

The variations carried out in this study were extraction temperature (35 – 45°C), extraction time (4 – 6 hours), and stirrer speed (200 – 600 rpm), optimization of the oil and resin extraction process from nyamplung seeds was carried out using Significant Surface Methodology (RSM).

3. RESULT AND DISCUSSION

3.1 Optimisation of Oil Extraction Process

3.1.1 Oil Yield

The oil yield produced in this study was 46.18 – 55.96%. Based on the results of sequential analysis, lack of fit, adjusted R², and predicted R² using design expert 11 (Table 2), the recommended model for optimising process conditions with yield response is a linear model.

Table 2 Values of optimisation parameters for oil yield response

Parameter	Sequential P-value	Lack of Fit P-value	Adjusted R ²	Predicted R ²	Description
Linear	0,0279	0,2039	0,3161	0,0103	Suggested
2FI	0,3301	0,2081	0,3511	-0,6022	
Quadratic	0,9892	0,1021	0,1592	-1,7443	
Cubic	0,2598	0,0702	0,3439	-22,7225	

Based on Table 2, it can be seen that the linear model has a significant sequential value ($P < 0.05$) of 0.0281, while for other models the sequential value gives insignificant results ($P > 0.05$). This shows that the linear model is more suitable than other models. This is supported by the lack of fit value, which is insignificant and higher than the lack of fit value of the quadratic model or the cubic model. The adjusted R² value shows the contribution of regression factors to the response [9]. The higher the adjusted R² value, the greater the contribution or influence of the factor on the response. Predicted R² is used to determine the suitability of the adjusted R² value obtained, where the smaller the difference between adjusted R² and Predicted R², the better the adjusted R² value. Based on table 2, it can be seen that the difference between adjusted R² and predicted R² is the lowest, this confirms that the linear model shows a high level of significance compared to other proposed models. The following is the linear polynomial model equation obtained:

$$y_1 = 36,541 + 0,1901X_1 + 0,959X_2 + 0,0073X_3 \dots \dots \dots (1)$$

Description:

y_1 = Oil yield response value (%)

X_1 = temperature (C)

X_2 = time (hours)

X_3 = stirrer speed (rpm)

Based on the results of the analysis of the significance of the influence of variables on the response, it was found that the variable speed of the stirrer had a significant effect on the oil yield response [10]. The other variables (temperature and time) did not significantly affect the oil yield. Figure 2 shows the contours of the effect of stirring speed on yield at various temperatures and times.

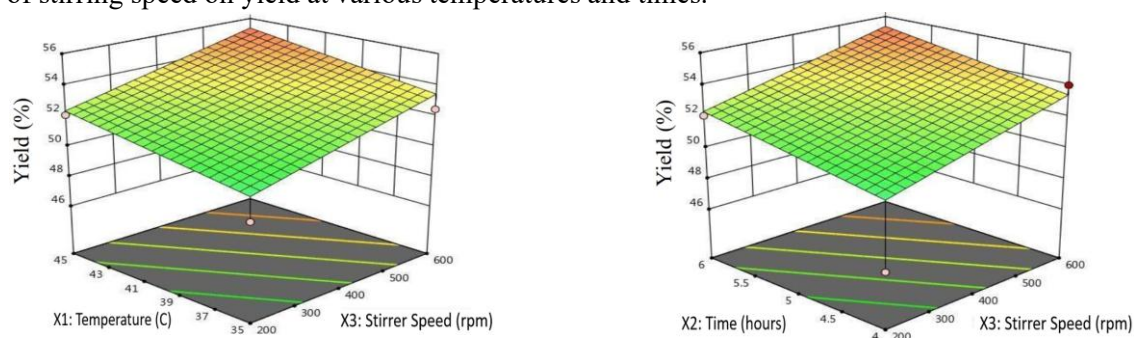


Figure 2 Contours of the effect of stirrer speed on oil yield at various process conditions

Based on Figure 2, it can be seen that the oil yield increases as the stirrer speed increases. The effect of stirrer speed is significant on oil yield, because the higher the stirrer speed, the more the mass transfer rate increases along with the distribution of material particles in the solvent faster [11]. The mass transfer rate will begin to slow down and stop as equilibrium conditions are reached in the solution [12].

