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Quality Analysis of Liquid Soap Formulation Made from Virgin Coconut Oil with Addition of White Tea Extract

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Abstract - *The production of bath soap based on natural ingredients is still rarely found in the market. Many in circulation still use synthetic materials as active ingredients. In this soap-making formulation using natural antioxidants in the form of white tea extract, anti-oxidants from plant extracts are usually added as additives (1-8% of the final soap composition). This research was making natural liquid soap based on virgin coconut oil (VCO), as a purpose to get an output or final product that is suitable for making liquid soap with the addition of natural antioxidants, namely white tea extract and to know the quality of liquid soap produced with quality standards from SNI 06-4085-1996 [3] and SNI 3532-2016 [4]. The research method used is a response surface methodology with a Central Composite Design (CCD) design. The resulting liquid soap product will be analyzed including physicochemical tests and organoleptic tests. Soap with the most appropriate quality according to SNI fell on the 4th variable, with the addition of KOH of 67 grams and a reaction time of 35 minutes. Variable soap 4 has a pH of 10, a free alkali content of 0.0541%, and an unsaponifiable fat content of 0.37%. In the analysis of KOH and time variables on free alkali levels using RSM, it was found that the concentration of KOH affects the free alkali levels. The higher the concentration of KOH in soap making, the more alkaline the soap is.*

Keywords- *liquid soap, VCO, white tea, saponification, RSM*

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1. Introduction

The production of bath soap based on natural ingredients is still rarely found in the market. Most of those in circulation still use synthetic materials as their active ingredients. The use of this synthetic active ingredient has a negative effect on human skin, because it can potentially cause irritation to users (consumers) who have sensitive skin. Examples of synthetic active ingredients that are harmful to human skin and are currently being highlighted are Sodium Lauryl Sulfate (SLS), as well as triclosan on the market [10]. Based on data from the Directorate General of Medical Services at the Indonesian Ministry of Health in 2014, it was found that the number of cases of skin and other subcutaneous tissue diseases was 147,953 cases. And the number of cases of dermatitis was 122,076 cases including 48,576 cases in men and 73,500 cases in women [7].

Along with the development of science and process technology, the development of cosmetics began to shift towards natural products because of the "back to nature" trend [6]. The most common sources of oil used in natural soaps are a mixture of palm, coconut, olive, rice bran, and sunflower seed oil [5]. One of the vegetable oils that is known to have a good saponification effect is virgin coconut oil (VCO). VCO is different from ordinary coconut oil, there is no addition of chemicals and does not use high heat in the manufacturing process. VCO has non-hydrogenated fatty acids like ordinary coconut oil and is easily saponified (saponified) [13].

The use of virgin coconut oil (VCO) as a raw material for soap with a liquid phase has not been developed until now. In fact, liquid soap in its use is more soluble in water. And the addition of active substances from natural ingredients to inhibit the growth of bacteria is still rarely used. The purpose of this research is to know the process of making natural liquid soap based on virgin coconut oil (VCO), to get an output or final product that is suitable for making liquid soap with the addition of natural antioxidants, namely white tea extract and to know the

quality of liquid soap produced with quality standards from SNI 06-4085-1996 [3] and SNI 3532-2016 [4].

2. Methodology

2.1. Materials

In this research, the tools used were analytical balance, beaker glass, stirrer, heater, magnetic stirrer, drop pipette, pH meter, burette, stative, and clamp. The material used were virgin coconut oil (VCO), KOH, propylene glycol, glycerin, aquadest, white tea extract, and coco-DEA.

2.2. Methods

The stages carried out include sample preparation, making liquid soap with a fixed variable of glycerin, virgin coconut oil, propylene glycol, coco-DEA, white tea extract, fragrance, and aquadest. The variables changed in soap making, namely the KOH used were 50 gr, 67 gr, and 84 gr and the stirring time was 35 minutes, 50 minutes, and 65 minutes. Furthermore, the soap physicochemical analysis (free fatty acid, pH, free alkali content, unsaponifiable fat, density, and viscosity), as well as organoleptic analysis was carried out.

