Journal of Vocational Studies on Applied Research

Optimization of Manufacturing Liquid Soap Based on Virgin Coconut Oil with a Combination of Potassium Hydroxide and Ammonium Hydroxide

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Abstract *– Virgin coconut oil has a better saponification effect than coconut oil in general. It has a high lauric acid content of 46%, suitable for skin moisture; it is good to be used as a primary ingredient for making natural liquid soap; hydrolysis is carried out on VCO to get free fatty acids ingredient raw material for making soap. This study will use a combination of KOH and NH_4OH bases to produce more soluble soap in water. This study aims to determine the quality of natural liquid soap from the saponification process between VCO hydrolysis and base, whether by applicable quality standards, and determine the effect of base concentration, time, and temperature of mixing on the quality of the soap produced. The method in this study uses the Response Surface Methodology, where the resulting soap product is tested for physicochemical tests. The critical value for optimizing liquid soap is obtained at the KOH base ratio of 8, time 140 minutes, and temperature 92^oC, and the critical value of FFA is 0, 21%. The best soap results are sample 6, which complied with SNI 06-4085-1996 and SNI 3532-2016.*

Keywords: *hydrolysis oil; liquid soap; saponification; virgin coconut oil*
Received: June 06, 2022 Revised: June 20, 2022 Accepted: June 22, 2022

Doi: http://dx.doi.org/10.14710/jvsar.v4i1.14463
[How to cite this article: Kurniawati, Y., and Paramita, V. (2022). Optimization of Manufacturing Liquid Soap Based on Virgin Coconut Oil with a Combination of Potassium Hydroxide and Ammonium Hydroxide. *Journal of Vocational Studies on Applied Research, 3*(3), 62-67. doi: http://dx.doi.org/10.14710/jvsar.v4i1.14463]

INTRODUCTION

From a chemical perspective, soap combines fat or oil, water, and alkali organic material that functions as a cleaning product (Chirani et al., 2021). According to Maotsela et al., (2019), there are two types of soap categories it is called natural soap and synthetic soaps are based on the composition of the ingredients used, natural soaps do not produce toxic waste, and the production process does not require minimal amounts of energy. Based on its characteristics, soap is divided into liquid, soft and solid soaps; liquid soap is more soluble in water (Fauzi et al., 2019). The process of soap formation is called the saponification reaction; it is the reaction between potassium and fatty acids from vegetable and animal oil which produces sodium salts from the hydrolysis of free fatty acids and glycerol (Sukesi and Sitorus, 2017).

In general, the alkalis used in soap making are NaOH (solid soap) and KOH (liquid soap) (Sukesi and Sitorus, 2017); based on the quote in Classic Bells, soap made with a combination of NH_4OH solution is more soluble in water than soap made from just KOH. Virgin coconut oil is a pure oil that is good for making liquid soap because it is easily saponified and has a high lauric acid content of 43%-53%; this content has a good effect on the skin moisturizing (Widyasanti et al., 2019). In preparing

raw materials, VCO will be hydrolyzed to obtain free fatty acids as raw materials; oil hydrolysis is a reaction to decompose triglycerides using water as a reagent to obtain free fatty acids (Trivana and Karouw, 2017). The purpose of the research conducted by the author is to determine the process of making natural liquid soap based on VCO hydrolysis and to determine the characteristics of the resulting liquid soap so that it can be seen how much influence the combined concentration of KOH and NH_4OH bases, time and temperature of mixing on the quality of the resulting product.

METHODOLOGY

The primary raw materials used for this research are the hydrolysis of virgin coconut oil, KOH, and NH_4OH; other ingredients such as glycerin, propylene glycol, aqua dest, coco-DEA, and essential oils oil. Several other materials for analysis include 96% ethanol, indicator PP and HCl. The tools used include a thermometer, beaker glass, hydrolysis tools, dropper pipette, measuring cup, universal indicator, stirring rod, magnetic stirrer, volume pipette, and digital balance. The fixed variables in this study were hydrolysis of VCO (50 ml), glycerin (8ml), propylene glycol (21, 5 ml), aqua dest (130 ml), coco-DEA (5 ml), and essential oil (1 ml). The variables changes are a percentage of KOH and NH_4OH concentrations of 25 ml; 50 ml; 75 ml, mixing time of 60 minutes; 90 minutes; 120 minutes, and a mixing temperature of 65^{o}C; 75^{o}C, and 85^{o}C. This research experiment uses the Central Composite Design with the Response Surface Methodology method by setting a variable to change so that the design in table 1.

