

PHYSICOCHEMICAL ANALYSIS FOR QUALITY DETERMINANT OF NATURAL BAR SOAP WITH BINAHONG LEAF EXTRACT AND LEMONGRASS ESSENTIAL OIL

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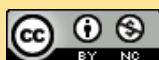
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ABSTRACT

There are two kinds of coconut oil in market that could be a natural soap base, filtered and unfiltered coconut oil that made the differences in fatty acid levels. In the previous study, the unfiltered coconut oil has the higher fatty acid than filtered coconut oil. It might could decrease pH level in the bar soap. It could appear caused by the excess of free fatty acid in the soap that not react with the lye and peroxidation may occur than made the rancid smell in the bar soap during the storage. In addition, the use of herbal component like plant extract and essential oil would contribute to pH level and water content of the soap that could affect the soap quality. Both parameters must be evaluated to determine the bar soap quality. This study used herbal extract of binahong leaf and serih wangi essential oil. There was divided to the four groups: formula 1 using filtered coconut oil and herbal extract, formula 2 using unfiltered coconut oil with herbal extract, formula 3 using filtered coconut oil without herbal extract, and formula 4 using non filtered coconut oil. The quality of the natural soap was evaluated for organoleptic, the pH level, moisture content, and the TLC Profile of the bar soap solution. All formulas were evaluated and all of them had green and white porcelain colour, hard soap, a pH between 9-10 on day 38, moisture content up to 15,5%, so from SNI 2021 the soap had a good quality.

Keywords: Binahong; lemongrass essential oil; Soap TLC; pH level; water content

1. INTRODUCTION

Natural bar soap was a choice for someone that has sensitive skin (Aznury et al., 2022). The synthetic bar soap uses Sodium Lauryl Sulfate (SLS) as surfactant that could irritate sensitive skin (Masunaga et al., 2021). In contrast, natural soap uses a natural polymeric surfactant that does not irritate the skin, could moisturize the skin, and is eco-friendly. The polymeric surfactant was a fatty acid salt that was made from saponification reaction between alkali and vegetable oil's fatty acid (Bhalerao & Karadbhaje, 2019). Many vegetable oils that could be a source of the fatty acid such as palm oil, coconut oil, olive oil, sunflower seed oil, canola oil etc (Cobzaru et al., 2017). They have the difference in kind of saturated fatty acid and unsaturated fatty acid (Dayrit, 2015). The saturated fatty acid will improve the hardness level of the bar soap than unsaturated oil (Mohammed et al., 2021). The use of coconut oil as saturated fatty acid will result the harder soap with high capability to make foam (Widyasanti et al., 2018). In the previous study, the used of vegetable oil as source of fatty acid in bar soap was combined over 1 kind of oil (Asemave & Edeka, 2021). Coconut oil contains fatty acid like lauric acid as low molecular weight saturated fatty acid, oleic acid and linoleic acid as

unsaturated fatty acid. It has carboxylic acid compound that act as antioxidant and esters aromatic group that could be softening and hydrating agents that good for the skin (Oji & Vivian, 2020). It might be possible to use coconut oil as a single oil for bar soap formulation which will reduce production cost and increase the profit. However, soap that used coconut oil has a demerit that it is easily has a rancid smell. The different quality of the soap from two kind of coconut oil in the market, filtered and unfiltered coconut oil might be caused by fatty acid peroxidation. Coconut oil has peroxide value between 0,11-0,12 meq/kg that include in low oxidation state (Oji & Vivian, 2020). The unfiltered coconut oil has higher level of fatty acid than filtered coconut oil (Pimentel et al., 2015). The pH level of the bar soap could be lower when any free fatty acid that not react with alkali (Supraptiah et al., 2022). However, there are lower quality caused by peroxidation of free fatty acid occuring on the bar soap, and a rancid smell appears.

