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Production of Liquid Soap from Virgin Coconut Oil with the Active Ingredient Citronella Oil

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Abstract
The use of hand-washing soap is able to kill germs because it can remove oil and dirt that sticks to the hands. In making this liquid soap, active substances from nature, which is citronella oil, are used as anti-bacterial and anti-fungal agents. This study aims to study the factors that influence the response of free alkali and optimum conditions in the manufacture of liquid soap. The optimization method uses Response Surface Methodology (RSM) based on Central Composite Design. Experimental variables were carried out at reaction temperature (60-80°C), KOH concentration (30-50%), and reaction time (30-50 minutes). The results of the analysis using statistical software obtained the optimum operating conditions for the reaction time of 56,818 minutes, KOH concentration of 46,818%, and reaction temperature of 86,818°C with a free alkali value response of 0.04158%. These results meet the SNI 06-4085-1996 standard with a free alkali value below 0.1%.

Keywords : *Liquid Soap, Citronella Oil, VCO, RSM*

INTRODUCTION

Population growth has increased daily needs, and one of them is the need for soap (Widyasanti et al., 2017). To wash hands cleanly, soap is needed which will remove dirt and bacteria attached to parts of the human body. This attached fat and dirt causes disease germs to grow (Annita et al., 2022).

Soap is an organic salt compound that has function as a body cleanser from dirt that comes from outside the body and from within the body such as dust and sweat. There are 2 types of soap, namely solid soap and liquid soap. The main ingredients for making soap are oils/fats and base compounds. Alkaline compounds that can be used in soap making are NaOH for making solid soap and KOH for making liquid soap (Sanharis & Wahyuningtyas, 2018). The saponification reaction in soap making is divided into two processes, namely the hot process and the cold process. The cold process is carried out at room temperature (25°C) or without heating, while the hot process involves heating at 60 to 80°C (Sastrawidana et al., 2020).

Liquid soap is a liquid preparation that is used to clean dirt, made from soap-based ingredients with the addition of surfactants, foam stabilizers, preservatives, dyes, and fragrances that are permitted and do not cause irritation to the skin (Sari et al ., 2019). In addition, the good quality of liquid body soap is influenced by raw materials such as Virgin Coconut Oil (VCO). The high content of lauric acid and oleic acid in VCO has good benefits for skin health such as natural moisturizers for the skin and has antibacterial properties (Purnamasari, 2020).

The main ability of liquid body wash as a cleanser needs to be added to natural ingredients which provide multiple benefits such as counteracting free radicals and preventing bacterial and microbial infections (Sukeksi et al., 2018). Lemongrass oil contains lemon or citral, which have anti-fungal and anti-microbial properties, and give it a distinctive lemon odor (Jalaluddin et al., 2018). Therefore, citronella oil can be added to soap making. The main compound in lemongrass is a natural antibacterial compound that can inhibit the growth of E. coli and Staphylococcus aureus (Rita et al., 2018). Antibacterial compounds are found in phenolics and flavonoids which have antimicrobial, antioxidant, and antibacterial functions (Adiwibowo, 2020). The addition of methyl paraben and propyl paraben is also needed as a supporting material in making soap which has function as a preservative to prevent the growth of microbes such as bacteria and fungi (Anggraeni et al., 2019).

Testing the characteristics of liquid soap is carried out to determine the quality of the liquid soap produced. This characteristic aims to determine the physical and chemical properties of liquid soap and determine the suitability of the resulting liquid soap product. Liquid soap testing is adjusted to the liquid soap quality specifications according to SNI 06-4085-1996 (Widyasanti et al., 2017).

This research studies the optimum operating conditions of the liquid soap manufacturing process and produces liquid soap according to SNI 06-4085-1996 standards. The method used for optimization is the Response Surface Methodology (RSM) based on Central Composite Design (CCD) which is commonly used for experimental design and minimizes the number of trials for certain factors and levels (Chelladurai et al., 2018). This study will discuss the effect of these variables on the optimum value of liquid soap-free alkali.