Table 1. Experimental Design with RSM

	Star Low	Low Point	Center Point	High Point	Star High
Base ratio	8:92	25:75	50:50	75:25	92:8
Mixing time	40	60	90	120	140
Temperature	58	65	75	85	92

RESULT AND DISCUSSION

The raw material is in virgin coconut oil; before reacting with bases, the preparation is done in oil hydrolysis. From the result of the hydrolysis carried out, it was found that the free fatty acid content increased from 1.52% to 2.12%; with an increase in the free fatty acid content, the amount of lauric acid in its constituent components increased, where this lauric acid has a good effect on the skin like moisture and anti-microbial (Widyasanti et al., 2017).

Physicochemical Analysis Results of Liquid Soap

The result of the physicochemical test on liquid soap can be seen in table 2. Table 2 shows the test results carried out, such as pH, specific gravity, viscosity, free fatty acids, and foam stability against the soap produced in the study.

Table 2. Physicochemical Test Result of Liquid Soap

Sample	KOH (ml)	NH_4OH (ml)	Temperature (^{o}C)	Time (minute)	pH	Specific gravity (g/ml)	Viscosity (cP)	Foam stability (cm)	Free fatty acid (%)
1	25	75	65	60	8	1.0476	28.58	30	2.870
2	25	75	85	60	8	1.0472	39.28	20	1.312
3	25	75	65	120	9	1.0512	30.47	15	1.230
4	25	75	85	120	8	1.0476	61.62	30	0.820
5	75	25	65	60	9	1.0867	30.57	60	2.665
6	75	25	85	60	9	1.0984	351.15	50	2.460
7	75	25	65	120	9	1.0823	69.20	70	0.820
8	75	25	85	120	8	1.1016	322.13	55	3.075
9	8	92	75	90	8	1.0423	79.97	5	1.640
10	92	8	75	90	9	1.0984	134.84	70	0.410
11	50	50	75	40	8	1.0815	71.91	65	2.911
12	50	50	75	140	9	1.0811	27.65	55	0.738
13	50	50	58	90	9	1.0605	22.60	20	1.640
14	50	50	92	90	8	1.0774	50.52	70	0.943
15	50	50	75	90	8	1.0516	25.10	70	2.665
16	50	50	75	90	8	1.0516	25.10	70	2.665

The pH value is an essential indicator in soap because it is related to the irritating nature of the skin; a good soap has a pH that is not far from pH (5,5-5,6). pH values that are too alkaline cause dry skin and damage microorganisms that maintain the skin surface (Hardiana, 2016). In 16 samples of liquid amount of KOH used will cause the pH value to be higher in the soap. Differences influence in pH value in the sample in base concentration where alkali that does not react perfectly in the saponification process will cause a high pH value, mixing time and temperature, increasing stirring time makes the pH value down (Hasibuan et al., 2019).

Specific gravity analysis aims to determine the effect of the ingredients used to manufacture liquid soap. According to SNI 06-4085-1996, the specific gravity of liquid soap ranges from 1.010-1.100 gr/ml. Although the analysis results for 16 samples of liquid soap showed that they were following SNI, from the results shown in the table, the addition of temperature tended to decrease the specific gravity (Wrasiati et al., 2019).

According to Widyasanti et al., (2019), viscosity depends on the viscosity of the solvent, the contribution of dissolved materials, and the integration of the two, the less water content in soap, the higher viscosity analysis on 16 samples obtained viscosity values ranging from 22.6012 cP to 351.15 cP. The foam in soap is an essential component because the foam produced is too high; it can dry out the skin and cause the skin to be prone to irritation (Hutauruk et al., 2020). According to SNI 3552-2016, the requirements for the height of liquid soap foam are 13-220 mm; in the table, it can be seen that 16 samples of liquid soap have met the standard for the foam height value, soap with the highest foam is found in samples 7, 10, 14, 15, 16 and the lowest in sample 9 it is also influenced by the concentration of KOH added to the manufacturing process.