The rancid smell in bar soap might be prevented using herbal component for aromatic scent, but the herbal component in the bar soap such as an extract from the plant has many phytochemical compounds like an essential oil that could be increase the level of water or moisture content (Handayani et al., 2021). The soft or mushy bar soap was caused by a high level of moisture content, which would be a reason for the low soap hardness, as well as organoleptic soap quality (Supraptiah et al., 2022). The quality of bar soap in Indonesia is regulated by Indonesian National Standard (SNI 3532:2021) where the good soap bar has water content not more than 23% and pH of 0,1% soap solution between 6,0-11,0 (BSN, 2021).

In this study, coconut oil used as single oil as a source of saturated and unsaturated fatty acid for saponification with sodium hydroxide to produce Sodium laurate as polymeric surfactant. The quality of natural soap bar with two kind of coconut oil (filtered and unfiltered) as single oil soap base with herbal component from binahong leaf ethanolic extract (*Anredera cordifolia* (Ten) Steenis) and essential oil from sereh wangi or lemongrass (*Cymbopogon nardus*) determined using two parameters, which are moisture content and pH level. Soap organoleptic was tested as smell, qualitative hardness, and colour during 38 and 180 day (1 month and 6 months). Chromatography profile used as fatty acid qualitative identification using iodine vapor as a novelty of detection of polymeric surfactant in herbal soap with four compotitions: there was coconut oil, binahong ethanolic extract, lemongrass essential oil, and sodium hydroxide.

2. METHODS

Coconut oil filtered and unfiltered from coconut milk shop in Purwomartani, Kalasan, Yogyakarta, Rotary evaporator Heidolph, pH meter, desiccator, oven (Mettler UN 110). Binahong dry powder leaf was extracted using maceration method with 2 times immersion for 24 hour each immersion and stirring every 30 minutes using ethanol 96% as the solvent (Karimatulhaji, 2020). The thicked extract was used as herbal component in the natural soap bar combined with sereh wangi oil that the soap bar made by cold process soap. The soap bar curing for the 38 day and 180 days to evaluated the organoleptic, pH level and moisture/water content used method in SNI 3532:2021 (BSN, 2021). Then phytochemical in the soap analyzed by TLC. Grouping and formula composition are listed in Table 1.

Table 1. Grouping and composition of formula

Composition	FEO	UEO	SBF	SBU
Binahong leaf extract	1% of oil	1% of oil	-	-
Sereh wangi oil	1% of oil	1% of oil	-	-
Filtered Coconut oil	50 gram	-	50 gram	-
Unfiltered Coconut oil	-	50 gram	-	50 gram
Sodium Hydroxide	18.3% of oil	18.3% of oil	18.3% of oil	18.3% of oil
Water	34.6 % of oil	34.6 % of oil	34.6 % of oil	34.6 % of oil

FEO (Filtered coconut oil with binahong extract and sereh wangi essential oil); UEO (Unfiltered coconut oil with binahong extract and sereh wangi essential oil); SBF (Soap base with Filtered coconut oil); SBU (Soap base with unfiltered coconut oil)

3. RESULTS AND DISCUSSION

3.1. Organoleptic, moisture content, and pH level

Soap was resulted from saponification reaction between the lauric acid and other (fatty acids in coconut oil) and the strong base NaOH. Coconut oil has saponification value at 127,62 mg KOH/g oil (Oji & Vivian, 2020). The reaction that occurs produces a fatty acid salt (sodium laurate) which acts as a polymer surfactant and glycerine as the skin moisture (Bhalerao & Karadbhajne, 2019). Strong base NaOH was a solid that is slowly dissolved in water and exothermic reaction occurs that causes the temperature in the solution to increase up to 80° C (Handayani et al., 2021). If water added to solid NaOH in a container, the temperature increase is too high and drastic that it can cause burns if exposed to hands. Therefore for inhibit the drastic increase temperature, the solid NaOH dissolved in the water jar gradually.