METHODS

Materials

Citronella oil is obtained from the distiller in Sukorejo, Kendal. Virgin Coconut Oil, KOH, Cocoamide DEA, Glycerin, Propylene glycol, Methyl paraben, Propyl paraben and HCl were obtained from the Indrasari Chemical Store, Semarang.

Methods

25 ml of VCO is added to 1 ml of citronella oil in a beaker glass, then stirred and heated according to temperature variations. KOH solution with a certain concentration of 30 ml is added to the mixture of VCO and citronella oil, then stirred for a certain time. 30 ml of aquadest and 8.13 ml of glycerin were added to the mixture and stirred until homogeneous. 10 ml of propylene glycol was added and stirred until homogeneous, then 6.13 ml of Cocamide DEA was added. Methyl paraben and propyl paraben in the amount of 0.15 g and 0.03 grams were added to the mixture. Liquid soap is left at room temperature and the soap is analyzed for free alkali number.

Experimental design

Optimization in this study uses the Response Surface Method (RSM) based on Central Composite Design (CCD). The independent variables examined in this study were reaction temperature, KOH concentration, and reaction time. CCD design code values and levels are presented in Table 1.

Table 1. Experimental range and factor level of the process variable.

Component	Variables	Pattern Code				
		-α	-1	0	+1	+α
X_1	Temperature,°C	53.18	60	70	80	86.82
X_2	KOH, %	13.18	20	30	40	46.82
X_3	Time, min	23.18	30	40	50	56.82

Optimization With RSM (Response Surface Methodology)

The RSM method is used to obtain data based on reaction time, temperature, and KOH concentration so that the quality of liquid soap meets the maximum SNI standard of 0.1 (SNI, 1996). The experimental design was carried out using Central Composite Design (CCD) with 20 trials using Statistica-10 software.

RESULTS AND DISCUSSION

The production of liquid soap uses VCO and citronella oil active ingredients

The independent variables are shown in Table 1. To express the free alkali value of liquid soap as a function of a set of independent variables, a second-order polynomial equation is used as follows (Patil et al., 2010)

$$y = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{\substack{i=1 \\ i<j}}^{k-1} \sum_{j=2}^{k} \beta_{ij} x_i x_j + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \varepsilon \quad (1)$$

Where y is the response variable, xi and xj are independent variables, βo, βi, βii, βij are the regression coefficients, and k is the number of variables.

In this study, the three independent variables were temperature (X_1), KOH concentration (X_2), and time (X_3) and Y was the response variable (value of free alkali) obtained from Equation 2.

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 \quad (2)$$

Table 2. The observed response of Free Alkali Values in the synthesis of liquid soap

Run	Temperature (°C)	[KOH] (%)	Time (min)	Experiment value,%	Predicted value,%
1	60.00	20.00	30.00	0.005	0.003
2	60.00	20.00	50.00	0.004	0.008
3	60.00	40.00	30.00	0.037	0.036
4	60.00	40.00	50.00	0.025	0.024
5	80.00	20.00	30.00	0.003	0.005
6	80.00	20.00	50.00	0.003	0.005
7	80.00	40.00	30.00	0.040	0.037
8	80.00	40.00	50.00	0.018	0.021
9	53.18	30.00	30.00	0.018	0.017
10	86.82	30.00	40.00	0.018	0.015
11	70.00	13.18	40.00	0.005	0.000
12	70.00	46.82	40.00	0.040	0.041
13	70.00	30.00	23.18	0.016	0.018
14	70.00	30.00	56.82	0.016	0.010
15	70.00	30.00	40.00	0.016	0.011
16	70.00	30.00	40.00	0.012	0.011
17	70.00	30.00	40.00	0.016	0.011
18	70.00	30.00	40.00	0.013	0.011
19	70.00	30.00	40.00	0.009	0.011
20	70.00	30.00	40.00	0.016	0.011

Based on Table 2, the accuracy of the predicted and observed values has been shown so that they can be analyzed to find out the equation of the mathematical model. The data obtained are shown in Table 3.

