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The production of paper soaps from coconut oil and Virgin Coconut Oil (VCO) with the addition of glycerine as plasticizer

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Abstract. Hand washing with soap is important because it is proven to clean hands from germs and bacteria. The paper soaps were made from coconut oil and virgin coconut oil (VCO) with the addition of glycerin as a plasticizer. The aims of this research were to determine both formulation of paper soap using coconut oil and VCO based with addition of glycerin, and to determine the quality of the paper soap which is a disposable hand soap. This research used laboratory experimental method using descriptive analysis. The treatments of this research were treatment A (paper soap without the addition of glycerin), treatment B (paper soap with the addition of glycerin 10% (w/w)), treatment C (paper soap with the addition of glycerin 15% (w/w)), treatment D (paper soap with the addition of glycerin of 20% (w/w)). Parameters tested were moisture content, stability of foam, pH value, insoluble material in ethanol, free alkali content, unsaponified fat, antibacterial activity test, and organoleptic test. The result of physicochemical characteristics for both coconut oil-paper soap and VCO-paper soap revealed that treatment C (the addition of glycerin 15% (w/w)) was the best soap formulation. Coconut Oil paper soap 15% w/w glycerin had water content 13.72%, the content of insoluble material in ethanol 3.93%, the content of free alkali 0.21%, and the content of unsaponified fat 4.06%, pH value 10.78, stability of foam 97.77%, and antibacterial activity against *S. aureus* 11.66 mm. Meanwhile, VCO paper soap 15% w/w glycerin had the value of water content of 18.47%, the value stability of foam of 96.7%, the pH value of 10.03, the value of insoluble material in ethanol of 3.49%, the value of free alkali content 0.17%, the value of unsaponified fat 4.91%, and the value of inhibition diameter on the antibacterial activity test 15.28 mm. Based on Mandatory Indonesian National Standard of solid soap SNI 3532:2016 showed that both of paper soap had not been accorded with SNI 3532:2016, unless the value of the insoluble material in ethanol. Moreover, organoleptic tests performed that both paper soap treatment D (20% w/w glycerine) were preferred by the most panelists.

Keywords: Paper Soap, Coconut Oil, Virgin Coconut Oil, Glycerin, Plasticizer



1. Introduction

Cleanliness is a very important thing due to the increasing number of diseases caused by bacteria and germs[1]. Soap is a substance used with water for washing and cleaning, made of a compound of natural oils or fats with sodium hydroxide or another strong alkali, and typically having perfume and coloring added. Even today, soap is not just used for cleaning to maintain the health of the skins, there are also some soap that also serves as softening soap and whitening soap. In making soap often used various kinds of fats or oils as raw material. For being used in soap manufacture the type of oil needs to be selected in accordance with the use of soap itself.

Based on the making process, soap is made in two ways, namely saponification process and oil neutralization process. Saponification process occurs because the reaction between triglycerides with alkali, while the neutralization process occurs due to free fatty acid reaction with alkali[2]. Oil saponification process will obtained by-product namely glycerol, while neutralization process could not get any glycerol.

Along with the advancement of the era, then also developed a kind of soap that circulates in the market. Bath soap that circulates in the market based on its shape is divided into two forms, namely solid soap and liquid soap[3]. Solid soap itself is divided into several types based on their appearance, ie, opaque soap (non-transparent solid soap), and translucent soap (solid soap with almost transparent color), and transparent soap (solid soap with transparent color).

The two main components of soap constituents are fatty acids and alkali. Selection of the type of fatty acids determines the characteristics of the resulting soap, because each type of fatty acids will give different properties to the soap [4]. Fatty acids are the main components of fat and oil composites, so the selection of the type of oil to be used as a raw material for making soap is very important. To produce soap with good quality, it must use raw materials with good quality too.

Today, people want hand-washing soap that is practical to carry anywhere. Paper soap itself is one solid soap product innovation that printed or molded as thin as paper, paper soap will dissolve when exposed to water, it turns out to be foam. Soap paper is generally used as a disposable hand-washing soap because of its small size and thin so it easy to carry anywhere and suitable for use during at any outdoor activities. In Indonesia there is still difficulty to find a factory that produces paper soap. Paper soap majority is produced in China and the paper soap being produced in a big industrial scale. The paper soap made from materials, such as *Axyl Sodium Sulfate*, *Cocoamido Propyl Betaine*, *Cocoamide DEA*, *Paraffinum Liquidum*, *Perfume*, *Glycerin*, *Metyl Paraben*, *Propyl Paraben*, *Cabomer*, *Dimethicone*, *Sodium Starch Polucrylate*, *Aqua*, as well as *Alternatifolia Melaleuca Oil*. Until now there is no paper soap that made from natural ingredients. Therefore, in this research try to develop paper soap that made from natural ingredients such as coconut oil and chemical which are safe and healthy for the skin.

Coconut oil rather than used for cooking, it also can be used for raw materials in cosmetics and some health products such as soaps, ointments, and others. Based on research was done by [5], states that the excess of soap made from coconut oil compared to other oils is that coconut oil soap has a good cleaning power due to the presence of lauric acid as the dominant fatty acids in coconut oil. The addition of other ingredients as a mixture in paper soap making can also maximize the benefits of the paper soap used. One of the ingredients of the mixture used as a reference treatment in the process of making paper soap is glycerine. Based on experiments that have been done glycerin as a plasticizer can make soap become elastic in texture such as industrial paper soap product.