Free fatty acids are one of the quality standards for soap because if the soap contains fatty acids that are too high, it will cause an unpleasant soap odor, less attractiveness, and short shelf life (Fauzi et al.,2019). Widiyawanti and Dewi (2020) conducted a similar study with the same primary VCO ingredient. In that study, KOH was varied at 20%; 30%; 40%, and 50%; it was found that free fatty acids would be higher as the concentration of KOH increased; besides that, the longer the stirring time will make, the results smaller while temperature has a direct influence on the number of free fatty acids produced. The analytical method used in acidimetric titration uses a standard base solution to determine acid. The analysis showed that the liquid soap following SNI was sampled 2, 3, 4, 6, 7, 9, 10, 12, 13, and 14 with no more than 2,5%. The highest levels of fatty acids are in sample 10. Free fatty acids are obtained from fat decomposition by water molecules producing glycerol. Therefore, free fatty soap, the pH values ranged from 8-9, according to SNI 3532-2016 (pH value 10-14). Previous research conducted by Agusta (2016), found that the smallest pH was in the conditions of VCO 4g and KOH 3g. The highest pH was at VCO 3 g and KOH 4 g, which can be concluded that the higher acids, free fatty acids, are part of the acid number, which helps determine the level of oil damage (Bidilah et al., 2017).

Analysis of Response Surface Design of Free Fatty Acids (FFA) in Liquid Soap

In analyzing the results of the study between free fatty acids and independent variables, surface design analysis was used on the Minitab 19 application to determine the effect of independent variables (base concentration, time, and temperature); it is known that the maximum free fatty acid content in the research result is 2.665%-3.075% and the minimum yield is 0.410%. The model summary of the research can be seen in table 3.

Table 3. Model Summary Response Surface Regression of Free Fatty Acids

S	R-sq	R-aq(adj)	R-sq(pred)
0.795879	41.59%	26.99%	0.00%

From table 3, the interpretation of the data is made by looking at the R-square value, where the value is sensitive to changes in the independent variable, and the R-square tends to increase if there are additional variables. The results obtained for the R-square adjust the value of 29, 99%, meaning that the independent variables influence 29, 99% of the value of free fatty acids in the study. The results of the analysis of variance are shown in table 4.

A variable is said to have a hypothetical effect on a free fatty acid level if the P-value of a variable is <0, 05, while it has no effect if the P-Value is >0, 05. Based on the table data above, it is known that the time variable affects the hypothesis. In contrast, the base concentration and temperature variables for partial hypothesis testing do not significantly affect the results. Therefore, the regression equation can be written :

% Free fatty acid=3.24+0.01423 base-0.0080 temperature – 0.017171 time (1)

From the data that has been analyzed, a Pareto chart can be mad to identify significant factors that affect the value of free fatty acid content of the study results. The Pareto chart can be seen in Figure 1. Based on the Pareto chart, the most significant factor influencing is time. The base concentration variable gives a large enough effect and temperature is slightest effect on the free fatty acid yield on the result. The base variable significantly influences the

free fatty acid levels in this results; this is related to where the fatty acids compounds. A similar study was conducted by Widiyati and Dewi (2020), where the concentration of KOH affected the levels of free fatty acid produced; the research showed that the concentration with the resulting free fatty acid was directly proportional. The The temperature has a minor effect compared to the time and base concentration; this can happen because the temperature is too high or low. Where the increase in reaction temperature will increase. The temperature needed to suppress the reaction towards the formation of free fatty acids, but if the temperature to high, it will not affect the formation reaction; this previous research has been carried out by Widiyanti and Dewi (2020).