3. Results and Discussion

3.1 Results of Soap Physicochemical Analysis

The results of the physiochemical tests for making liquid soap are presented in the table below:

Table 1. Soap Physicochemical Test Results along with SNI Data

Variable	KOH weight (gr)	Time (min.)	pH	Free Fatty Acid (%)	Free Alkali (%)	Unsaponifiable Fat (%)	Soap Density (g/ml)	Soap Viscosity (cp)
1	50	35	7	1,198	0,0125	0,19	0,748	0,275
2	50	50	7	1,233	0,0262	0,26	0,833	0,254
3	50	65	8	1,456	0,0497	0,31	0,953	0,228
4	67	35	10	1,728	0,0542	0,37	1,022	0,198
5	67	50	9	2,013	0,0997	0,45	1,053	0,181
6	67	65	9	2,397	0,1512	0,86	1,089	0,157
7	84	35	10	2,539	0,5793	0,83	1,127	0,138
8	84	50	12	2,721	0,7237	0,92	1,138	0,120
9	84	65	12	2,837	0,8331	1,12	1,154	0,107
	SNI		8-11	Max. 2,5	Max. 0,1	Max. 0,5	1,01 – 1,1	400 - 4000

The pH test is one of the quality requirements of a liquid soap, where liquid soap has direct contact with the skin. Problems can arise when the pH of the soap does not match the pH of the skin. The skin is resilient and can adapt quickly to products having a pH of 8.0 to 11 [12]. From the results of the study, it was seen that the soap variables that matched the skin were on variables 3, 4, 5, 6, and 7. The pH value of soap could be influenced by the soap constituents, namely KOH, where KOH is a strong base group. According to the Indonesian National Standard (SNI 3532-2016) [4], the pH of the available liquid soap ranges from 8 to 11. The high pH in soap is caused by the presence of alkali which does not react with fatty acids in the saponification process.

Testing the free alkali content of soap is included as a preparation for whether the soap meets the quality standard requirements or not. In addition, the free alkali content can also be used as an indicator of the imperfection of the saponification process. According to the Indonesian National Standard [4], the free alkali content in soap should not exceed 0.1% for Na-based soap, and for KOH-based soap it should not exceed 0.14%. As for variables 6, 7, 8, and 9 have high free alkali levels, this is due to the concentration of alkali (KOH base) which is concentrated or excessive in the saponification process [9].

The unsaponifiable fat content test was carried out to determine the fat fraction that could not react with basic compounds, where the fraction was usually in the form of sterols and reactions. the presence of unsaponifiable fat can reduce the foam produced by soap [1]. Based on the soap requirements in SNI 3532-2016 [4], the amount of unsaponifiable fat should not exceed 0.5%. From the results of the study, it can be seen that the soap variables that meet the soap quality requirements are variables 1, 2, 3, 4, and 5. Meanwhile, variables 6, 7, 8, and 9 have unsaponifiable fat content that exceeds the standard, this is due to by imperfect reaction between fatty acids and alkali, so that soap is not formed completely.

It can be seen in the table that the effect of the addition of KOH and the length of the reaction time affect the density of the soap. This can happen because the density of KOH is greater than the density of VCO as soap. The density of KOH is 2.04 g/ml while the density of VCO is 0.92 g/ml [8].

The viscosity of a product on the viscosity of the solvent, the contribution of the solute and the integration of the two [14]. The more balanced the composition between fatty acids and bases, the saponification process runs perfectly, so the thicker the soap product is. In addition, other factors that resulted in a large increase were the driving factor in the liquid bath soap formula and the temperature used in the process of making the liquid bath soap formula [11]. The decrease in viscosity caused by an increase in the water/soap ratio is caused by the water content in the soap.

