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Temperature Optimization and Addition of Aloe Vera Gel as Antibacteria In Coconut Oil-Based Liquid Soap

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Abstract - *Soap is a substance commonly used for washing and cleaning with water, made from natural oils with strong alkaline compounds such as sodium hydroxide or other strong alkalis and usually added with fragrances and dyes. This study aims to determine the optimization of the ratio of coconut oil to KOH and temperature in the manufacture of liquid soap.This study aims to determine the optimization of the ratio of coconut oil to KOH and temperature in the manufacture of liquid soap. In this study, the saponification reaction was used by reacting coconut oil with a strong base of KOH. The mole ratio between coconut oil and KOH used is 1:3, 1:4 and 1:5. The temperature used 3 points, there are 30^oC, 40^oC, and 50^oC. Parameters observed were free fatty acid, pH, free alkali content, unsaponication fat content, density, and viscosity. The best results in the physiochemical test were running 4 (KOH ratio 5 and temperature 50^oC) with pH 8, free alkali content of 0%, unsaponication fat content of 0.00175%, density 1.02 g/ml, viscosity 715.87 cP, and 1% free fatty acids. The critical value for the optimization of the liquid bath soap formula on the level of unsaponification fat based on RSM data is achieved at a KOH ratio of 4.28758 with a temperature of 62.41^oC.*

Keywords- *soap, saponification, temperature, KOH, virgin coconut oil*

1. Introduction

Soap is a substance commonly used for washing and cleaning with water, made from natural oils with strong alkaline compounds such as sodium hydroxide or other strong alkalis and usually added with fragrances and dyes. Soap can be made from oil (triglycerides), free fatty acids (FFA) and fatty acid methyl esters by reacting an alkaline with each substance, which is known as a saponification process [1]. Saponification is the hydrolysis reaction of triglycerides by a strong alkaline such as NaOH or KOH in the formation of soap. The principle is hydrolysis of fats or oils in the form of triglycerides by a strong alkaline which will produce soap and glycerol [2]. To produce hard soap, the alkali used is NaOH, while in soft soap the base used is KOH. The difference between hard (solid) and soft (liquid) soap can be seen from its solubility in water. Hard soap is less soluble in water, while soft soap is easily soluble in water [3].

Pure coconut oil or VCO is a natural oil obtained from fresh and ripe coconut kernels without any heating process and the addition of chemicals. Coconut oil has a high content of saturated fatty acids. In the industrial world, coconut oil can be used as a raw material for making soap because it contains antibacterial properties. VCO has medium chain fatty acids and is rich in saturated fatty acids, such as lauric acid with antibacterial activity. Another study reported that VCO inhibited the growth of Staphylococcus aureus through the mechanism of destroying bacterial cell walls and increasing the ability of the cell's immune system. Lauric acid (C12) is a saturated fatty acid that most inhibits the growth of Gram-positive organisms [4].

2. Methodology

The main ingredients used in this study were KOH and virgin coconut oil. While the auxiliary ingredients are distilled water, glycerin, propylene glycol, coco-DEA, and rose fragrance, 96% neutral ethanol, PP indicator, and HCl. The tools used are beaker glass, magnetic stirrer, hotplate stirrer, clamps and statives, thermometer, electric stove, pH

paper, burette, Erlenmeyer, upright cooler, three neck flask, pycnometer, and Ostwold viscosimeter.

This study uses a saponification process, by reacting KOH and triglycerides in the form of virgin coconut oil. The independent variables used were the ratio of KOH: coconut oil (1:3, 1:4, 1:5) and temperature (30ºC, 40ºC, 50ºC). While the fixed variables used were glycerin 10.25 g, propylene glycol 22.5 g, distilled water 300 g, coco-DEA 5.46 g, and rose fragrance 1 g.

In this study, the Response Surface Methodology (RSM) method is a design and model that works with various treatments continuously when finding the optimum value or describing the response according to the goal. The main goal of RSM is to find the optimal response.