Based on Figure 2, it can also be seen the effect of temperature and time factors on yield. The higher the reaction temperature, the higher the oil yield. An increase in temperature can generally cause the pores of solids to expand so that the rate of diffusion of solvents into the pores of solid materials or cell walls and the dissolution of solutes by solvents in cells increases, resulting in yields can be increased by increasing the extraction temperature [13]. In addition, higher temperatures can also accelerate the diffusion rate so that the extraction diffusion process can run faster [14]. However, the use of a temperature of 40°C is optimal enough to extract oil from nyamplung seeds [15].

The effect of temperature can be seen through the results of the linear equation obtained, where temperature has a positive factor coefficient on the oil yield response, meaning that the positive value indicates the correspondence between the increase in factors and responses. The higher the factor value, the higher the resulting response.

Extraction time is the length of the process used in the extraction process, which is related to the amount of oil conversion in Nyamplung seeds into oil. The longer the extraction time, the higher the oil yield. This is in accordance with research, which states that the longer the extraction time, the higher the yield will be because more oil dissolves in hexane until equilibrium conditions are reached [16]. However, at an extraction time of 5 hours the resulting oil yield is more than that of 7 hours [15]. This indicates that equilibrium conditions have been reached and oil can be extracted optimally at an extraction time of 5 hours. Equilibrium conditions in extraction are conditions where the solute in the material can no longer dissolve in the solvent and the concentration of oil in the solution remains the same before decreasing [17]. When the concentration of oil in the solvent (hexane) is maximum, the solvent is no longer able to bind the oil both in the wall and in the cell [18].

Based on the results of analysis using design expert 11, the optimum oil yield obtained was 55.33% obtained at a temperature of 45°C, extraction time of 6 hours and stirring speed of 600 rpm.

3.1.2 Acid Number of Oil

The acid number of the oil produced in this study was 5.11 - 21.97 mgKOH/g. Based on the results of sequential analysis, lack of fit, adjusted R^2 , and predicted R^2 using design expert 11 (Table 3), the recommended model for optimizing process conditions with acid number response is a linear model.

Table 3 Optimization parameter values for acid number response

Parameter	Sequential P-value	Lack of Fit P-value	Adjusted R^2	Predicted R^2	Description
Linear	0,0351	< 0,0001	0,2960	-0,083	Suggested
2FI	0,9248	< 0,0001	0,1642	-1,6521	
Quadratic	0,7212	< 0,0001	0,0429	-2,9172	
Cubic	0,0521	< 0,0001	0,5989	-26,674	

Based on Table 3, it can be seen that the linear model has a significant sequential value ($P < 0.05$) of 0.0348, while for other models the sequential value gives insignificant results ($P > 0.05$). This shows that the linear model is more suitable than other models. This is supported by the difference between Adjusted R^2 and Predicted R^2 which is the lowest among other models, thus confirming that the linear model shows a high level of significance compared to other proposed models. The following is the linear polynomial model equation obtained:

$$y_2 = 34,149 + 0,252X_1 - 1,189X_2 - 0,013X_3 \dots \dots \dots (2)$$

Description:

y_2 = Oil acid number response value (mgKOH/g)

X_1 = temperature (C)

X_2 = time (hours)

X_3 = stirrer speed (rpm)

Based on the results of the analysis of the significance of the influence of variables on the response, it was found that the variable of stirring speed significantly influenced the response of oil acid number. Other variables (temperature and time) did not significantly affect the acid number of oil. Figure 3 shows the contours of the effect of stirring speed on acid number at various temperatures and times.