To know the best glycerine concentration, proper formulation, and quality of resulting from paper soap, it is necessary to do this research. Until now there has been no research on making coconut oil-based paper soap and using the concentration of glycerine as a treatment. So there is no provision regarding soap formulation of palm oil based paper and how the printing soap process. The purpose of making paper soap is expected to produce soap paper that can be used everyday whenever and wherever, and able to cause a sense of comfort on the skin and able to prevent the skin from infections caused by bacteria.

2. Materials and Methods

The materials used were coconut oil and virgin coconut oil (VCO) obtained from Balai Besar Industri Agro, Bogor. The chemical materials equipped were 30% NaOH solutions, stearic acid, alcohol, perfume

soap (fragrance oil) citrus scented, glycerine and aquadest. While the supporting ingredient for the quality test were 96% neutral ethanol, 0.9 N and 2 N, 0.1 N, 0.1 N hydrochloric HCl, 1% phenolphthalein indicator, sodium hydrogen carbonate, and n-hexane (technical grade). The test bacteria used in soap bacterial activity test was *Staphylococcus aureus* bacteria. Bacteria media used was Nutrient Agar (NA). The supporting materials consist of pH indicator paper, tissue, plastic wrap, aluminum foil, bread paper, disc paper, and transparent plastic.

The equipment used were beaker glass, hot plate stirrer, stirrer rod, thermometer, digital scales, dropper drops, spiral cake paste, spatula, petri dish, desiccator, oven, analytical scales, threaded tube, vortex, Erlenmeyer flask, acid chamber, separator funnel, microburet, upright cooler, tweezers, micro pipettes, test tubes, ose needles, bunsen burners, autoclaves, incubators, and slicers.

This research used laboratory experimental method using descriptive analysis. The treatments of this research were treatment A (paper soap without the addition of glycerin), treatment B (paper soap with the addition of glycerin 10% (w/w)), treatment C (paper soap with the addition of glycerin 15% (w/w)), treatment D (paper soap with the addition of glycerin of 20% (w/w)). This research consists of several stages, the first stage was the preparation of the raw materials for making transparent soap, the second stage was the making of paper soap, the third stage was the quality analysis of the coconut oil and VCO paper soap.

2.1 Raw Materials Preparation

The first step in this research was create 30% NaOH solutions, then prepared the raw material needed for making paper soap which includes pure coconut oil (or Virgin Coconut Oil) as fatty acids for about 23 gram, 30% NaOH as alkali 25 gram, stearic acid as additional material 11 grams, aquades as a solvent 22.5 grams; 12.5 grams; 7.5 grams; and 2.5 grams depends on the soap treatments, fragrance oil as a perfume of 0.5 grams, 96% ethanol as a solvent 18 grams, and glycerin as plasticizer ingredient of 0 grams, 10 grams; 15 grams; 20 grams along with the variation in glycerine concentration used 0% as control, 10%, 15%, and 20%.

2.2 Paper Soap Making

In the process of making paper soap there were several stages, first heating pure coconut oil or VCO upto 60°C, then mixed with 30% NaOH solution. Then it is stirred until the solution become more thicken to produce the mixture solution between the oil and 30% NaOH solution. The next step was heating and melting the stearic acid, after that glycerine was added, followed by 96% ethanol and aquades, then stirred until homogeneously. The mixed solution in the previous step were combine with the stearic acid solution which is then heated for approximately one hour. The soap solution can be poured into the silicone mold, wait for approximately 24 hours for the soap to be removed from the mold. After the soap hardens, the soap is cut using a wooden slicer. The size of paper soap produced is 2x2 cm with a thickness of 1 mm. Next curing process is done to reduce the pH value, this process is done by storing paper soap at room temperature for about two weeks. Flowchart of process of making pure coconut oil or VCO based paper soap can be seen in Figure 1.