Table 2. Prediction coefficients for second order polynomial model of Free Alkali Values

Parameter	Predicted Coefficients	Standard. Error.	t	P value
Mean/Inc.	0.011910	0.002439	4.88297	0.004541
X_1	-0.001025	0.003237	-0.31673	0.764248
X_1^2	0.003355	0.003151	1.06474	0.335685
X_2	0.023997	0.003237	7.41422	0.000703
X_2^2	0.006537	0.003151	2.07465	0.092690
X_3	-0.005126	0.003237	-1.58363	0.174129
X_3^2	0.001941	0.003151	0.61590	0.564915
X_1X_2	-0.000250	0.004229	-0.05912	0.955149
X_1X_3	-0.002250	0.004229	-0.53206	0.617470
X_2X_3	-0.008250	0.004229	-1.95088	0.108548
R^2	0.88765	Adjust R^2	0.78653	

Based on Table 3. the value of R^2 is 0.88765. This shows that the model used is accurate because it is close to 1. Adjust R^2 of 0.78653 indicates that the relationship between the independent variable and the response variable is getting stronger. The p-value for the temperature factor (X_1) was 0.764248 and for time (X_3) was 0.174129 which showed no significant effect on the free alkali response because the p-value> 0.05. Based on the interaction effect and significance, the response equation is shown in Equation 3.

$$Y = 0.011910 - 0.001025X_1 + 0.023997X_2 - 0.005126X_3 + 0.003355X_1^2 + 0.006537X_2^2 + 0.001941 X_3^2 - 0.000250 X_1 X_2 - 0.002250 X_1X_3 - 0.008250X_2X_3 \quad (3)$$

Equation 3 is a mathematical model of a second-order polynomial equation with significant coefficients in the model as shown in Table 3. The above equation is not the final equation because there is still an insignificant effect. Temperature linear factor (X_1), time linear factor (X_3), temperature squared factor (X_{12}), KOH concentration squared factor (X_{22}), time squared factor (X_{12}), temperature linear factor to KOH concentration (X_1X_2), temperature linear factor to time (X_1X_3) and the linear factor of KOH concentration over time (X_2X_3) must be ignored so that Equation 3 is simplified to Equation 4.

$$Y = 0.011910 + 0.023997X_2 \quad (4)$$

The effect of interaction between variables was validated using Analysis of Variance (ANOVA) as presented in Table 4. ANOVA determines the significance value of the independent variables that affect the dependent variable. The error tolerance limit (α) used is 5% or 0.05 so that the confidence level is 95% or 0.95. In Table 4, the temperature (X_1) and time (X_2) factors have a significance value of 0.764248 and 0.174129. So that these two factors do not affect the value of free alkali. As for the linear factor, the concentration of KOH (X_2) has a significant effect on free alkali because it has a p-value<0.05, which is 0.000703. The interaction factor between the variables temperature and KOH concentration (X_1X_2), temperature and time (X_1X_3), and KOH concentration and time (X_2X_3) has no significant effect on optimum free alkali because it has a p value > 0.05, which is 0.955149; 0.617470; and 0.108548.

Table 3. Analysis of variance in the production of liquid soap from VCO and the active ingredient citronella

Parameter	SS	df	Ms	F	P
X_1	0.000004	1	0.000004	0.10032	0.764248
X_1^2	0.000041	1	0.000041	1.13368	0.335685
X_2	0.001966	1	0.001966	54.97071	0.000703
X_2^2	0.000154	1	0.000154	4.30416	0.092690
X_3	0.000090	1	0.000090	2.50788	0.174129
X_3^2	0.000014	1	0.000014	0.37933	0.564915
X_1X_2	0.000000	1	0.000000	0.00349	0.955149
X_1X_3	0.000010	1	0.000010	0.28308	0.617470
X_2X_3	0.000136	1	0.000136	3.80592	0.108548
Lack of fit	0.000124	5	0.000025	0.69187	0.652039
Pure Error	0.000179	5	0.000036		
Total SS	0.002693	19			
R^2	0.88765	Adjust R^2	0.78653		

Determination of Optimal Conditions

Optimum conditions in the process of making liquid soap are obtained from statistical software through profiles. The interaction of the three variables is known for its optimum condition by drawing a straight line vertically. A desirability value of 1 indicates that the objective solution has been met, while a desirability value of 0 indicates the response must be discarded (Ramadhani et al., 2017). The optimum conditions for the quality of liquid soap are shown in Figure 1.