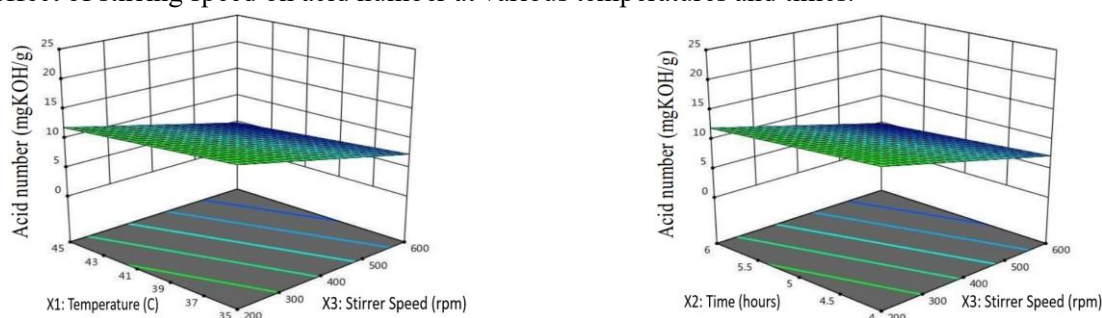


Figure 3 Contours of the effect of stirrer speed on acid number at various process conditions

Based on Figure 3, it can be seen that the acid number of the oil decreases as the stirrer speed increases. The effect of stirring speed is significant on the acid number of oil, because its increase accelerates the solubility of acidic compounds into the solvent and prevents the deposition of materials at the bottom of the reactor tank, so that equilibrium is reached more quickly [19]. The lower the acid number of the oil, the higher the quality of the oil produced. Oil with a low acid number is more stable so that the shelf life is longer [20]. Stable oil does not easily react with the surrounding conditions, such as air, light and heat so it is not easily damaged [15].

Based on Figure 3, it can also be seen the effect of temperature and time factors on acid number. The higher the temperature and extraction time, the lower the acid number obtained. This can be seen through the results of the linear equation obtained, where the temperature and extraction time have a negative factor coefficient, meaning that the acid number decreases when the temperature and extraction time are increased. The same was done with the results of nyamplung oil acid number decreased by increasing the temperature and reaction time [15].

Based on the analysis using design expert 11, the optimum oil acid number obtained is 4.79 mgKOH/g obtained at a temperature of 45°C, extraction time of 6 hours and stirring speed of 600 rpm.

3.1.3 Oil Density

The density of the oil produced in this study was 0.923 – 0.99 g/ml. Based on the results of sequential analysis, lack of fit, adjusted R^2 , and predicted R^2 using design expert 11 (Table 4), the recommended model for optimizing process conditions with density response is a linear model.

Table 4 Optimization parameter values for oil density response

Parameter	Sequential P-value	Lack of Fit P-value	Adjusted R^2	Predicted R^2	Description
Linear	0,0161	0,0013	0,3671	0,1011	Suggested
2FI	0,4013	0,0011	0,3742	-0,6973	
Quadratic	0,0568	0,0027	0,6019	-0,5239	
Cubic	0,0589	0,0054	0,8244	-8,9087	

Based on Table 4, it can be seen that the linear model has a significant sequential value ($P < 0.05$) of 0.0158, while for other models the sequential value gives insignificant results ($P > 0.05$). This shows that the linear model is more suitable than other models. This is supported by the difference between Adjusted R^2 and Predicted R^2 which is the lowest among other models, thus confirming that the linear model shows a high level of significance compared to other proposed models. The following is the linear polynomial model equation obtained:

$$y_3 = 1,041 - 3,5 \times 10^{-4} X_1 - 1,3 \times 10^{-2} X_2 - 5,28 \times 10^{-6} X_3 \dots \dots \dots (3)$$

Description:

y_3 = Oil density response value (g/ml)

X_1 = temperature (C)

X_2 = time (hours)

X_3 = stirrer speed (rpm)