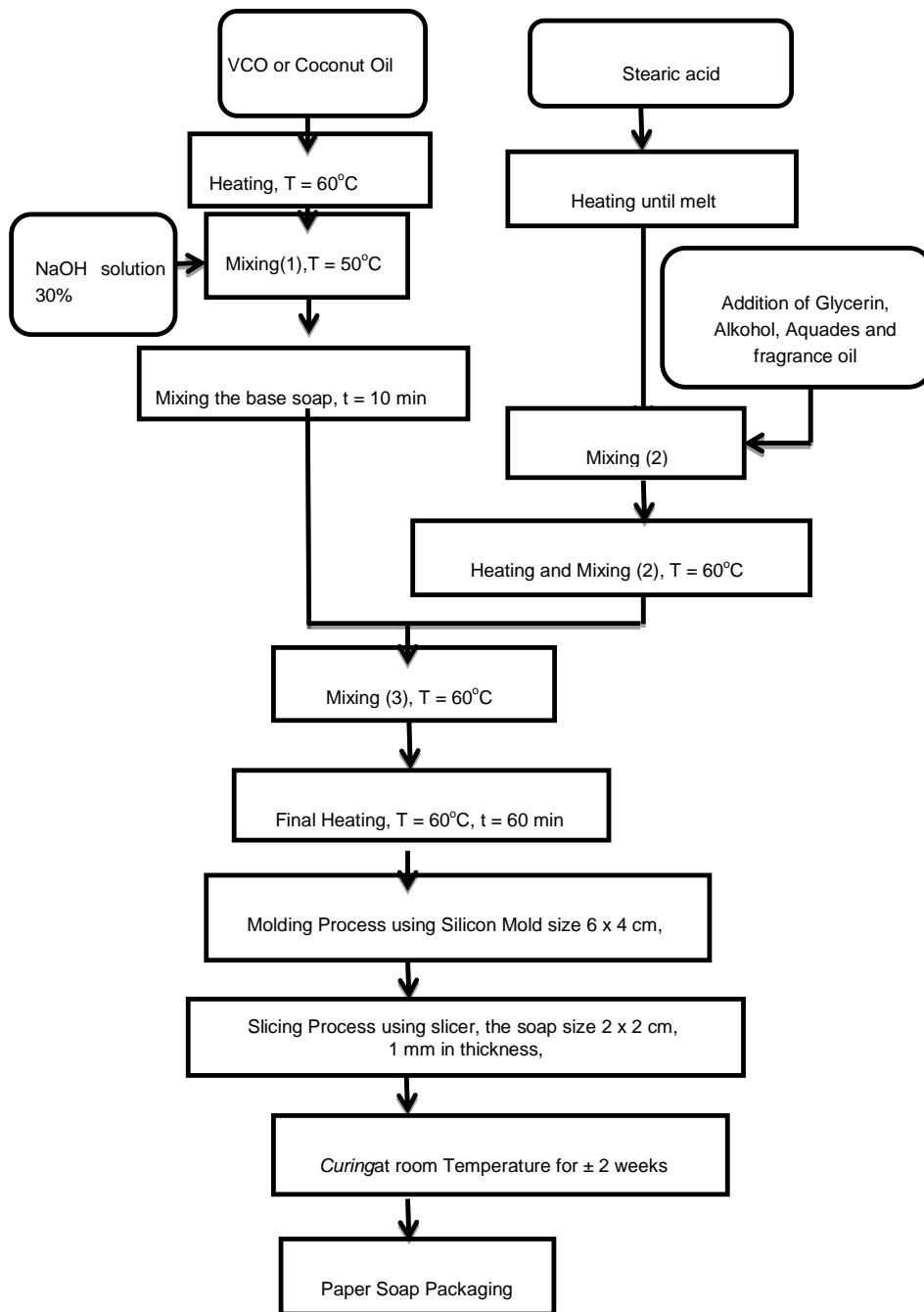


Figure 1. Flow diagram of the making paper soap process.

2.3 Quality Test

Observations to be made on paper soap produced were organoleptic test, physicochemical properties of soap, and antibacterial test. For organoleptic test conducted that is covering color, aroma, shape, texture, and

many foam. For the physical properties of the soap, the foam stability test was performed, while for soap chemical properties, the water content, the insoluble material in ethanol, the free alkali, the unsaponified fat, and the pH contained in the resulting soap. For test of chemical properties based on SNI 3532: 2016 regarding the standard of solid soap bath quality. While organoleptic testing determined by the subjective value from 1 to 4 based on an assessment of the importance of texture, aroma, amount of foam, power of detergency, and after usage impression.

3. Results and Discussions

3.1 The Characteristic of Coconut Oil based Paper Soap and VCO based Paper Soap

The resulting paper soap product was the result of a transparent soap formulation with a combination of variation in glycerine concentration used 0% as control, 10%, 15%, and 20%. The transparent soap produced after cutting into paper soap can be seen in Figure 2.

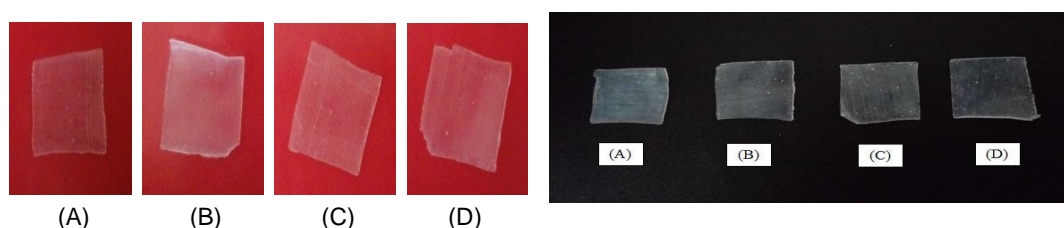


Figure 2. Coconut Oil Paper Soap (right) and Virgin Coconut Oil Paper Soap (left)

3.2 The Quality of Paper Soaps

3.2.1 Water Content

Moisture content of solid soap according to SNI 3532: 2016 is 15% (wb) maximum. From the results of the measurement of water content on the paper soap, soap with no glycerine (treatment A) revealed the highest water content. From Figure 3 it can be seen that the more concentration of glycerin added to paper soap making, the moisture content contained in the soap is lower. Glycerine has the ability to bind water. From these statements it is suspected that the more glycerin added to the paper soap formulation the more water the glycerin bound together to cause the water content to decrease[6].

