Journal of Vocational Studies on Applied Research

The Effect of Moisture Content on Reducing the Free Fatty Acid Content of Nyamplung Seed Oil *(Callophylum inophyllum)* Using Factorial Design Method

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Abstract – *Nyamplung seeds (Calophyllum inophyllum) have an oil content of 50–70%, in Indonesia nyamplung seeds have a high productivity of 20 t/ha. The process of extracting nyamplung seed oil chemical process produces a higher yield than mechanical. Extraction was collected using the soxhletation extraction method because the oil can be extracted perfectly and requires little solvent. The FFA content is very high, ranging from 15-30% which causes high oil quality to decrease. The high FFA content is due to the high % water content (23-25%) in nyamplung seeds, which causes a hydrolysis process where triglycerides dissociate in the presence of water, glycerol and FFA. The research variables are the moisture content of nyamplung seeds (8% and 12%), the size of the material (15 and 25 mesh), and the ratio of the material to the solvent (1:4 and 1:6) using the factorial design method for research optimization using variable moisture content Nyamplung seed is 8%, size is 20 mesh, and raw material to solvent ratio is 1:5. Novelty value research is effect of % water content on reduction of free fatty acid(FFA) levels contained nyamplung seed oil. Whereas in previous research Adenuga et al., (2021) % water content was not the main parameter for reducing free fatty acid levels. But the main parameters are only drying temperature and oven temperature. FFA content without reducing the % water content of 24 mg KOH/g Jahirul et al. (2015) and 29.2 mg KOH/g Hasibuan et al. (2013) whereas the results of the study showed an FFA level of 13.30%, which means that there was a decrease in FFA levels directly proportional to the decrease in the moisture content of nyamplung seeds.*

Keywords: *crude oil; nyamplung seed; extraction; free fatty acid*

Received: March 10, 2023 Revised: June 07, 2023 Accepted: June 12, 2023

Doi: http://dx.doi.org/10.14710/jvsar.v5i1.17619

[How to cite this article: Pambayun, F., and Broto, R.T.D.W. (2023). The Effect of Moisture Content on Reducing the Free Fatty Acid Content of Nyamplung Seed Oil *(Callophylum Inophyllum)* Using Factorial Design Method. *Journal of Vocational Studies on Applied Research, 5*(1), 31-35. doi: http://dx.doi.org/10.14710/jvsar. v5i1.17619]

INTRODUCTION

Nyamplung has not been fully utilized by the people of Indonesia. Government programs regarding biofuels that are used as renewable energy, such as biodiesel. Budiman (2004), the seeds of nyamplung are round and contain resin on the skin, essential oils, calophylloids, calophyllic acids, sitosterol, musilago glycerin, fatty oils, tannins, takaferols, and carotenoids with an oil content of 50-70% and a water component by 20-30% Hapsari et al., (2022). According to Udarno (2019), the content of nyamplung seed oil is dominated by unsaturated fatty acids with 37.57% oleic acid and 26.33% linoleic acid, so that nyamplung seed oil is well used as a pharmaceutical product and beauty product, as well as a raw material for making biodiesel. Extraction of nyamplung seed oil can be done mechanically by pressing or chemically by extraction. Bhuiya et al., (2020). The extraction used in this study was a hot liquid-solid extraction method, namely soxhletation extraction. Extraction is taking the extract with the help of a solvent. The choice of solvent is determined based on the characteristics of

nyamplung seed oil Leksono et al., (2018). Nyamplung seed oil is classified as a non-polar compound, so the right solvent is also a non-polar compound. This study used the solvent petroleum ether because it has a non-polar compound, is cheap and stable, and has a relatively low boiling point Kartika et al., (2012).

Manurung et al., (2018), parameters commonly used to determine oil quality are % water content, free fatty acid (FFA) content, acid number, iodine number, and peroxide value. Nyamplung seed oil has a high free fatty acid (FFA) content of around 20% Bhuiya et al., (2020). High levels of fatty acids are an indicator of damage to nyamplung seed oil. Candra, et al., (2013) Damage occurs due to natural reactions that occur in nyamplung seeds. The reaction that occurs in nyamplung seeds is a hydrolysis reaction, according to Rantawi et al., (2017). The reaction takes place between triglycerides and water, and the lipase enzyme, which functions as a natural catalyst in the reaction, acts by breaking the ester bonds present in nyamplung seed oil, then producing free fatty acids and glycerol. The iodine number, according to Hasibuan and Siahaan (2013). Indicates the degree of unsaturation of oils and fats. In addition, the iodine number can also classify oil into 3 parts, namely non-drying oil, semi-drying oil, and drying oil. Ochigbo and Ikechukwu (2011). According to Sanjihani et al., (2015), the acid number analysis is due to the formation of aldehydes as a result of the oxidation of unsaturated fatty acids. Then the aldehyde will be further oxidized to form a carboxylic acid.