Soap organoleptic is a physical parameter related to the quality of soap. Hard soap indicates low water content or moisture, not brittle that can increase the shelf life of soap (Aznury et al., 2022). The soap used for this parameter has been stored for 1 year from the manufacturing process. The results of the soap organoleptic test showed that in the herbal soap group and soap base using coconut oil with filtration had a non-rancid aroma. Besides that, the soap in SBU group had a rancid aroma because of unfiltered soap-based usage (Table 2). The rancid smell produced can occur due to the fatty acid peroxidation process of excess oil that is not saponified (Oji & Vivian, 2020). As the theory, soap with refined coconut oil has fewer fatty acids compared to unfiltered raw coconut oil so if the fatty acids have been fully saponified, no remaining fatty acids of coconut oil will undergo a peroxidation reaction. Therefore, the use of refined coconut oil as a solid soap base ingredient can improve the physical quality of soap. In the previous study, binahong leaf extract and citronella oil, have antioxidant and antibacterial activities, that might improve the quality and shelf life of soap (Fachriyah et al., 2019).

The saponification reaction process continues until the soap is neutralized until the content in the mixture is saponified. The process that occurs including the hydrolysis reaction will produce water vapor up to day 180 during the storage after removing the soap from the mold. If the weight still changed during the storage it was indicated that the saponification process not over yet (Table 2). The bar soap tested for water content to determine the water content or moisture content in the soap. In the Indonesian National Standard Number 3532 of 2021 (SNI 3532:2021), there is a requirement for a maximum soap water content of 23% (BSN, 2021).

Table 2. Organoleptic bar soap stability in 1 year storage in room temperature

Group	Colour	Hardness	Smell
FEO	Green	Hard	Aromatic Lemongrass oil
UEO	Green	Hard	Aromatic like ant
SBF	White	Hard	Coconut oil aromatic
SBU	Porselen white	Hard	Rancid

FEO (Filtered coconut oil with binahong extract and sereh wangi essential oil); UEO (Unfiltered coconut oil with binahong extract and Lemongrass essential oil); SBF (Soap base with Filtered coconut oil); SBU (Soap base with unfiltered coconut oil)

In the study resulted shows that the soap with two kind of coconut oil in all formula still within the allowed range of water content in SNI because the soap has moisture content level between 15-16% after 38 day curing time. The soap making method with increase the temperature mixing like in the hot process soap could accelerate the saponification process so it would decrease the curing time until the soap neutrilized about 2 weeks (Hayati et al., 2020). The mixing process between the oil and the lye water was affecting the yield of the soap. If the mixing process used different temperature between oil and the lye solution, it will be separated to 2 phase that the soap failed to saponificated. The perfect saponification was related to homogenous mixing in the same temperature. The final product of the soap will have free alkali that not react with fatty acid, and during the storage it would remove the unsaponified oil to the final product. Therefore need to maintain the mixing process until the trace happened. In the single used of coconut oil as the base soap had the long time until the trace level occure (Mohammed et al., 2021). The water content in day 180 was tested and it had moisture content decreased. This shows that the soap still undergoing hydrolysis (Table 3).

Table 3. Percent of Moisture content

Group	W ₀ (g)	W ₁ (g)	Moisture content (%)	
			38 day	180 day
FEO	5.02±0.01	4.26±0.02	15.02±0.45	12%±3.4%
UEO	4.99±0.02	4.24±0.01	15.02±0.3	12%±4.9%
SBF	5.03±0.02	4.22±0.03	16.10±0.25	14%±0.9%
SBU	5.042±0.01	4.23±0.01	16.02±0.12	15%±0.4%

FEO (Filtered coconut oil with binahong extract and sereh wangi essential oil); UEO (Unfiltered coconut oil with binahong extract and sereh wangi oil); SBF (Soap base with Filtered coconut oil); SBU (Soap base with unfiltered coconut oil)