The water content of VCO paper soap (17.19-23.13%) were slightly higher than the water content of coconut oil paper soap (13.38-22.01%). Only treatment C dan D of coconut oil paper soaps were met the requirement of SNI. In paper soap making, several things are thought to affect the water content of paper soap, one of which is some type of fatty acid contained in coconut oil and volatile substances, such as alcohol. Several types of fatty acids, for example lauric acid, are water soluble and volatile when using water or steam[7].

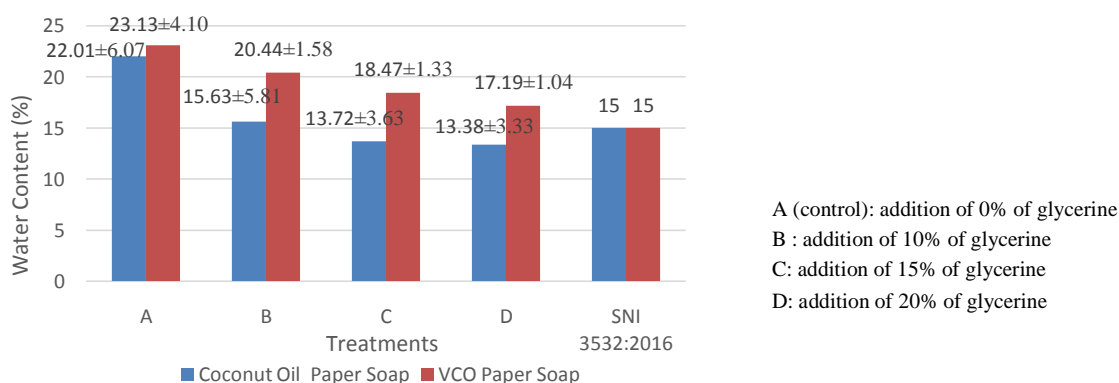


Figure 3. The relations between glycerine addition to the water content of paper soaps.

3.2.2 Insoluble Materials in Ethanol

Based on SNI 3532: 2016 on the standard of solid soap baths states that the value of insoluble material in ethanol is a maximum of 5.0%. The results of insoluble material testing in ethanol on paper soap can be seen in Figure 4. The range value of insoluble materials in ethanol presence in the coconut oil paper soap were from 2.57-3.93%, where as in the VCO paper soap were around 1.26-3.49%.

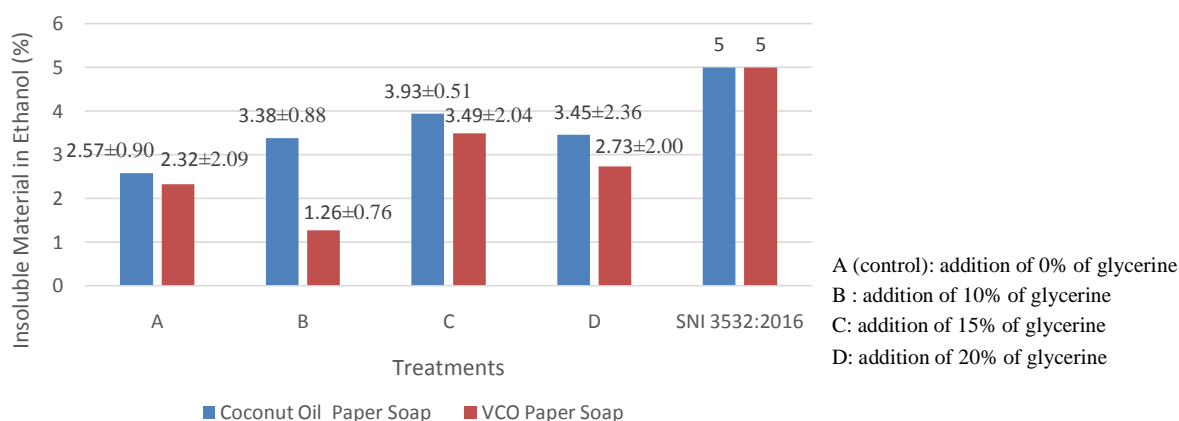


Figure 4. The relation between the glycerine addition to the insoluble materials in ethanol.

The amount of insoluble material in ethanol contained in the resulting of both paper soap was in accordance with SNI 3532: 2016 which is a maximum of 5.0%. From the results of the measurements showed that the addition of glycerin to paper soap formulations was not expected to affect the value of insoluble content in ethanol, glycerin is a soluble ingredient in alcohol[8]. The presence of an insoluble material in ethanol is suspected by the use of ingredients in the soap-making process. These substances are thought to contain insoluble molecules in ethanol. The value of insoluble material in ethanol in this paper soap is thought to be influenced by the presence of oil which is not well saponified and the oil is not completely soluble in water and ethanol.

3.2.3 Free Alkali Content

Free alkali is an unattached alkaline compound as a compound at the time of soap making, it is due to the excessive addition of alkali during the saponification process [5]. The percentage of free alkali content (calculated as NaOH) on paper soaps can be seen in Figure 5.