To reduce the level of damage or free fatty acids in nyamplung seed oil based on the reaction, pre-treatment can be carried out by reducing the % water content to reduce the hydrolysis reaction that occurs or deactivating the lipase enzyme. The novelty value of our research is that it shows that there is an effect of % water content on the reduction of free fatty acid levels contained in nyamplung seed oil. Whereas in previous research Adenuga et al., (2021) % water content was not the main parameter for reducing free fatty acid levels. But the main parameters are only drying temperature and oven temperature. In the process of extracting nyamplung seed oil, the most influential thing on the level of damage to nyamplung seed oil (free fatty acids) is the moisture content of nyamplung seeds. This study aims to (1) determine the effect of % water content on the free fatty acid content of seed oil *(Calophyllum inophyllum)* using the soxhletation method. (2) knowing the main effect and interaction that most influences the product yield of the best FFA *(Calophyllum inophyllum)* content; and (3) knowing the optimum operating conditions so that crude oil *(Calophyllum inophyllum)* with the best grade is obtained. (4) utilizing Nyamplung seed waste that does not pass the minimum specifications for factory raw materials.

METODOLOGY

Materials and tools

The materials used are Nyamplung seeds from Bantul, DIY, petroleum ether, and aquadest. The tools used in this study were filter paper, a series of soxhletation and distillation tools, an Erlenmeyer grinder and sizing oven, an analytical balance, a burette, a measuring cup, a measuring flask, a magnetic stirrer, and a pH meter.

Method

Sample Preparation

The nyamplung seeds were peeled, and then the % water content was reduced according to the research variables using an oven at 80℃ using the gravimetric method until the % water content of the nyamplung seeds became between 8% and 12%. The nyamplung seeds are put into a grinding machine to break them into smaller pieces, which are then sized according to the variables, namely 15 mesh and 25 mesh.

Extraction dan distillation

Nyamplung seed powder was extracted using a soxhletation method with 99% petroleum ether solvent at 60℃, four times solvent circulation, and a material to solvent ratio of 1:5. The nyamplung seed oil extract was then distilled to remove the solvent contained in the extract. The simple distillation process was carried out at 60℃ for 50 minutes.

Analysis FFA.

5 gr crude nyamplung in to 250 mL erlenmeyer, add 50 mL 96% ethanol PA. Add 3 drops fenolftalein indicator and titrate with 0.1 N NaOH until it turns pink. Then it is calculated using the equation:

$$\text{FFA} = \frac{56{,}1 \times V \times N}{W} \qquad (1)$$

Information :

V = Volume of titration results (mL)
N = Normality of the titration solution
W = Sample weight (g)

RESULTS AND DISCUSSION

Factorial design 2^3 was used in this study with 8 experiments to determine the effect on the process variables used, and the optimum conditions obtained were more precise because interaction factors were included.The most influential variables in this study can be identified by factorial design analysis using the quicker method to calculate the main effect and the interaction effect on the resulting free fatty acids.

Table 1. Main Effect Calculation Results and Interaction Against FFA

Effect	Total
Moisture content (%)	1,57
Size (Mesh)	0,39
Ratio material : solvent	0,44
Moisture content * size	0,56
Moisture content * Ratio material : solvent	0,84
Size (mesh) * Ratio material :solvent	0,11
Moisture content * size (mesh) *Ratio material : solvent	0,28

Main effect FFA content is determined by the moisture content of nyamplung seeds, and the most influential interaction effect is the % water content and the ratio of ingredients to solvents.

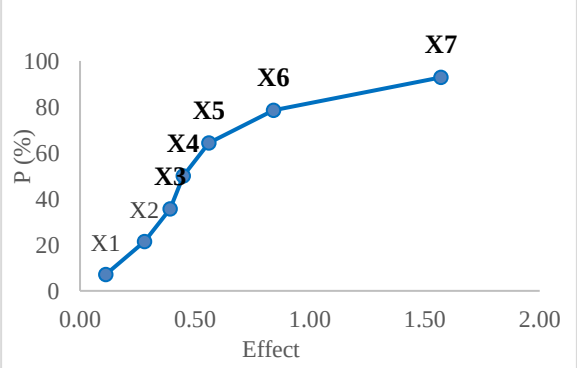

Figure 1. Normal Probability *Plot* 2^3

Normal probability plot graph between the P value and effect obtained by the regression (R^2) is 0.8143 which means that 81.43% of total model variation can represented by regression equation.

$$y = 57.483\,x + 15.449 \qquad (1)$$

So it can be concluded that % water content is the most influential main variables.

Optimization of Nyamplung Oil Crude

Main effect optimization research , the variable moisture content (K) changes while the material size (M) and the ratio of solvent and raw material (R) change.