The predicted value of the model resulted in a free base response of 0.04158% with optimum conditions of 56.82 minutes, 46.82% KOH concentration and 86.82°C temperature. These results meet the SNI 06-4085-1996 standard for free of alkali, which is a maximum of 0.1%. Based on the response surface, it shows that the reaction time, heating temperature, and KOH concentration can affect the free alkali value and the optimum conditions for the process of making liquid soap from VCO with the active ingredient citronella.

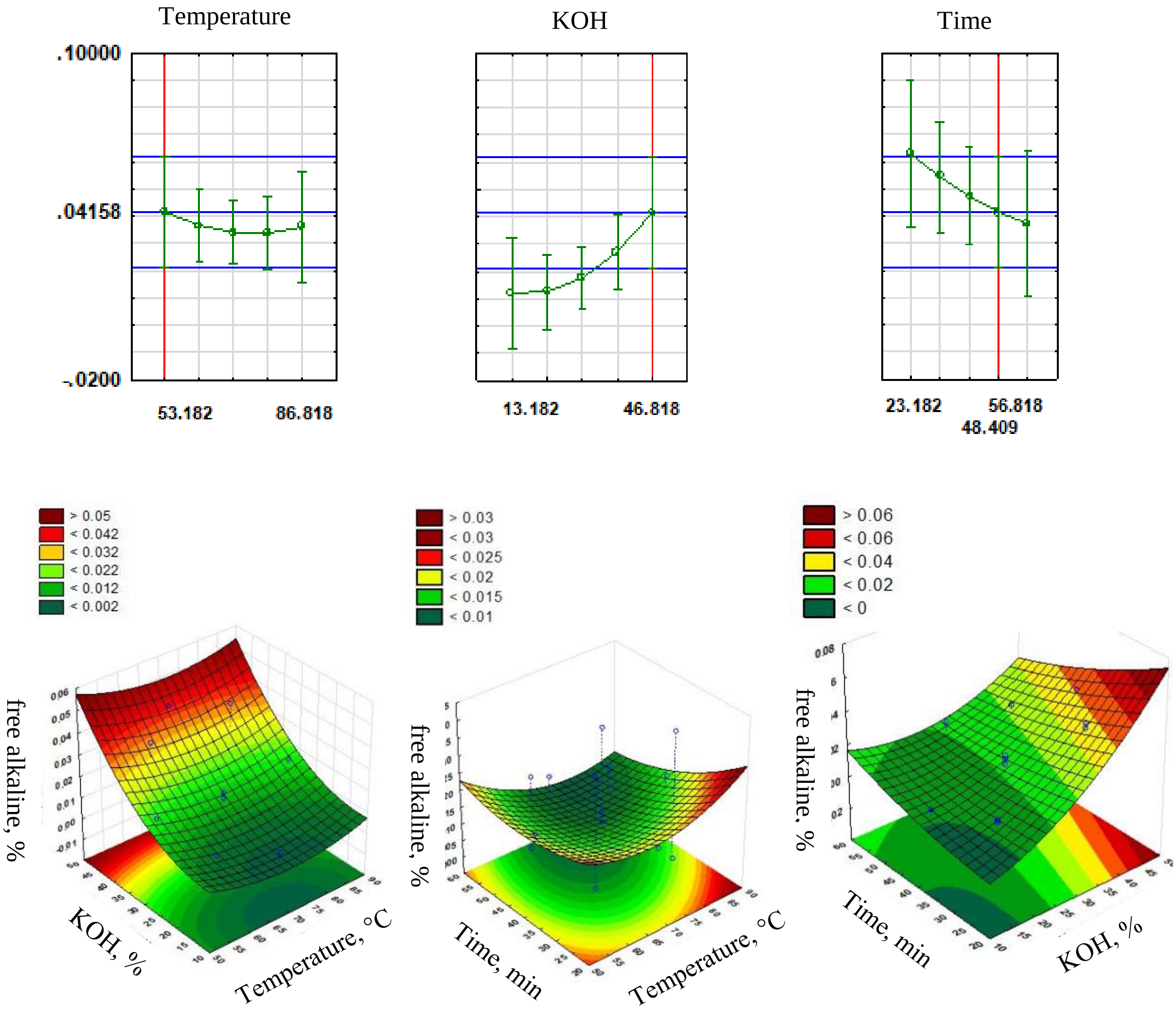

Figure 1. Optimum Process Conditions in the manufacture of Liquid Soap

Optimization of process conditions in the production of liquid soap was validated by running the process and analyzing the free alkaline content. This aims to find out whether there is a match between the experimental value and the predicted value from optimization with RSM. The validation value of the optimization results is shown in Table 5.

Table 4. The Optimum Values of Free alkaline

Variable	Optimum condition	Free alkaline value	
		Predicted	Observed
X_1	86.82		
X_2	46.82	0.041%	0.082%
X_3	56.82		
% error		0.41%	

Based on the data in Table 5, the difference in the response value between the prediction software and the actual data is 0.41%. The difference in these values is less than 5% so the validation results are acceptable. This proves that the free alkaline value from the test results is in accordance with the program's predicted value (Mariana et al., 2017).

Chemical Characteristics of Liquid Soap

The characteristics of liquid soap are presented in Table 6. The results of testing the degree of acidity (pH) of the liquid soap sample showed a value of 9.3-11.72. This value is in accordance with the quality requirements of SNI 06-4085-1996, namely 8-11. The pH value that exceeds the SNI 06-4085-1996 standard is caused by the high concentration of KOH. The higher the KOH concentration, the higher the viscosity, foam, pH, and free alkali content. A large pH value affects the free alkali value because if the resulting pH is high, the free alkali produced will also be high which will affect the quality of liquid soap (Devi et al., 2017). This is appropriate because the resulting free alkali value shows a value between 0.003-0.04%. This value is in accordance with SNI 06-4085-1996, which is a maximum of 0.1.