Table 1. Respon Surface Methodology (RSM)

Standard Run	KOH ratio	Temperature (ºC)
1	3,000000	30,00000
2	3,000000	50,00000
3	5,000000	30,00000
4	5,000000	50,00000
5	2,921910	40,00000
6	5,078090	40,00000
7	4,000000	29,21910
8	4,000000	50,78090
9 (C)	4,000000	40,00000
10 (C)	4,000000	40,00000

3. Results and Discussion
3.1 Liquid Soap Physicochemical Test Results

The results of the density test show that the more KOH ratio and the higher the temperature used, density will increase. According to SNI 06-3532-1994 [5], the density requirement for liquid bath soap is 1.01-1.1 gr/ml. If seen from the table above, those that meet the requirements are run 3, run 4, run 6, run 8, and run 9.

Table 2. Liquid Bath Soap Physiochemical Test Results

Run	KOH ratio	Temperature (ºC)	Density (gr/ml)	Viscosity (cP)	% free alkali		pH		% free fatty acid
					before	after	before	after	
1	3,00	30,00	0,98	118,31	2,13	0	14	8	0,4
2	3,00	50,00	0,99	166,24	2,13	0	14	8	0,6
3	5,00	30,00	1,02	834,02	3,92	0	14	8	0,8
4	5,00	50,00	1,02	715,87	3,36	0	14	8	1,1
5	2,92	40,00	0,98	108,10	2,24	0	14	8	0,5
6	5,08	40,00	1,01	807,68	3,58	0	14	8	0,9
7	4,00	29,22	0,99	248,99	3,02	0	14	8	0,6
8	4,00	50,78	1,01	576,40	2,35	0	14	8	0,8
9	4,00	40,00	1,01	535,95	2,58	0	14	8	0,6

The results of the viscosity test showed that the more KOH ratio and the higher the temperature used, viscosity will increase. The viscosity of a product depends on the viscosity of the solvent, the contribution of the solute and the integrity of both [6]. The more balanced the composition between fatty acids and bases, the saponification process runs perfectly, so the thicker the resulting soap product. The decrease in viscosity is due to an increase in the water/soap ratio because the viscosity is affected by the water content in the soap. According to SNI 06-3532-1994 [5], the viscosity requirement of liquid bath soap is 400-4000 cP. If seen from the table above, those that meet the requirements are run 3, run 4, run 6, run 8, and run 9.

In the results of the free alkali levels before the addition of citric acid on all variables did not meet the standards of SNI 06-4085-1996 [7], namely because the free alkali levels produced were more than 0.1% [8]. This is because the amount of alkali used is excessive, so that a lot of alkali does not react with fatty acids in the saponification reaction. Large alkaline levels in soap usually fall into the laundry soap class. Excess alkali can also cause skin irritation [9]. At the free alkaline level after the addition of citric acid, all variables produced are 0%. This is because when the PP indicator was added to the test, the sample did not change color to purple ping. This final product is in accordance with SNI 06-4085-1996 [7], because the free alkali content is less than 0.1%.

In the pH test before the addition of citric acid, the resulting pH was 14. At this pH, liquid soap has not yet

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entered the SNI, where the SNI pH for liquid bath soap ranges from 8-11 (SNI 3532-2016) [9]. To lower the pH of the liquid soap produced, citric acid is added. After adding citric acid, the pH contained in the liquid soap dropped to 8 and has entered the SNI.

The table shows that all the variables in the liquid bath soap produced before adding citric acid did not contain free fatty acids, but contained free alkali. Free fatty acids are acids that are not bound as compounds with sodium or triglycerides (neutral fats) [8]. After being added with citric acid, the free alkali contained was 0 and the free fatty acids contained increased. This is because citric acid or weak fatty acids are not bound to potassium compounds or triglycerides, causing the presence of free fatty acids. Based on SNI 3532-2016 [9] free fatty acids contained in liquid soap should not be more than 2.5%. High levels of fatty acids will cause a lack of soap binding capacity to dirt, trigger rancidity, and reduce the shelf life of soap [8].