Based on the results of the analysis of the significance of the influence of variables on the response, the results show that the time variable has a significant effect on the oil density response. The other variables (temperature and stirrer speed) do not significantly affect the oil density. Figure 4 shows the contours of the effect of time on density at various temperatures and stirrer speeds

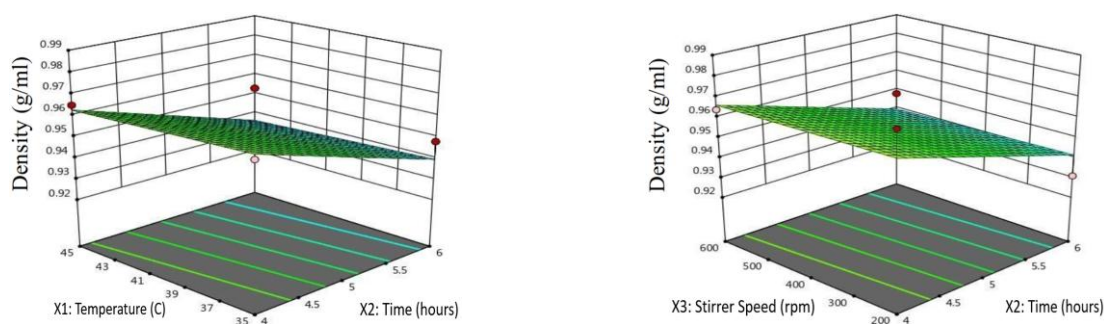


Figure 4 Contours of the effect of time on oil density at various process conditions

Based on Figure 4, it can be seen that the oil density decreases as the extraction time increases. The effect of extraction time is significant on oil density, but the increase is not very significant. Based on the results of research which states that the effect of extraction time on oil density is not significant because the higher the extraction time, the density of the oil produced is relatively constant [21]. The lower the oil density, the higher the quality of the oil produced. Oil with high density is of lower quality because it shows that there are more impurities contained in the oil [15].

Based on Figure 4, it can also be seen the effect of temperature and stirrer speed factors on oil density. The higher the temperature and speed of the stirrer, the density of the oil obtained decreases. This can be seen through the results of the linear equation obtained, where the temperature and stirring speed have a negative factor coefficient, meaning that the oil density decreases when the temperature and stirring speed are increased.

Based on the results of analysis using design expert 11, the optimum oil density obtained is 0.94 g/ml obtained at a temperature of 35°C, extraction time of 6 hours and stirring speed of 600 rpm.

3.1.4 Oil Viscosity

The viscosity of the oil produced in this study was 5.11 – 21.97 cP. Based on the results of sequential analysis, lack of fit, adjusted R^2 , and predicted R^2 using design expert 11 (Table 5), the recommended model for optimizing process conditions with viscosity response is the quadratic model.

Table 5 Optimization parameter values for oil viscosity response

Parameter	Sequential P-value	Lack of Fit P-value	Adjusted R ²	Predicted R ²	Description
Linear	0,0603	< 0,0001	0,2421	-0,0911	
2FI	0,2101	< 0,0001	0,3329	-0,9823	
Quadratic	0,1032	< 0,0001	0,5189	-0,9067	Suggested
Cubic	0,0461	0,0002	0,8072	-11,7761	

The quadratic model is the most suitable model in describing the influence between the factors and the oil viscosity response. The following is the linear polynomial model equation obtained:

$$y_4 = 113,4 - 3,231X_1 - 10,158X_2 - 0,060X_3 - 0,116X_1X_2 - 7,3 \times 10^{-4}X_1X_3 + 0,011X_2X_3 + 0,050X_1^2 + 0,802X_2^2 + 3,5 \times 10^{-5}X_3^2 \dots\dots\dots(4)$$

Description:

y_4 = Oil viscosity response value (Cp)

X_1 = temperature (C)

X_2 = time (hours)

X_3 = stirrer speed (rpm)

The quadratic model produces consequences that involve interactions between factors with each other (interaction between temperature – process time, temperature – stirrer speed, and process time – stirrer speed).