Table 5. Chemical Characteristics of Liquid Soap with the active ingredient citronella oil

Characteristics	Analysis	SNI 06-4085-1996	Information
pH	9.3 – 11.72	8-11	Fulfill
Free Alkaline,%	0.003-0.04	max. 0.1	Fulfill
Specific gravity, g/ml	1.02-1.107	1.01 – 1.10	Fulfill
Organoleptic :			
Shape	Homogeneous liquid	Homogeneous Liquid	Fulfill
Smell	Special lemongrass	Typical	Fulfill
Color	Clear yellow	Typical	Fulfill

A low free alkali value indicates a good quality liquid soap because a high free alkali content can cause dry and irritated skin (Hutauruk et al., 2020). The specific gravity value of the test results shows a value of 1.02-1.107g/ml, this is following the quality requirements of SNI 06-4085-1996, namely a maximum of 1.10. Specific gravity is related to the viscosity of the soap. The higher the specific gravity produced in liquid soap, indicates the higher the viscosity and the thicker the texture of the liquid soap (Laksana et al., 2018). Other characteristics that are not a requirement for the quality of liquid soap according to SNI 01-3743-1996 include smell, texture, and color. The test results for these characteristics liquid soap has a distinctive aroma of lemongrass, clear yellow color, and a liquid texture.

CONCLUSION

The results of this study obtained optimum operating conditions at a temperature of 86.8°C, 46.82% KOH concentration, and 56.82 minutes of time with a free alkali response of 0.04158%. These results meet the SNI 06-4085-1996 standard for a maximum free base value of 0.1%. The effect of the independent variables on the response of the free alkali is that the higher the reaction temperature and the longer the stirring time used, the lower the free alkali produced. Meanwhile, the smaller the concentration of KOH used, the lower the free alkali produced. The mathematical polynomial equation obtained is $Y = 0.011910 + 0.023997X_2$.

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