The moisture content in soap consists of the water content and essential oils founded in binahong leaf extract and citronella oil. The test results (Table 3) showed that the herbal soap group had a smaller moisture content compared to the soap base. Binahong leaf extract and citronella oil contain fatty acids that could undergo a saponification process with NaOH. The saponification process is accompanied by a hydrolysis reaction that will reduce the moisture content in the soap (Purwanto et al., 2021). Therefore, it is possible that soap with herbal content, binahong extract and citronella oil has a smaller moisture content than a soap base alone. The smaller the moisture content or moisture content of the soap will be harder than the soap bar. The allowed moisture content in SNI 3532: 2021 is a maximum of 23%. The results of the moisture content test obtained was between 12% to 15% which shows that the moisture content in the soap was qualified with SNI. The moisture content is related to the hardness of the soap (Maulidya et al, 2020).

The degree of acidity of the solution also increases to 13 which indicates the lye solution is strongly alkaline (Figure 1). The lye solution will cause corrosion if it contacts to the skin. Therefore, in making soap, safety equipment such as masks and gloves are needed to avoid unwanted things. In this study, the saponification process that continues until the soap is neutral will make the soap have a pH between 9-10. The pH requirement for soap in Indonesian National Standard No. 3532 of 2021 is between 6 to 11. Acidic pH in soap can occur if an acidic component is added to the soap mixture or there is any free fatty acid that not saponified with NaOH. While the alkaline pH of soap can occur due to the excess of the lye (NaOH) that not react with fatty acid. There is an unsaponified content which is indicated by a high pH value or in other words there is a high free alkali content in the soap (Vidal et al., 2018).

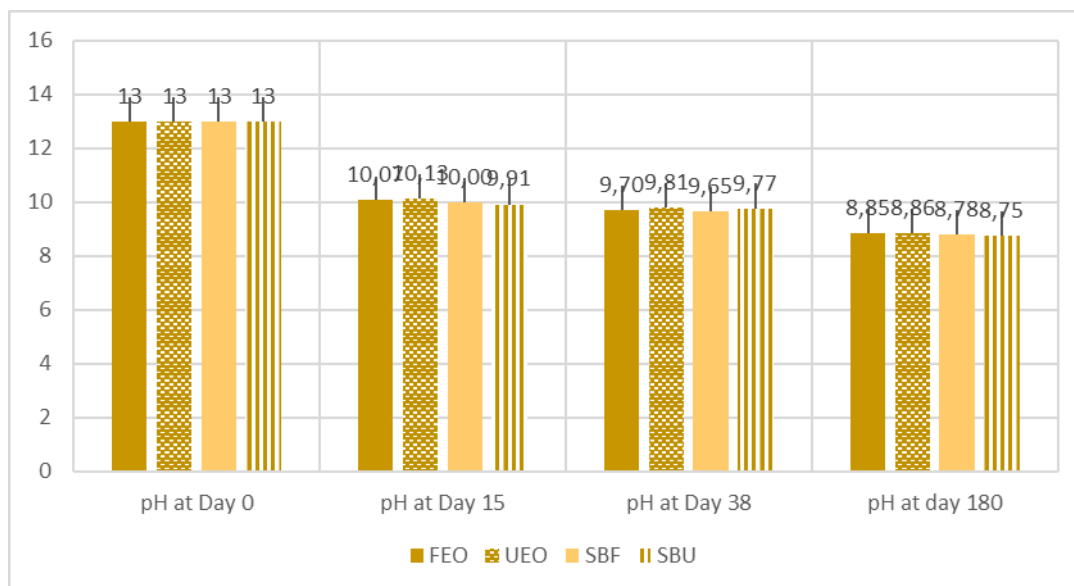


Figure 1. Diagram of pH after 2 week (day 15), 5 week (day 38), and 26 week (day 180) after unmolding soap, FEO (Filtered coconut oil with binahong extract and sereh wangi essential oil); UEO (Unfiltered coconut oil with binahong extract and lemongrass oil); SBF (Soap base with Filtered coconut oil); SBU (Soap base with unfiltered coconut oil)

The soap pH diagram shows that the soap at week 5 on day 38 has a pH between 9-10 which is not significant different between groups. On the 15th day, the pH of the soap was still above 10 and continued to fall until 38th day, indicating that the neutralization process was still taking place. On the 180 day the pH level was still decrease because hydrolysis and neutralization still occur. Level of pH between 8-10 on the soap is according to the SNI 3532:2021 that pH of 0,1% soap solution include good quality if the soap bar has pH level between 6,0-11,0 (BSN, 2021).