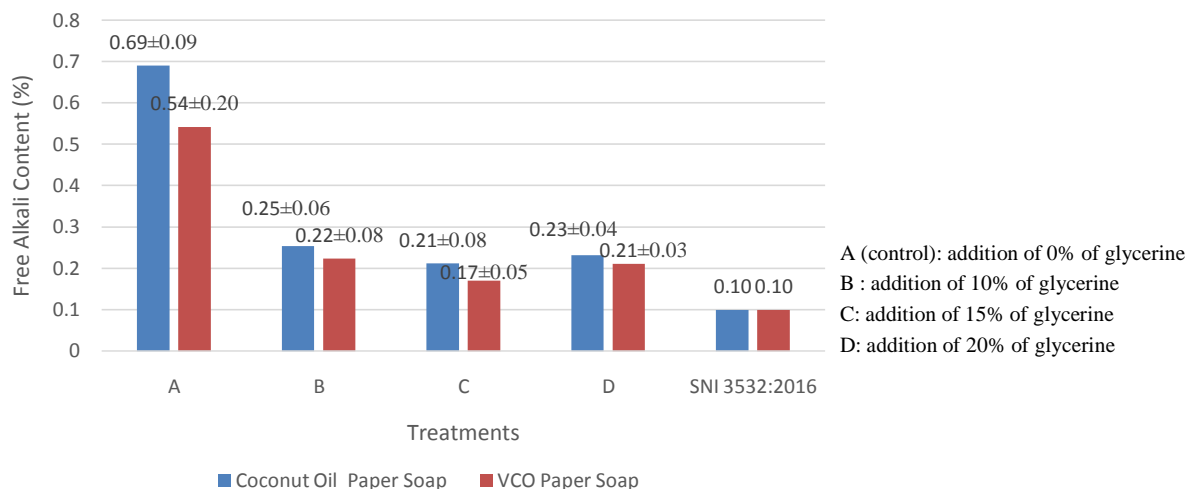


Figure 5. The relation between the glycerine addition to the free alkali content.

In Figure 5 it can be seen that treatment A (soap without addition of glycerin) has the highest free alkali content, that are equal to 0.69% (coconut oil papersoap) and 0.54% (VCO paper soap). The free alkali content contained in the resulting paper soap is quite high, presumably because the added concentration of the alkali is too thick or due to the excessive addition of alkali. In addition, the difference in the amount of NaOH usage in paper soap formulations with solid soap bath formulations generally makes the alkali content of paper soap has a higher mean value compared to the standard of SNI 3532: 2016.

Free alkali test on the paper soap it showed the effect of adding glycerin to paper soap formulations to free alkali values. The addition of glycerin to a paper soap formulation is thought to bind OH ions so as to decrease the free alkali value of the paper soap. Treatment C paper soap with 15% (w / w) addition of glycerin has a lower free alkali value compared with soaps of B and D treatments with 10% (w / w) and 20% (w / w) glycerine additions. This shows that paper soap with the addition of 15% (w/w) of glycerin addition is the optimum treatment to obtain free alkali values closed to SNI.

3.2.4 Unsaponified Fat Content

The presence of unabsorbed fraction or fat can decrease the ability to clean (detergency) on soap (Fachmi, 2008). The percentage of unabsorbed fat content in the resulting paper soap can be seen in Figure 6. From Figure 6, it can be seen that treatment A (coconut paper soap) has the lowest unsaponified fat content of 1.15%. The amount of fat not covered with soap paper has not fulfilled the quality criteria of unsaponified fat content according to SNI 3532: 2016, which is maximal 0.1%. Based on these observations, an increase in the added concentration of glycerin used may increase the unsaturated fat content in the resulting paper soap. The higher the addition of glycerin concentration, the higher the unabsorbed fat content contained in the paper soap. This is presumably because glycerine is a by-product of breaking oils or fats to produce fatty acids as well. The more glycerin means fatty acids that react with coconut oil are also more and more so that the amount of unsaponified fat contained in paper soap is higher. The amount of OH (*hydroxyl compound*) contained in glycerin produce a lot of unsaponified fat in the saponification process.