Table 4. Analysis of Variance Response Regression of Free Fatty Acids

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Regression	3	5.4132	1.80441	2.85	0.082
X_1 (KOH)	1	1.7270	1.72705	2.73	0.125
X_2 (Suhu)	1	0.0883	0.08827	0.14	0.715
X_3 (Waktu)	1	3.5979	3.59790	5.68	0.035
Error	12	7.6011	0.63342		
Lack-of-Fit	11	7.6011	0.69101	*	*
Pure Error	1	0.0000	0.00000		
Total	15	13.0143			

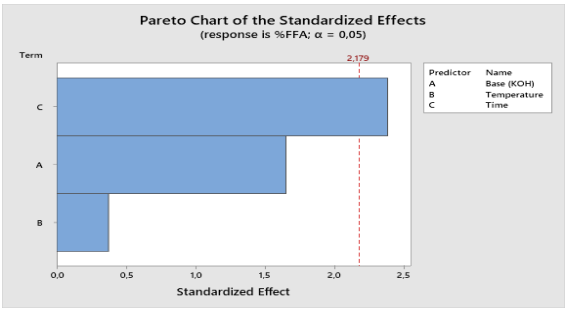

Figure 1. Standard Pareto Chart of Effect

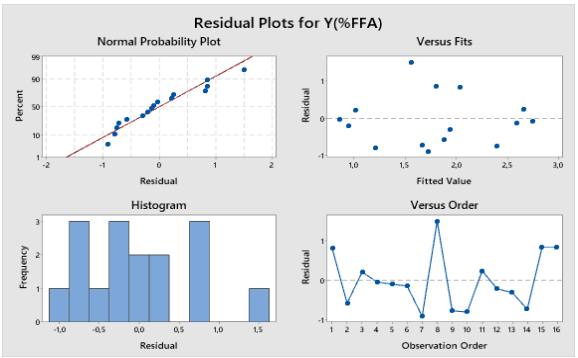

Figure 2. Residual Plot of Free Fatty Acid

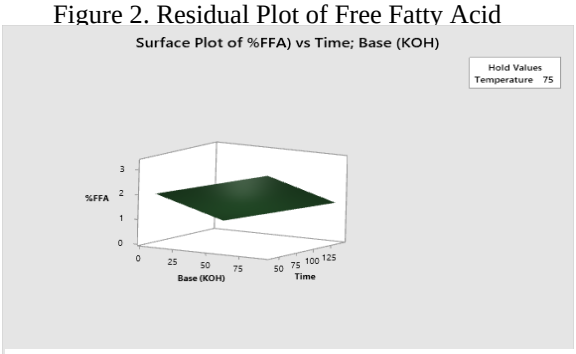

Figure 3a. Analysis Effect Time and Base Concentration on Free Fatty Acid Levels

After the regression analysis, observations were made on the residual free fatty acid plot, where the residual is the difference between the dependent variable and the prediction. The result of the residual plot is shown in figure 2. Based on the results of the residual plot, several points can be observed; the histogram graph shows the residuals usually contribute. On the standard probability graph, the plots are not randomly distributed; follow and approach a straight line so that it can be interpreted that the residual plot is usually distributed. The fits versus order graph show the plot spreads evenly above and below the axis without forming a pattern which means there are no symptoms of heteroscedasticity. Finally, contour and surface plots can be used to analyze the effect of independent variables on the free fatty acid contained in the research samples. The results of the analysis can be seen in figures 3(a,b) and 4(a,b).

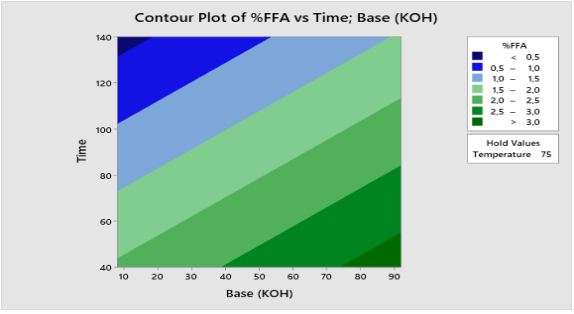

Figure 3b.Analysis Effect Time and Base Concentration on Free Fatty Acid Levels (Surface Plot)