Results of the free fatty acid (FFA) content in crude nyamplung seed oil have an optimization value, namely with a % water content of 8% nyamplung seed material, with a material size variable of 20 mesh, and a ratio of raw material to solvent 1:5, the FFA content result is 13.30 %. The greater % % water content nyamplung seed, based on results of refference, the greater the FFA value in Nyamplung seed oil

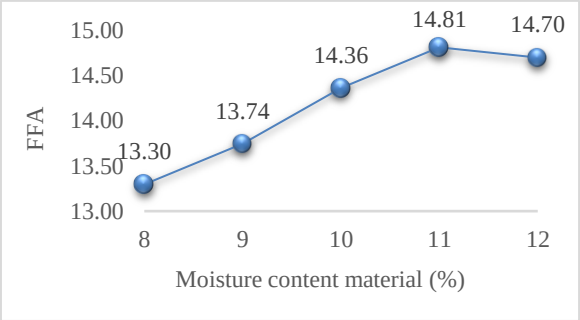

Figure 2. Chart Optimization of Nyamplung Seed Oil Crude

The highest FFA content is shown in the condition of the material, which has a moisture content of 11% to 14.81%. To control the moisture content, the drying process is carried out using the oven method until it reaches the moisture content in accordance with the research variables. Taking nyamplung oil is done using the extraction method because based on research Widiastuti *et al*., (2019) extraction/chemical methods produce more yield than mechanical methods by pressing.

Analysis Free Fatty Acid

Sample optimization Previous research on samples showed FFA values of 33.53% and 29.53%, which were not pre-treated to reduce % water content.

Table 2. Analysis FFA

Analysys	(%)
Optimation variable	13.3 %
Adenuga *et al*., (2021)	10.86 %
Amalia Kartika *et al*., (2017)	14.89 %
Pratama *et al*., (2017)	29,53 %
Candra *et al*., (2013)	8,23 %
Widiastuti *et al*., (2019)	33.79 %

While the previous three research samples had insignificant differences, 8.23%, 10.86%, and 14.89% had already completed the pre-treatment process, namely by reducing the % water content. Based on optimization data and literature, it can be inference that the pre-treatment process of reduce the moisture content of nyamplung seeds can reduce free fatty acid (FFA) levels by 15%–21%. The FFA content of the optimized crude nyamplung seed oil is 13.3%. By reducing the % water content of the raw material for nyamplung seeds, it will inhibit the hydrolysis reaction that occurs in nyamplung seeds; the reaction will take place when there is oil with water with the help of the lipase enzyme. But according to a reduction in % water content that is too large, increasing the drying temperature will also have an impact on the yield of crude nyamplung seed oil because it allows

polymerization, oxidation, and hydrolysis reactions to occur, which will affect product quality such as taste, color, texture, and shelf life. This can be proven by FFA analysis and the peroxide value of crude nyamplung seed oil.

Analysis Acid Number and pH

The optimization of the research shows that the acid number is the lowest compared to the research literature. Whereas in the pH test, the optimization results produce a pH of 5.1, which means that the optimization pH is higher than the literature. High value of the acid number oil, lower its quality, which is an indication of more oil oxidation. The acid number and pH values obtained cannot be simulated to determine whether they meet the requirements or not because there are no quality standard requirements for nyamplung seed oil.

Table 3. Acid Number And pH

Acid Number (mgKOH/gr)	pH	Reference
27.85	5.3	Optimation variabel result
28.81 ± 1.95	4.65	Adenuga et al., (2021)
38.86	4.58	Bhuiya et al., (2020)
59.94	4	Pratama (2017)

Analysis Iod Number

According to (Hasibuan & Siahaan, 2013) the iodine number can be used to indicate the degree of unsaturation of the oil. Analysis of the iodine number shows 69.16 g of iodine per 100 grams of oil and is classified as non-drying oil because the iodine value of Nyamplung seed oil is 100 g of iodine per 100 grams of oil. The lower the iodine number, the less scale will form on the vehicle engine. Crude nyamplung seed oil is suitable for use as a renewable biofuel.

Tabel 4. Analysis Iod Number

Analysys	(g iod/100 gr)
Optimation variable	69.16
Adenuga et al., (2021)	81.67
Amalia Kartika et al., (2017)	60.75
Pratama et al., (2017)	29.53 %
Candra et al., (2013)	8.23 %
Widiastuti et al., (2019)	98.75

CONCLUSION

This study used the factorial 2^3 method design. The results showed that the process variable that greatly influenced the optimization of Nyamplung seed oil extraction was the variable moisture content of the raw material (K), with an effect value of 1.57. The process of optimizing the free fatty acid (FFA) content, namely with the operating conditions of nyamplung seed moisture content of 8%, a material size of 20 mesh, and a ratio of material to solvent weight of 1:5 (60 grams: 300 mL), obtained an FFA content of 13.30%. The function of extracting nyamplung seed oil is to utilize nyamplung seed waste, which is off-spec industrial material and produces an iodine number of 100 g iodine per 100 g, which means it is classified as a non-drying oil. Nyamplung seed oil is good for use as a pharmaceutical and beauty product, and biodiesel raw material with a low iodine number will reduce scale formation on vehicle engines.

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