Based on the results of the significance analysis of the influence of variables on the response, it was found that the extraction time variable had a significant effect on the oil viscosity response. The results of the analysis of the P value show that the extraction time and the interaction between extraction time and stirring speed give significant results compared to other factors and interactions ($P < 0.05$). This indicates that the viscosity level of nyamplung oil formed during extraction is influenced by the extraction time and its interaction with the stirring speed (a combination of settings between the extraction time that takes place and the required stirring speed). Figure 5 shows the contours of the effect of time on viscosity at various temperatures and stirrer speeds.

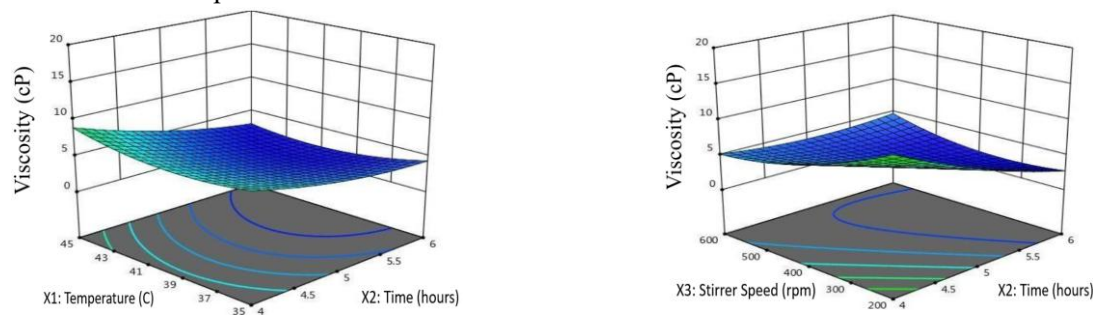


Figure 5 Contours of the effect of time on oil viscosity at various process conditions

Based on Figure 5, it can be seen that the viscosity of the oil decreases as the extraction time increases. The effect of extraction time is significant on oil viscosity, but the increase is not very significant. Based on the results of research which states that the effect of extraction time on oil viscosity is not significant because the higher the extraction time, the viscosity of the resulting oil is relatively constant [21]. The lower the viscosity of the oil, the higher the quality of the oil produced. Oil with high viscosity has a lower quality because it shows that there are more impurities contained in the oil [15].

Based on Figure 5, it can also be seen the effect of temperature and stirring speed factors on oil density. The higher the temperature and stirring speed, the viscosity of the oil obtained decreases. This can be seen through the results of the linear equation obtained, where the temperature and stirring speed have a negative factor coefficient, meaning that the oil density decreases when the temperature and stirring speed are increased.

Based on the results of analysis using design expert 11, the optimum oil viscosity obtained is 2.79 cP obtained at a temperature of 41.45°C, extraction time of 5.6 hours and stirring speed of 354 rpm.

3.2 Optimization of Resin Extraction Process

3.2.1 Resin Yield

The resin yield produced in this study was 6.2-10.86%. Based on the results of sequential analysis, lack of fit, adjusted R², and predicted R² using design expert 11 (Table 6), the recommended model for optimizing process conditions with yield response is the 2FI model.

Table 6 Optimization parameter values for resin yield response

Parameter	Sequential P-value	Lack of Fit P-value	Adjusted R ²	Predicted R ²	Description
Linear	0,2803	0,0495	0,0589	-0,3508	
2FI	0,2609	0,0532	0,1395	-0,9227	Suggested
Quadratic	0,3563	0,047	0,179	-1,8641	
Cubic	0,0216	0,4331	0,7479	-1,3244	