3.2. TLC

Binahong leaf ethanolic extract contains phytochemical compounds such as alkaloid, saponins, terpenoids, and flavonoids (Dwitiyanti et al., 2019; Fachriyah et al., 2019). The essential oil in citronella can only be detected with UV 254 light by visible dark blue blackout spots on a fluorescence green background. In it, there are semi-polar to non-polar compounds visible in the middle area of the plate to above the elution limit (Figure 2). Phytochemical compounds from binahong leaf extract and citronella oil have small levels in the soap because each soap with an average weight of 72 grams contains only 0.5 grams (0,69%) of binahong leaf extract and citronella oil each. Therefore, the TLC results from soap samples did not show obvious phytochemical compound spots because the concentration of the test soap solution made just only 1%.

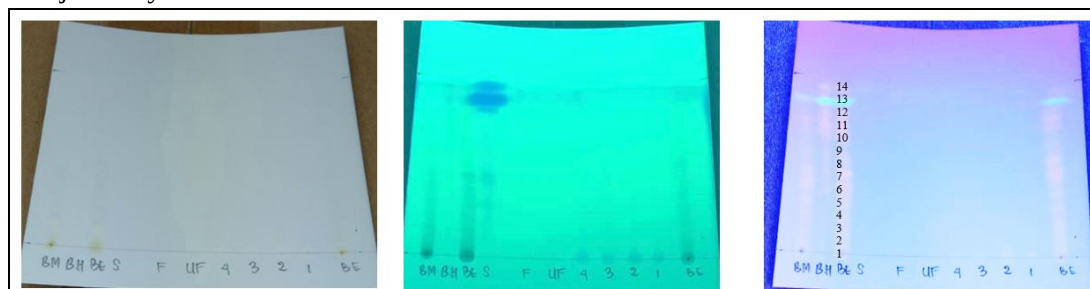


Figure 2. Chromatography profile of binahong-sereh wangi natural bar soap, BM (Extract of binahong in methanol); BH (Extract of binahong in hexan); BE (Extract of binahong in ethanol 96%); S (lemongrass oil); F (filtered/refined coconut oil); UF (unfiltered/nonrefined coconut oil); 4 (SBU); 3 (SBF); 2 (UEO); 1 (FEO), FEO (Filtered coconut oil with binahong extract and sereh wangi essential oil); UEO (Unfiltered coconut oil with binahong extract and sereh wangi oil); SBF (Soap base with Filtered coconut oil); SBU

(Soap base with unfiltered coconut oil). Stationary phase: TLC Silica Gel 60 F₂₅₄; mobile phase: Hexan:Ethyl acetate:Methanol:acetic acid (7:2:0,9:0,1). Detection:visible, UV light 254 nm and 366 nm

The presence of essential oils in citronella extract and oil would be shown in the results of testing phytochemical compounds using thin-layer chromatography (TLC). Solvents that have different polarities could separate phytochemical compounds with the appropriate polarity. Methanol is a polar solvent that will attract polar until nonpolar compounds (Xu et al., 2017). Hexane solvents have low polarity (non-polar) could separate semi-polar to non-polar compounds found in spots number 6 to 14 (Figure 2). The 96% ethanol solvent can separate polar to non-polar compounds in spots number 1 to 14. Pink spot can be seen on binahong extract using ethanol solvent with a 366 nm UV light detector. Spot number 13 with a greenish-yellow color is a non-polar group compound that can be dissolved with hexane and ethanol 96% (Karimatulhaji, 2020). The compound is still slightly soluble in methanol so that on the TLC plate there are still thin spots at number 13. While spot number 14 is the most non-polar compound in binahong extract.