3.2 Optimization of Unsaponification Fat Using Response Surface Methodology (RSM)

The data above shows that the more KOH used and the higher the temperature, then the level of unsaponification fat will decrease. According to SNI 3532-2016 [9], in liquid bath soap the unsaponification fat content is a maximum of 2.5%. All tested products have met SNI because the value of unsaponification fat content is below 2.5%.

Table 3. Unsaponification Fat Content Test Results

Run	KOH ratio	Temperature (^{o}C)	Unsaponification Fat (%)
1	3,00	30,00	0,0056
2	3,00	50,00	0,0039
3	5,00	30,00	0,0030
4	5,00	50,00	0,0017
5	2,92	40,00	0,0048
6	5,07	40,00	0,0035
7	4,00	29,21	0,0043
8	4,00	50,78	0,0021
9	4,00	40,00	0,0043

The effect of the variable on the response can be determined by a first-order polynomial regression equation. Based on Response Surface Methodology (RSM) data processing, the first-order equation provides data on the effect of the ratio of KOH (x) and temperature (y) so that the equation becomes Z(x,y) as follows:

$$Z = 5036,95+72,89*x+0,37*x^2+25,36*y+0,60*y^2-1,43*x*y+0,$$

Table 4. Estimating Effect of Making Liquid Bath Soap

Factor	Effect	Standar error
Mean/Interc.	105,4598	0,430602
(1) KOH Ratio (L)	1,2197	1,538768
KOH ratio (Q)	1,6471	2,027501
(2) Temperature (L)	3,0903	0,988129
Temperature (Q)	1,1940	0,872349
1L by 2L	-4,2967	2,511971

The table above explains that there is a closeness between the model and experimental data on the observed and predicted values. The data is declared to be getting better if the R-square is close to 0.98 or 1. The resulting (R)-Square data is worth 96884 then the equation that has been obtained from the initial equation z (x,y) can represent 0.96884 or equivalent to 96.88% soap.
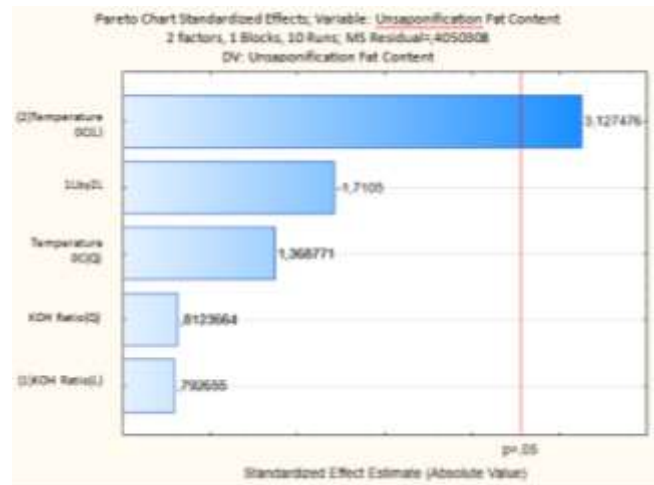