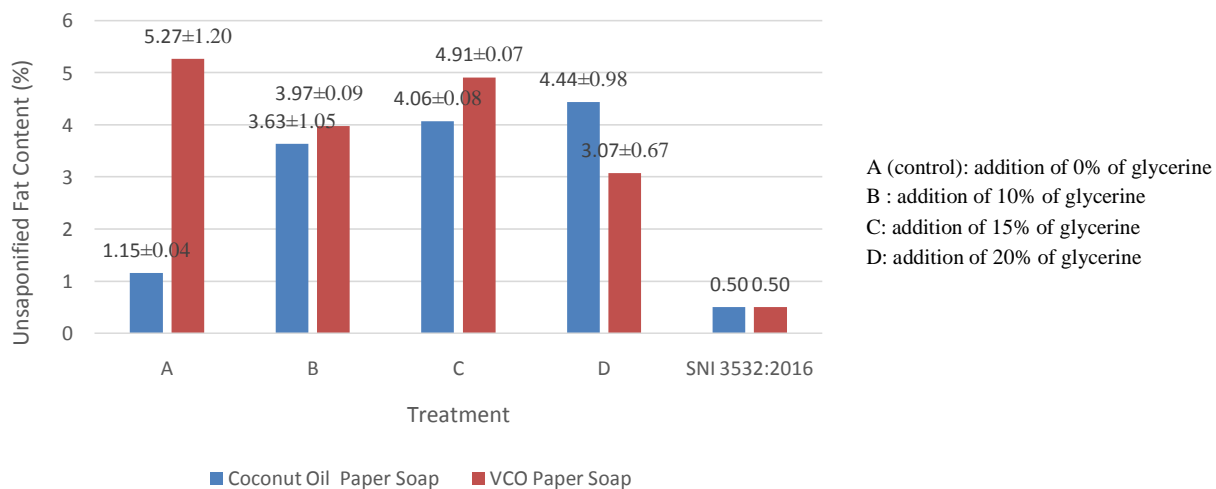


Figure 6. The relation between the glycerine addition to the unsaponified fat content.

Meanwhile, VCO paper soap treatment A (0% glycerine) had the highest unsaponified fat content 5.27%. The more glycerine added the VCO paper soap tend to decrease the unsaponified fat content value. The addition of glycerine to paper soap formulations has an effect on the value of unabsorbed fat. The addition of glycerine to paper soap formulations is thought to assist the saponification process so as to decrease the value of unabsorbed fat on paper soap. This may be due to an inadequate saponification process between oil and NaOH, so that there is an oil that is not well-stuffed / unsaponified. The stirring process between oil and NaOH was carried out for about 10 minutes, presumably the stirring time was shortened so that it still left unreacted fat forming soap with alkali.

3.2.5 pH

The degree of acidity or pH is a chemical parameter to determine whether the resulting paper soap is acidic or alkaline. The soap that is too alkaline can cause skin irritation, so based on ASTM D 1172-95 the standard pH value for bath soap is 9 – 11. It shows that paper soap in this study already meet the pH standard of bath soap [9]. The pH value measurements of paper soap are done after the paper soap has been cured for about two weeks. From the results of pH measurements that have been done show that the addition of glycerin in paper soap formulations does not affect the pH value, glycerine has a neutral pH value [8]. The pH value of paper soap is influenced by the curing process, the longer the curing process will decrease the pH value of the paper soap. In addition, the pH value of the paper soap is also influenced by the value of free alkali, the higher the free alkali value the higher the pH value and the lower the free alkali value the lower the pH value of paper soap. The pH value contained in the resulting paper soap can be seen in Figure 7.

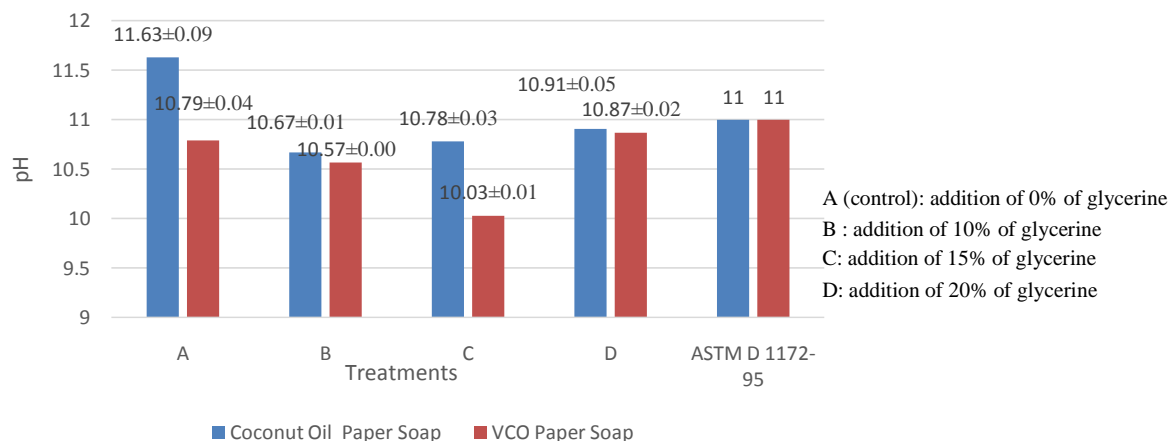


Figure 7. The relation between the glycerine addition to the pH value.

From Figure 7 it can be seen that the treatment A (coconut paper soap) has the degree of acidity or the highest pH value of 11.63. Overall the degree of acidity or pH value resulting from this paper soap is in accordance with the criteria of soap bathing quality, which is 9 - 11 based on ASTM D 1172-95. Results of pH measurements that have been done show that the addition of glycerin in paper soap formulations does not affect the pH value, because according to experiments that have been done using pH indicator states that glycerine has a neutral pH value.

The pH value measurements of paper soap are done after the paper soap has been cured for about two weeks. The amount of alkali present in the paper soap may affect the pH value. This paper soap manufacture involves the use of large amounts of NaOH, which accounts for 25% of all paper soap making materials. The pH value of the paper soap is affected by the curing process, the longer the curing process, the more pH value will decrease on the paper soap. In addition, the pH value of paper soap is also influenced by the value of free alkali, the higher the free alkali value the higher the pH value because alkali is a strong base.