The content seen on the TLC plate in the soap sample is a polymer surfactant which is a fatty acid salt from the saponification between lauric acid in coconut oil and NaOH to produce sodium laurate. The results of detection with iodine vapor showed the presence of brown color on the plate (Figure 3). The process of evaporation of TLC plates with crystalline iodine can be seen in Figure 4.

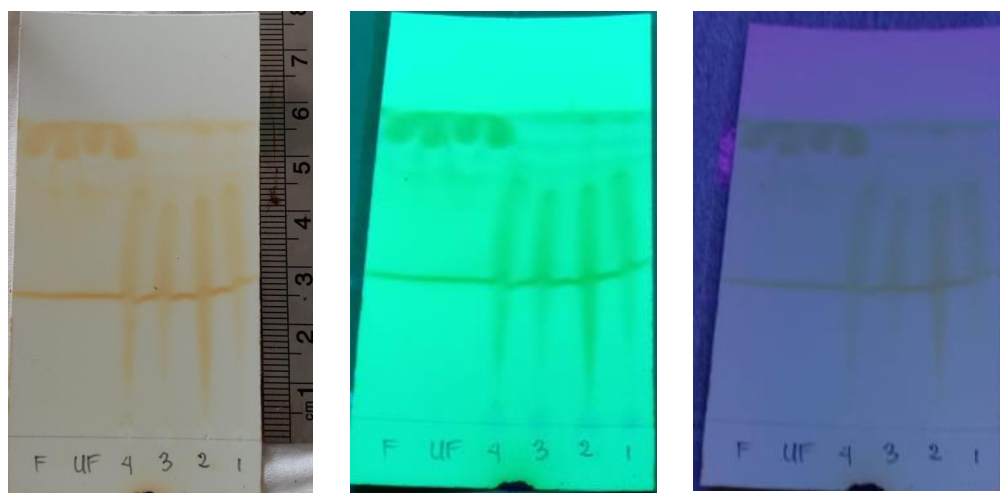


Figure 3. Detection of fatty acid in binahong-sereh wangi natural soap bar ; Stationair phase: TLC Silica Gel 60 F₂₅₄, Mobile phase : Hexan:Etil asetat:Metanol:Asam asetat 7:2:0,9:0,1. F (Filtered coconut oil); UF (Unfiltered coconut oil); 4 (soap base B); 3 (soap base A); 2 (herbal soap B); 1 (herbal soap A), Reagen detection: iodine vapor, visible, UV light 254 nm and 366 nm



Figure 4. Silica gel plat evaporation using Iodin cristal vapor for fatty acid detection

3.3. The influence of phytochemical compound in herbal extract to pH level and moisture content

Coconut oil has two kind of fatty acid, lauric acid as saturated fatty acid, oleic acid and linoleic acid as unsaturated fatty acid. The excess of fatty acid in coconut oil that might not react with the lye completely could decrease the pH level. However if the excess lye not react completely with fatty acid it would be increase pH level at the soap bar (Uzwatania et al., 2020).

In this study the free alkali and free fatty acid in the soap not determined, so that couldn't know what is the caused of the pH level between 9 until 10 in the soap product. In the previous study, the phytochemical constituent in the plant extract could be cause of low of pH level. The plant extract contains of acidic phytochemical constituent like flavonoid and phenolic acid that could decrease the pH level of the herbal soap (Megawati et al., 2022).

Flavonoid was found in the chloroform fraction of ethanolic binahong extract (Karimatulhaji, 2020). In the 50 mg of ethanolic binahong extract contain 1,35% of flavonoid. Vitexin as flavonoid was found in ethanolic binahong extract using 40%, 70%, and 96% ethanol as the solvent maseration method (Dwitiyanti et al., 2019). Phenolic acid p-coumaric acid found in ethanolic binahong extract (Fachriyah et al., 2019).