Figure 1. Pareto Chart Standarized Effect

From the Pareto chart in Figure 1, the presentation of the Pareto Chart data aims to see the most influential variables. Seen on the graph the most influential is the temperature variable, which is 3.127476 where the bar chart crosses the line limit (p = 0.05). The temperature factor has the most significant effect on the level of unsaponification fat produced.
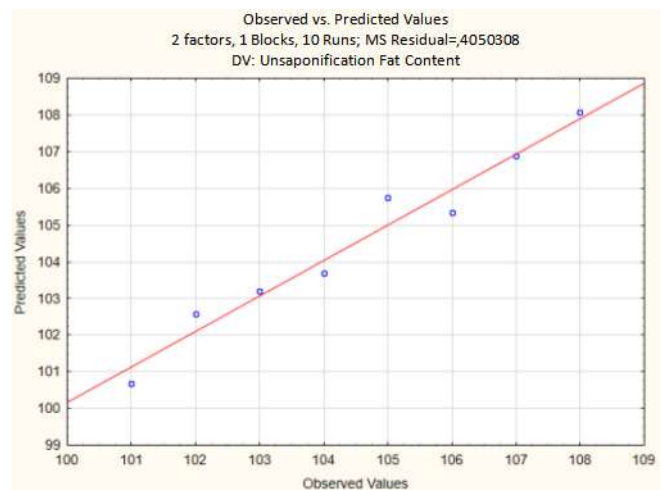

Figure 2. Observed Diagram vs Predicted Values

The graph above states the comparison between experimental data and predictive data, it can be seen that the plot is spread out following a straight line. From this it can be concluded that the results obtained are normally distributed and in accordance with the predicted data.
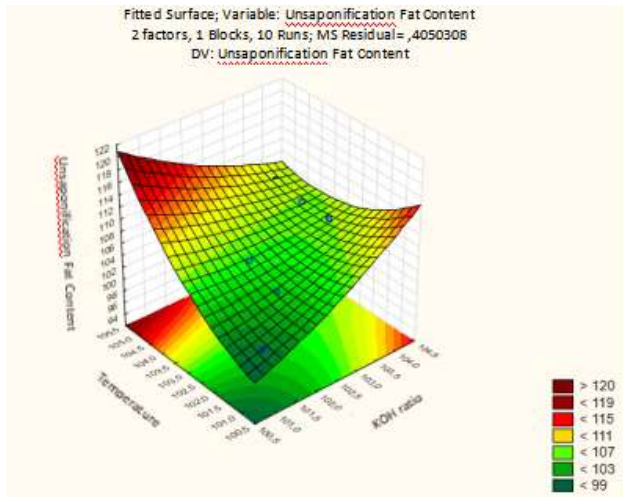
Figure 3. Contour Plot of KOH Ratio and Temperature

The picture above explains the existence of a three-dimensional response surface on a fixed variable which is plotted with two independent variables (KOH ratio and temperature) with data processed using statistical software central composite design. The figure above illustrates the contour plot of the variable KOH ratio and temperature in the manufacture of liquid soap.

Table 5. Analysis of Variance (ANOVA)

Factor	SS	df	MS	F	p
(1) KOH Ratio (L)	0,254	1	0,254	0,628	0,472
KOH Ratio (Q)	0,267	1	0,267	0,659	0,462
(2) Temperature °C (L)	3,962	1	3,962	9,781	0,035
Temperature °C (Q)	0,759	1	0,758	1,873	0,242
1L by 2L	1,185	1	1,185	2,925	0,162
Error	1,620	4	0,405		
Total SS	52,000	9			

The results of the second-level response surface model are in the form of Analysis of Variance (ANOVA) given in Table 4.6. It is necessary to test the significance and adequacy of the model. Fisher's ratio of variance, F value (= S2r/S2e), is a valid statistical measure of how well a factor explains variation in the data about the mean. The total F obtained is 15.86867. The greater the value of F, the greater the uniformity.

Table 6. Predicted Value of Unsaponification Fat Content with Independent Variables

Factor	Observed Minimum	Critical Values	Observed Maximum
KOH Ratio	2,92191	4,28758	5,07809
Temperature	29,21910	62,40793	50,78090

The optimization parameter of the liquid bath soap formula with the variable ratio of KOH and temperature is determined by the critical value (critical value). Thus, the critical value for the optimization of the liquid bath soap formula is achieved at the KOH ratio of 4.28758 with a temperature of 62.41 °C.

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