3.2.6 Antibacterial Activity against *Staphylococcus aureus*

The bacteria used for this paper soap test is the gram-positive bacteria *Staphylococcus aureus* that can attack the skin. Antibacterial activity test was done by paper disc method, while the used disc paper had a diameter of 6 mm. The results of the analysis of paper soap clearance testing against *Staphylococcus aureus* bacteria can be seen in Figure 8.

In this antibacterial activity test used control with the test sample using aquadest. From Figure 8 it can be seen that the control treatment has the clearest zone or the lowest inhibitory diameter (DDH) against the *Staphylococcus aureus* bacteria of 0.32 mm. The control treatment was carried out to find out the comparison when washing hands using water alone using paper soap. Clear zones or inhibitory forces against the growth of *Staphylococcus aureus* bacteria on paper soaps may be derived from antiseptic and antimicrobial ingredients such as alcohol, glycerine, and coconut oil used in such paper soap formulations. The largest clear zone is owned by the soap of the third treatment paper (paper soap with 15% (w / w) glycerine addition), which is 11.66 mm. This proved that the ability of soap treatment C to inhibit *Staphylococcus aureus* bacteria is greater than other paper soap.

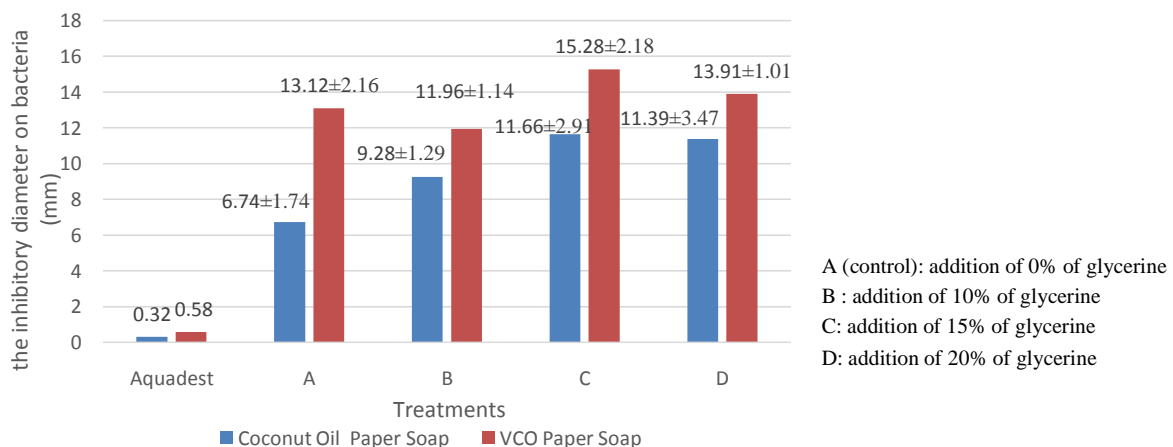


Figure 8. The relation between the glycerine addition to the inhibitory diameter on bacteria

Based on Fig. 8 also shows that the higher concentration of glycerin added to paper soap, clear zone or bacterial inhibition zone is also greater. Glycerine can be used as a solvent, sweetener, lubricant, plasticizer, and as an antimicrobial agent as well[10]. In this study, in addition to soap treatment C (15.28 mm) which has the largest clear zone for coconut oil paper soap, while treatment B (11.96 mm) gained the largest inhibitory diameter on VCO paper soap. It is assumed that the more glycerine added, it will increase the antibacterial activity up to certain concentration then it will decrease because the bacteria will difficult to diffuse.

3.2.7 Foam Stability

Foam stability is the consistency of the amount of foam produced by paper soap. The resulting foam on paper soap is smoother compared to regular soap. The foam can be stable in the presence of a foaming agent. Solutions containing surface-active agents will produce stable foams when mixed with water. Glycerin generally does not contain surface active ingredients so it does not have a significant effect on foam stability. The percentage of foam stability for treatment A, treatment B, treatment C, and D treatment was presented in Figure 9.

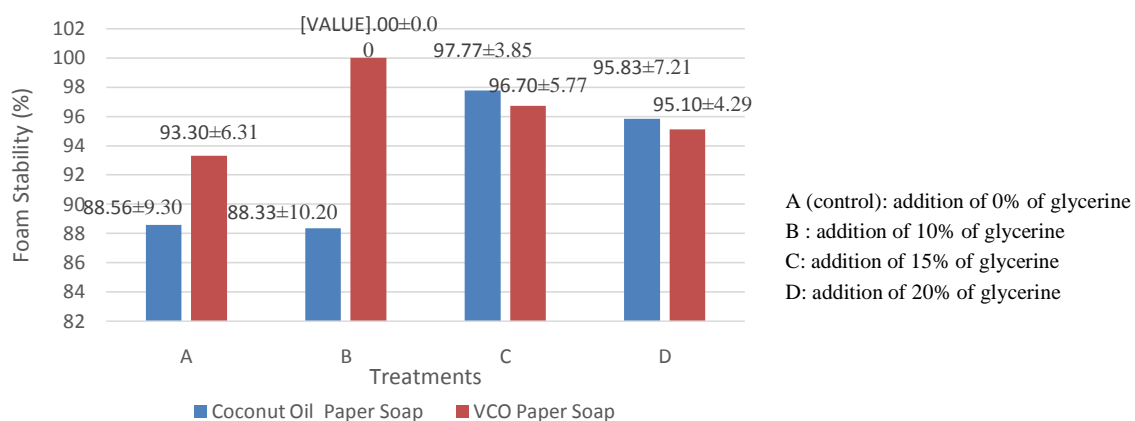


Figure 9. The relation between the glycerine addition to the foam stability.