3.2 Unsaponifiable Fat Content Analysis with Response Surface Methodology (RSM)

Response Surface Methodology is an empirical statistical technique used for multiple regression analysis. In this study, a central composite design was used which gave a response in the form of unsaponifiable fat content contained in soap. Where in this study, the RSM method was used to see the effect of the amount of KOH (gr) and time (minutes) on the levels of free alkali produced. The data that has been obtained is tabulated in the table below:

Table 2. Data Tabulation Free Alkali Content on Liquid Soap Making

KOH Weight (gr)	Time (minute)	Unsaponifiable fat (%)
50	35	0,19
50	50	0,26
50	65	0,31
67	35	0,37
67	50	0,45
67	65	0,86
84	35	0,83
84	50	0,92
84	65	1,12

From the table above, it can be seen that the free alkali content obtained a maximum yield of about 0.92-1.12% with a KOH weight of 84 g and a time of 50-65 minutes, and the minimum yield obtained was around 0.19-0.02% by weight. KOH of 50 g and time 35-50 minutes. The effect of the variable on the response was investigated using the second-order polynomial regression equation as follows:

$z = 0{,}662 - (0{,}0085*x) + (0{,}000155*x2) - (0{,}0322*y) + (0{,}000311*y2) - (0{,}000167*x*y) + 0$

In the above equation, the value of x is the weight of KOH and the value of y is the time. From the regression model, it is shown that the weight of KOH (x) has a positive effect on the level of unsaponifiable fat. Meanwhile, time (y) has a negative effect on the level of unsaponifiable fat. The statement can be seen in the following table:

Table 3. Effect Estimate of Unsaponified Fat Content on Liquid Soap Making

Factors	Effect	Standard error
(1) KOH Weight (L)	0,7033	0,0912
KOH Weight (Q)	0,0900	0,1581
(2) Time (L)	0,3000	0,0912
Time (Q)	0,1400	0,1581
1L by 2L	0,0850	0,1118
Mean	0,5133	0,0833
R^2	0,95991	

The closeness of the model to the experimental data on the observed and predicted values is shown in the table above. The value of R2 provides a benchmark on how much of the variability in the observed response values can be explained by the experimental variables and the interactions between variables. The value of R^2 obtained gives the conclusion that the value estimated by the model is close to the value obtained from the experimental results, where the value of R^2 is always between 0 and 1. If the value of R^2 is getting closer to number 1, then the model shows good results in predicting the response. In the table above, the coefficient of determination (R^2) is 0.95991. That is, the variability in the response can be explained by the model of 95.991%. Furthermore, it will be explained on the Pareto diagram of the standard effect, in the following figure:

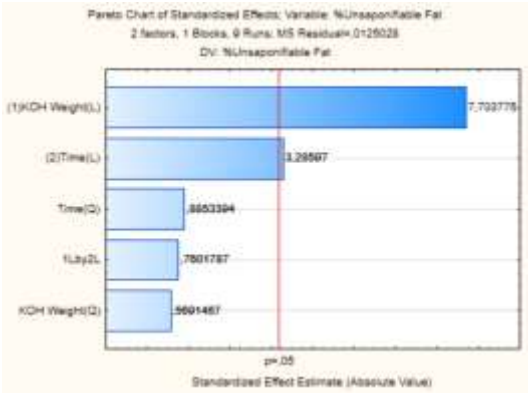
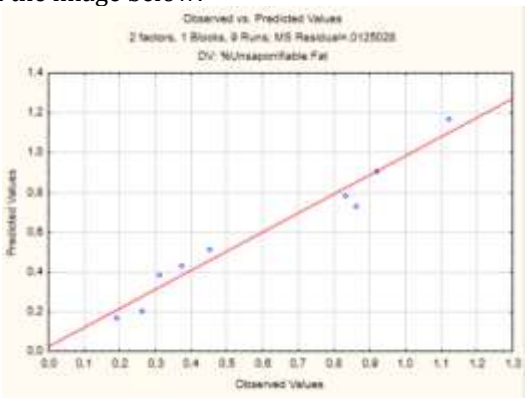


Figure 1. Pareto Chart Standardized Effect

The Pareto chart was created with the aim of helping to identify the significant factors that had an effect on the levels of unsaponifiable fat resulting from the experiment. From the Pareto diagram above, it can be seen that the weight factor of KOH (Q) has the most significant effect on the level of unsaponifiable fat produced. Likewise, the time variable (Q) also has an impact on the level of unsaponifiable fat produced.