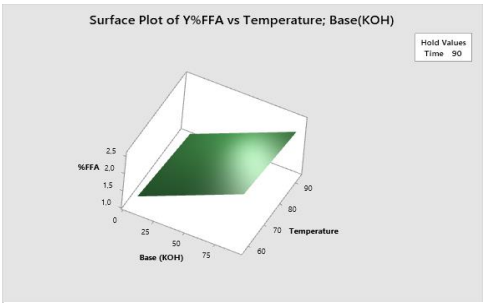

Figure 4a. Analysis Effect Temperature and Base Concentration on Free Fatty Acid Levels (Surface Plot)

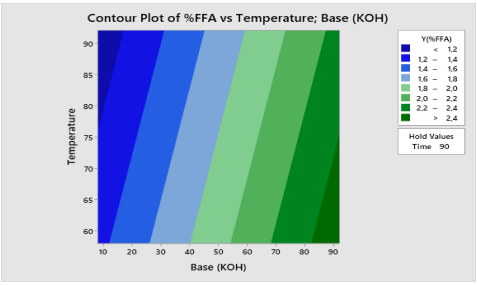

Figure 4a. Analysis Effect Temperature and Base Concentration on Free Fatty Acid Levels (Contour Plot)

Figures 3 and 4 show the contour plot of the effect of independent variables on yield (free fatty acid). In figure 3, it can be seen that the dark blue color (content < 0, 5) to light green (2-2, 5) at a temperature of 75ºC is following SNI. In the comparison between the time and the concentration of KOH, it can be seen that the smaller the concentration of KOH and the longer the mixing time the lower levels of free fatty acids (FFA) produced; thus is because the free fatty acid levels and KOH concentration are directly proportional to the stirring time. Bidilah et al.,(2017) conducted a similar study regarding the time and concentration of KOH soap; the result obtained that the longer time, the lower free fatty acid content. Figure 4 shows that the conditions produced according to SNI are marked with dark blue contour color (content<1, 2) to green color (content 2, 2-2, 4) at a time condition of 90 minutes on the contour plot. It can be seen that the higher of temperature, the FFA produced lower; research conducted by Widiyati and Dewi (2020), obtained results where temperature too high will not affect the formation, so temperature too high or too low make free fatty acid results not optimal.

The optimization of liquid soap with independent variables of base ratio, mixing time, and temperature is determined by response optimization in table 5. The critical value for optimization of liquid soap is obtained at 8 ml KOH base ratio, which means the ratio of NH_4OH is 92 ml; time 140 minutes and temperature 140ºC and obtained a minimum critical response of 0,2115% fatty acids.

Table 5. Response Optimization Free Fatty Acid Levels

Solution	KOH (ml)	Temperature (ºC)	Time (minute)	FFA (%)	Composite Desirability
1	8	92	140	0.211598	1

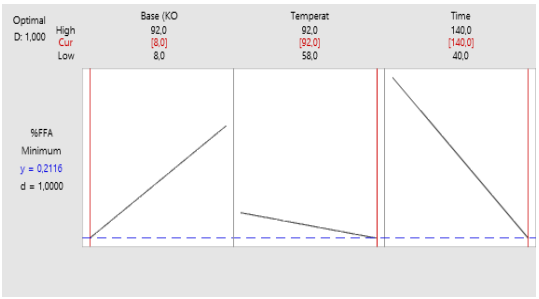

Figure 5. Prediction Response Minimum Value of Free Fatty Acid Levels

CONCLUSION

From the research that has been done, physicochemical tests, and analysis of the results using the Minitab 19 software, it is possible to determine the sample of liquid soap with the best quality following SNI 06-4085-1996 and SNI 3552-2016. The best sample was found in sample 6 with the addition ratio of 75 ml of KOH, 25 ml of NH_4OH, mixing time of 60 minutes with a temperature of 85ºC. The level of free fatty acids in sample 6 was 2,460% which is still below the SNI standard of 2,5%. The independent variable that most influenced the yield of free fatty acids in soap was time; then, the ratio of bases gave a large enough effect, and the temperature had the slightest effect on the yield.

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