Based on the results of the analysis using design expert 11, the recommended model is 2FI. The 2FI model was chosen because the difference between adjusted R² and predicted R² is lower than other models. This is supported by the lack of fit value which is not significant and higher than the lack of fit value of other models. The following is the 2FI model equation:

$$y_5 = -2,549 + 0,090X_1 - 0,058X_2 + 0,047X_3 - 0,0367X_1X_2 - 7,7 \times 10^{-4}X_1X_3 - 2,6 \times 10^{-3}X_2X_3 \dots\dots(5)$$

Description:

y_5 = Resin yield response value (%)

X_1 = temperature (C)

X_2 = time (hours)

X_3 = stirrer speed (rpm)

Based on the results of the significance analysis of the effect of variables on the response, it was found that none of the variables had a significant effect on the resin yield response. Figure 6 shows the contours of resin yield at various temperatures, extraction times, and stirrer speeds.

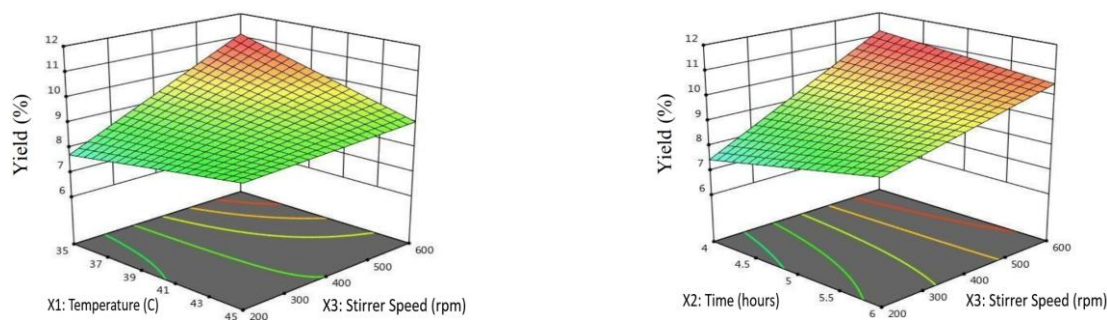


Figure 6 Contours of resin yield at various process conditions

Based on Figure 6, the resin yield has no significant effect on temperature, time and stirring speed, so it can be said that each combination of temperature, time and stirring speed produces yields that are not significantly different. The solvent (methanol) ratio factor has a significant effect on resin yield, the higher the solvent (methanol) used, the higher the resin yield [15]. In addition, the more solvent (methanol) used, the surface contact area increases and the mass transfer between the solid and the solvent also increases so that the solute that can be extracted is higher [22].

Based on the results of the analysis using design expert 11, the optimum resin yield obtained was 10.91% obtained at a temperature of 36.35°C, an extraction time of 4 hours and a stirrer speed of 591.78 rpm.

3.2.2 Acid Number of Resin

The acid number of the resin produced in this study was 98.86-181.24 mgKOH/g Based on the results of sequential analysis, lack of fit, adjusted R², and predicted R² using design expert 11 (Table 7), the recommended model for optimizing process conditions with acid number response is a linear model.

Table 7 Optimization parameter values for resin acid number response

Parameter	Sequential P-value	Lack of Fit P-value	Adjusted R ²	Predicted R ²	Description
Linear	0,3942	0,0445	0,0091	-0,4854	Suggested
2FI	0,7722	0,0294	-0,1223	-2,1551	
Quadratic	0,976	0,0128	-0,4301	-4,3287	
Cubic	0,0216	0,0048	0,3838	-1,8997	

Based on the results of the analysis using design expert 11, the recommended model is a linear model. The linear model was chosen because the difference between adjusted R² and predicted R² is lower than other models. The following is the linear model equation:

$$y_6 = 60,914 - 1,403X_1 - 6,562X_2 - 0,022X_3 \dots\dots\dots(6)$$

Description:

y_6 = Resin acid number response value (mgKOH/g)

X_1 = temperature (C)

X_2 = time (hours)

X_3 = stirrer speed (rpm)

Based on the results of the significance analysis of the influence of variables on the response, it was found that none of the variables had a significant effect on the resin acid number response. Figure 7 shows the contours of resin acid number at various temperatures, extraction times, and stirrer speeds.