The highest percentage of foam stability of paper soap produced is in treatment C that is 97.77%. This is assumed because in the second treatment, the value of each loop of high measurement of paper soap foam is

generally unchanged after being silenced for approximately 1 hour. A stable foam is also affected by the type of fatty acids used. In coconut oil there are lauric and myristic acids that can produce soft foams, while palmitic and stearate acids have a stabilizing properties of foams. While oleic acid and risinoleat can produce a stable and soft foam [11].

3.2.8 Overall Results Paper Soap in term of Physicochemical Properties and Organoleptic Quality

The paper soap with glycerine addition was tabulated in each treatment and compared with solid soap's standard including Indonesia National Standard SNI3532[12] and ASTM D 1172-95. Here are the recapitulated results which were presented in Table 1 and Table 2. The result of physicochemical characteristics for bothcoconut oil-paper soap and VCO-paper soap revealed that treatment C (the addition of glycerin 15% (w/w)) was the best soap formulation. Table 3 and Table 4 revealed the organoleptic results. For organoleptic tests performed thatboth paper soap treatment D (20% w/w glycerine) were preferred by the most panelists.

Table 1. Overall Results of Physicochemical Properties and Quality of VCO based Paper Soap

Parameters	Analysis Result				Standard
	The addition of glycerine (w/w)				
	A 0%	B 10%	C 15%	D 20%	
Water content (%)	23.14	20.44	18.47	17.32	Max. 15 [*]
Free Alkali content (%)	0.54	0.22	0.17	0.21	-
Insoluble Material in Ethanol (%)	2.32	1.26	3.49	2.73	-
Unsaponifiable Fatty matter (%)	5.27	3.97	4.91	3.07	-
pH	10.79	10.57	10.03	10.87	9 – 11 ^{**}
Foam stability (%)	93.30	100.00	96.70	95.10	-
Antibacterial activity (mm)	13.12	11.96	15.28	13.91	-

*: (SNI 3532:2016) , **: ASTM D 1172-95 (2001)

Table 2. Overall Results of Physicochemical Properties and Quality of Coconut Oil Based Paper Soap

Parameters	Analysis Result				Standard
	The addition of glycerine (w/w)				
	A	B	C	D	
	0%	10%	15%	20%	
Water content (%)	22.01	15.63	13.72	13.38	Max. 15*
Free Alkali content (%)	0.69	0.25	0.21	0.23	-
Insoluble Material in Ethanol (%)	2.57	3.38	3.93	3.45	-
Unsaponifiable Fatty matter (%)	1.15	3.63	4.06	4.44	-
pH	11.63	10.67	10.78	10.91	9 – 11**
Foam stability (%)	88.56	88.33	97.77	95.83	-
Antibacterial activity (mm)	6.74	9.28	11.66	11.39	-

*: (SNI 3532:2016) , **: ASTM D 1172-95 (2001)

Table 3. Overall Results of Organoleptic Quality of VCO based Paper Soap

Parameters	Analysis Result			
	The addition of glycerine (w/w)			
	A 0%	B 10%	C 15%	D 20%
Texture (Elasticity)	2.57	2.87	2.97	2.93
Aroma	2.87	3.07	3.17	3.17
The Amount of Foam	2.27	2.70	3.17	3.43
Power of Detergency	2.53	2.77	3.13	3.23
After Usage Impression	2.47	2.73	3.10	3.13

Table 4. Overall Results of Organoleptic Quality of Coconut Oil based Paper Soap

Parameters	Analysis Result			
	The addition of glycerine (w/w)			
	A 0%	B 10%	C 15%	D 20%
Texture (Elasticity)	2.63	2.66	2.93	3.10
Aroma	2.60	2.86	3.03	3.16
The Amount of Foam	1.93	2.50	2.90	3.20
Power of Detergency	2.40	2.63	3.03	3.16
After Usage Impression	2.43	2.73	2.93	3.06

4. Conclusions

The process of making paper soap is performed by hot process soap making by using hot plate stirrer as heating medium. The process of molding paper soap, paper soap is first molded as a solid soap by using silicon mold and then the size is reduced using slicer to form paper soap with size 2 x 2 cm and thickness 1 mm. Paper soap formulations in this study consisted of coconut oil (or VCO), 30% NaOH, stearic acid, aquades, 96% alcohol, fragrance oil, and glycerine with concentrations of 0% (w/w), 10% (w/w), 15 % (W/w), and 20% (w/w). Based on this, the best paper soap from organoleptic test was treatment D (paper soap with 20% glycerin (w/w) concentration added). Meanwhile, based on physicochemical properties test result, the treatment C (paper soap with addition of 15% glycerine (w/w) concentration).

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