Sereh wangi/lemongrass oil (citronella oil) contains many of phytochemical constituent such as citronellal, citronellol and geraniol (Azmin et al., 2021) (Figure 5). They was an monoterpenoid compound with the presence in the sereh wangi (*Cymbopogon nardus*) essential oil was 11,3%, 3,22%, and 0,08% (Kamal et al., 2020). The oil could increase the level of moisture content of the soap (Aznury et al., 2022). In contrass with this study, soap with binahong extract and essential oil has lower moisture content than soap base without essential oil because it might saponification occure between NaOH with fatty acid from the binahong extract and the lemongrass essential oil. Sereh wangi oil contains fatty acid like lauric acid, myristic acid, palmitic acid, and stearic acid (Duru et al., 2020). While binahong ethanolic extract has fatty acid like hexadecanoic acid and linoleic acid (Feriyani et al., 2020) that might be saponified with NaOH caused by has saponification value (Tanko et al, 2017). So the moisture content of FEO and UEO group has a lower level than soap base without herbal component. In the analysis of moisture content using the gravimetric method, the vapor that out from the soap could contains excess of water and oil that was not saponified with NaOH.

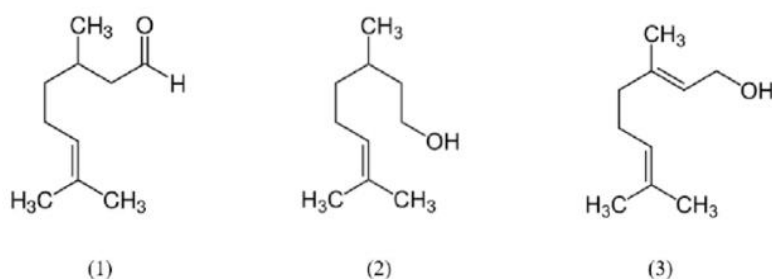


Figure 5. Molecular structures of citronellal (1), citronellol (2) and geraniol (3) markers in citronella oil (Higuchi et al, 2023)

3.4. The influence pH level, moisture content, and phytochemical constituent the plant extract in bar soap quality

The good quality soap bar has range 6,0-11,0 that still acceptable in the body skin. If the soap bar has pH level out from it range, it could be irritate or made the dry skin. The excess of fatty acid in coconut oil that might not react with the lye completely could made rancid smell during the soap storage. Phytochemical compound in binahong ethanolic extract like saponin, flavonoid, terpenoid, alkaloid and terpenoid from sereh wangi essential oil could be a source of antioxidant that could inhibit oxidation process from the excess fatty acid (Cobzaru et al., 2017).

Soap moisture content needs to be determined to know the stability of soap during the storage. The water content that exceeds the required value can cause mold growth on the soap so that it will affect the physical and chemical properties of soap (Aznury et al., 2022). The presence of a high-water content can also cause the soap to become soft and easy to melt. The excess of essential oil that not saponified could act as antioxidant that inhibit the rancid smell from the soap bar (Purwanto et al., 2021). In contrast with that, the excess of fatty acid from the extract that not saponified could increase the possibility rancid smell in the soap (Oji & Vivian, 2020).

4. CONCLUSION

The natural soap bar using filter an unfiltered coconut oil as the soap base would made the good quality from the pH level and water content level according to SNI 3532:2021. Coconut oil could be a base oil without combine it with other vegetable oil for the soapmaking because has complete kind of saturated and unsaturated fatty acid. It will be more profitable if the soap will be a hilirization product. Herbal component that added in the soap could be an antioxidant that inhibit the rancid smell to increase the stability and soapbar quality, beside it used as scent in the soap bar. However the excess of free fatty acid, unsaponified fat content and the soap hardness using this soap base should be determine in the future study to know the relation with the pH and water content level for determine the quality of the soap bar.

5. ACKNOWLEDGMENT

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6. CONFLICT OF INTEREST

All authors declared that there was no conflict of interest.

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