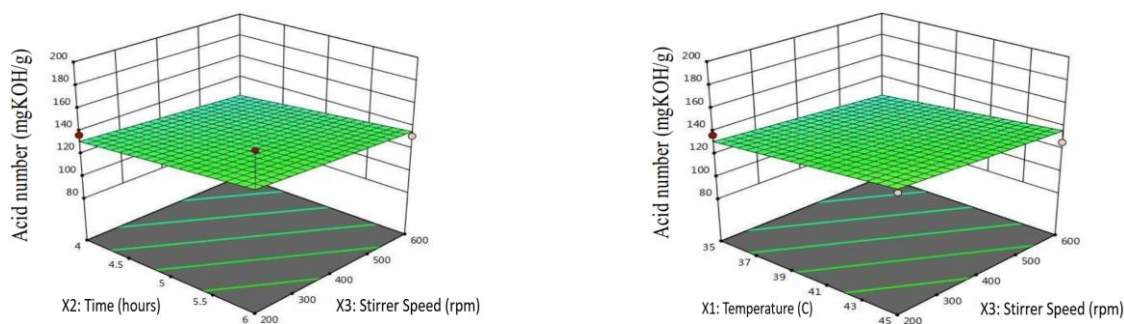


Figure 7 Contours of resin acid number at various process conditions

3.3 Optimization of Oil and Resin Quality

The oil quality parameters tested were oil yield, acid number, density, and viscosity, while the resin yield and acid number. The oil yield obtained in the study was 46.26 – 56.19%. The yield is not much different from the results of research that has been done with a value of 58.2%. The acid number of oil obtained in this study was 5.23 – 22.18 mgKOH/g. This value is supported by the results of research which states that the acid number of nyamplung oil ranges from 6 – 75 mg KOH/g [24].

The oil density obtained in this study ranged from 0.912 – 0.98 g/ml. This value is close to the results of research that has been done with the results obtained, namely 0.89 – 0.91 g/ml [21]. The viscosity of the oil obtained in the study amounted to 5.24 – 22.21 cP. The viscosity of the oil obtained is smaller than the results of research that has been done with a value of 26.48 – 41.88 cP [25], because the oil still contains residual hexane solvents that make the viscosity of the oil in the study low.

The resin yield obtained in the study was 7,1 – 11,16%. The yield is smaller than the results of research that has been done before which is around 16% [25]. This is influenced by the ratio of solvents used, methanol solvent used in this study is less than hexane solvent, so the solvent ratio factor (methanol) significantly affects the resin yield, the higher the solvent (methanol) used, the higher the resin yield [15]. The acid number of the resin obtained in this study was 98.92 – 178,13 mgKOH/g. This value is not much different from the results of research that has been done with the obtained value of 106.16-146.68 mgKOH/g [25], and other studies with a value of 104.0 - 123.5 mgKOH/g [26].

3.4 Optimization and Validation of Optimum Process Conditions

Optimization of oil yield with resin yield constraint is aimed at optimizing oil and resin yield simultaneously. In this simultaneous optimization process, a compromise was made by considering the constraints as shown in Table 8. In Table 8, oil and resin quality were not considered as constraints because none of the models had a significant effect on the response, so that regardless of temperature, extraction time and stirring speed, the values were not significantly different. The process conditions that optimize oil and resin yields are temperature of 40°C, extraction time of 5 hours and stirring speed of 500 rpm with response values as in Table 9. The optimized extraction time is also supported by previous research which states that the best oil and resin extraction time is around 5 hours compared to 7 hours because in these conditions there is almost equilibrium, so the ability of the solvent to extract oil and resin has decreased [21]. A temperature of 40°C is optimal enough to extract oil from nyamplung seeds [15].