After doing a regression analysis on the Response Surface Design, the next step that needs to be seen is the comparison of experimental data with predictive data. Comparison of experimental data to predictive data can be seen in the image below:



Figur 2. Observed vs Predicted Value Diagram

The graph above shows that the observed value data is compared to the predicted value, the observed value data shows a plot of data that is close to the predicted value. The observed value data also follows a straight line from the predicted value. So, it can be concluded that the results

obtained are normally distributed. So, the results obtained are in accordance with the predicted data.

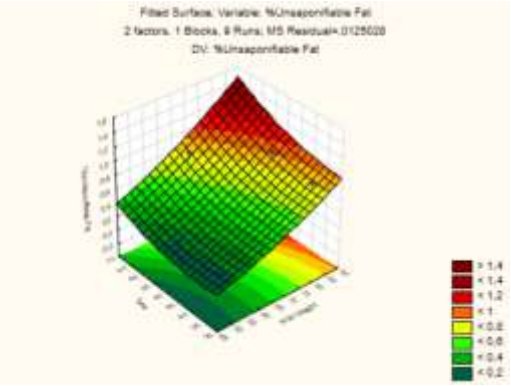


Figure 3. Contour Plot KOH Weight and Reaction time

The fitted surface graph above illustrates the plot of KOH weight parameters and reaction time for making liquid soap. The graph shows a three-dimensional response surface on the dependent variable which is plotted with two independent variables (KOH weight and reaction time) which are processed using Statistica software. It can be observed in the graph that KOH weighing 84 grams with a reaction time of 65 minutes produces the highest unsaponifiable fat content of 1.12%, while KOH weighing 50 grams with a reaction time of 35 minutes produces the lowest unsaponifiable fat content of 0.19%. . There are several things that must be considered in the manufacture of liquid soap, such as the amount of Potassium Hydroxide (KOH) and the length of the saponification reaction. Based on the research that has been done in the manufacture of liquid soap from various concentrations of KOH used, it can be concluded that the concentration of KOH affects the level of unsaponifiable fat. The higher the concentration of KOH in soap making, the greater the level of unsaponifiable fat.

Table 4. Critical Value of Independent Variable Unsaponifiable Fat Content in Soap Making

Factors	Minimum Value	Critical Value	Maximum Value
KOH Weight (gr)	50	65,50125	84
Time (minute)	35	52,00926	65

The table above is the determination of the optimum unsaponifiable fat value with the KOH weight and time variables using the critical value on Statistica software. The critical value for the optimum unsaponifiable fat was obtained when the KOH weight was 65.50125 g with a reaction time of 52 minutes.

3.3 Determination of The Best Quality Liquid Soap

After the pH test, free alkali level test, and unsaponifiable fat content test were carried out, then from the 9 variables, 1 variable with the best quality was taken. The soap variable with the closest quality to SNI 3532-2016 [4] is variable 4, with the addition of KOH of 67 g and the reaction time of 35 minutes.

From the table 1 above, the pH of variable soap 5 is in the range of soap pH set in SNI, namely pH 9, where the pH of soap allowed in SNI is between 8 and 11. For free alkaline soap variable 5 is 0.0997%, where the free alkali content still meets SNI because it does not exceed 0.1%. Meanwhile, the unsaponifiable fat (UF) content in variable 5 soap is 0.45%, where the unsaponifiable fat content still meets SNI because it does not exceed 0.5%. Variable 5 liquid soap that has passed SNI will be added with white tea extract as a basic ingredient that is characteristic of soap and then the free fatty acid content is calculated with a calculation result of 1.728%. The results of these calculations also show that variable 4 soap meets SNI, where the SNI for free fatty acids should not exceed 2.5%.

4. Conclusion

From the experiments that have been carried out, the determination of the soap variable with the most appropriate quality in accordance with SNI falls on the 4th variable, with the addition of KOH of 67 g and a reaction time of 35 minutes. Variable soap 4 has a pH of 10, free alkali content of 0.0541%, unsaponifiable fat content of 0.37%. In the analysis of KOH and time variables on free alkali levels using RSM, it was found that the concentration of KOH affects the free alkali levels. The higher the concentration of KOH in soap making, the more alkaline the soap is.

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