Table 8 Limiting optimization of oil and resin extraction operating conditions

Parameter	Goal	Lower	Upper
Temperature (C)	In range	35	40
Extraction time (hours)	In range	4	6
Stirring speed (rpm)	In range	200	600
Oil yield (%)	Maximize	46,26	56,19
Resin yield (%)	Maximize	7,1	11,16
Oil acid number (mgKOH/g)	None	5,23	22,18
Oil density (g/ml)	None	0,912	0,98
Oil viscosity (cP)	None	5,24	22,21
Resin acid number (mgKOH/g)	None	98,92	178,13

Table 9 Optimal value of simultaneous optimization results

Respon	Nilai
Oil yield (%)	52,43
Resin yield (%)	9,56
Oil acid number (mgKOH/g)	8,992
Oil density (g/ml)	0,953
Oil viscosity (cP)	6,575
Resin acid number (mgKOH/g)	138,84

Validation aims to see the suitability of prediction results based on the model with actual conditions. The process conditions applied in this validation are temperature 40°C, extraction time 5 hours and stirring speed 500 rpm. Comparison of prediction and validation results can be seen in Table 10 below:

Table 10 Comparison of prediction and validation values of optimization results

Respon	Prediction	Validation	Difference
Oil yield (%)	52,43	51,09	2,55
Resin yield (%)	9,56	9,75	1,98
Oil acid number (mgKOH/g)	8,992	10,51	16,88
Oil density (g/ml)	0,953	0,94	1,39
Oil viscosity (cP)	6,575	4,47	32,01
Resin acid number (mgKOH/g)	138,84	142,79	2,84

The difference (%) is one of the parameters used to see the accuracy of the validation results obtained against the prediction results. The smaller the difference value, the closer the validation value is to the prediction value. A good difference value is < 5% which indicates the level of accuracy of validation results against predictions > 95% [18]. Responses with difference values < 5% are oil yield, resin yield, oil density, and resin acid number, while responses with difference values > 5% are oil acid number, and oil viscosity. Although there are responses with a difference value of > 5%, this value is still acceptable because the validation value of each response is still in the prediction interval (PI) range recommended by the design expert software, which can be seen in table 11.

Table 11 Comparison of validation value against prediction interval (PI)

Respon	Validasi	95% PI	
		Lower	Upper
Oil yield (%)	51,09	50,15	60,50
Resin yield (%)	9,75	6,51	12,15
Oil acid number (mgKOH/g)	10,51	1,22	20,44
Oil density (g/ml)	0,94	0,92	0,98
Oil viscosity (cP)	4,47	2,16	10,1
Resin acid number (mgKOH/g)	142,79	92,99	188,67

4. CONCLUSION AND SUGGESTIONS

Optimization of process conditions for simultaneous extraction of oil and resin from nyamplung (*Calophyllum inophyllum* L.) seeds using a mixture of hexane and methanol solvents resulted in oil yields of 52.43% and resin of 9.56% obtained at a temperature of 40°C, extraction time of 5 hours and stirring speed of 500 rpm. Optimization with the aim of maximizing the oil yield response obtained a yield of 55.33% at a temperature of 45°C, extraction time of 6 hours and stirring speed of 600 rpm. Optimization with the aim of maximizing the resin yield response obtained a yield of 10.91% at a temperature of 36.35°C, extraction time of 4 hours and stirring speed of 591.78 rpm. Optimization of each quality of oil and resin does not produce a significant model of the response. Oil quality optimization results simultaneously oil and resin yields are oil acid number 10.51 mgKOH/g, oil density 0.94 g/ml, and oil viscosity 4.47 cP, while for resin is resin acid number 142.79 mgKOH/g.

The hexane/methanol ratio needs to be increased to improve oil and resin yields. Then the oil and resin need to be analyzed for the content of phenolic compounds, polyphenolics, phthalic acid esters, iodine number, saponification number and total phenol content.

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