Development of lengkuas (*Alpinia galanga*) and cortex of banyuru (*Pterospermum celebicum, miq*) extract for topical preparation on validation methods analysis of total flavonoid levels

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Abstract. The research has been to develop cortex banyuru (*Pterospermum celebicum, miq*) [1] and lengkuas (*Alpinia galanga*) extract topical preparations to validate the method of analyzing total flavonoid levels [2]. The aim of this study was to find a method suitable for developing preparations with total flavonoid priority be applied to soap Banyuru and Lengkuas extracts [3]. Validation parameters used in this study are specificity, linearity, range, precision, and accuracy [4]. The results of the study will show that this method is specific and has a standardized linearity value, i.e., the range value, obtained the acquisition value, in the precision test obtained RSD (relative standard deviation) value. Based on the results of this study it can be concluded that the validation method of analysis of total flavonoid levels in soap [5]. Measurements of spectrophotometry visible with AlCl₃ reagents has specificity, linearity, range, accuracy, and precision showed the good results. The valid analysis method was then applied to the analysis of the total flavonoid content of 5 soap formulas extracted from Banyuru and Lengkuas bark, and from these results total flavonoid levels were obtained.

1. Introduction

The skin is the outermost layer of tissue that covers the entire body and protects the body from the dangers that come from outside, especially against bacteria. The skin barrier function is in the epidermis, the stratum corneum layer. This is due to the presence of intracellular lipids which are one of the constituents of the stratum corneum. In addition to protecting the body, the skin also functions as a place of excretion. Fatty substances, water, ions, and sweat are examples of skin excretion. The excretion mixes with dirt, causing many bacteria in the skin, and can cause infection if there is a vulnus on the skin [7].

Pharmaceutical dosage forms that can be used to maintain skin health, one of which is soap. Soap is a product that is produced from the reaction between fatty acids with strong bases that serve to wash and clean fats. Transparent soap is one type of soap that has an attractive appearance because of its appearance [7].
Besides being able to clean the skin from dirt, soap can also be used to maintain healthy skin from bacteria. Soap that can kill bacteria is known as antiseptic soap. In the market, many antiseptic soaps containing antibacterial agents such as triclosan, the use of triclosan can trigger antibiotic resistance. The antibacterial ingredients used can come from natural ingredients that contain antibacterial compounds, plants that have antibacterial compounds including Banyuru (*Pterospermum celebicum* Miq.) and Lengkuas (*Alpinia galanga* (L.)Willd.) [8].

Banyuru (*Pterospermum celebicum* Miq.) is a medium to large-sized tree, many contain chemical compounds such as flavanoids and tannins which can inhibit bacterial growth.

The classification of Banyuru plants (*Pterospermum celebicum* Miq.) as follows:

- **kingdom**: Plantae
- **Division**: Spermatophyta
- **Subdivision**: Angiospermae
- **Class**: Dicotyledoneae
- **Subclass**: Dialypetalae
- **ordo**: Malvales
- **family**: Sterculiaceae
- **genus**: Pterospermum
- **Species**: *Pterospermum celebicum* Miq. [1,9]. (Sulawesi : Banyuru)

It is a medium to large tree, about 10-15 cm tall and up to 100-120 cm in diameter. The leaves are single green slippery on the bottom of the leaves are brown-gray. The inflorescence is near the end. The flowers are yellowish-white and flowering in May-June, the surface of the bark is smooth, scaly or shallow-cut, flexible stringy inner skin. Contains chemical compounds such as tannins, catechins, phenols, steroids, fats, and water [1]. used to treat hives, boils, zits, colds, and dysentery [10,11].

**Soap** is sodium and potassium salts of fatty acids derived from vegetable oils or animal fats. The soap used as a cleaner can be solid (hard), soft and liquid. The National Standardization Agency states that soap is a material used for washing and emulsifying, consisting of fatty acids with carbon chains C12-C18 and sodium or potassium [12].

The main function of soap is as a cleaning agent. The soap lowers the surface tension of the water, allowing water to wet the washed material more effectively, the soap acts as an emulsifier to disperse oil or fat, and soap is adsorbed in the dirt granules [13].

The quality requirements of bath soap required by SNI 3532-2016 are as follows BSN (2016)

| No | Test Criteria | Unit | Quality |
|----|---------------|------|---------|
| 1  | Water content | % mass fraction | Max. 15,0 |
| 2  | Fat total     | % mass fraction | Min. 65,0 |
| 3  | Insoluble material in ethanol | % mass fraction | Max. 5,0 |
| 4  | Free alkali (as NaOH) | % mass fraction | Max. 0,1 |
| 5  | Free fatty acids (as oleic acid) | % mass fraction | Max. 2,5 |
| 6  | Chloride content | % fraksi massa | Max. 1,0 |
| 7  | Unsweetened fat | % fraksi massa | Max. 0,5 |

**Note:** Free alkaline or free fatty acids are choices depending on the nature of acid or base.
2. Methods

Table 2. The base formula for transparent solid soap

| materials          | Concentration % |
|--------------------|-----------------|
| Stearic acid       | 6.5             |
| Virgin Coconut Oil | 15.0            |
| Olive Oil          | 6.0             |
| NaOH 30%           | 20.0            |
| Etanol 96%         | 17.0            |
| Glicerin           | 12.0            |
| Sucrosa            | 10.0            |
| Trietanolamine     | 2.0             |
| Citric acid        | 4.5             |
| BHT                | 0.1             |
| Cocobetain         | 2.0             |
| Oleum citri        | 0.5             |
| Aqua destilata     | 4.4             |

Table 3. Transparent solid soap formula Banyuru bark extract and Galangal extract

| Materials          | concentration (%) | F0 | F1 | F2 | F3 | F4 |
|--------------------|-------------------|----|----|----|----|----|
| Lengkuas Extract   | -                 | 1.0|     | 1.0| 1.0| 1.0|
| Cortex Banyuru extract | -   | -  | 1.0| 1.0| 2.0| 3.0|
| Bases sabun ad     | 100               | 100| 100| 100| 100| 100|

2.1 Transparent Solid Soap Making Procedure

All ingredients are weighed first. Stearic acid is melted at 60 °C in a goblet above the water bath, then add the oil mixture (VCO and olive oil) and BHT to the cup and stir until homogeneous. 30% NaOH solution was added to the cup glass if the temperature had reached 70 °C and stirred for 2-4 minutes until soap was formed, the temperature was lowered to 50 °C, then added a mixture of glycerin, TEA, sucrose, cocobetain and citric acid which had been over first dissolved in hot water added to the mixture while continuing to stir for about 7-10 minutes until the mixture becomes homogeneous. Then slowly add 96% ethanol until a clear solution is formed. Banyuru bark extract and galangal extract were dissolved in 96% ethanol residue and added to the base mixture then stirred at 40 °C until homogeneous, then added orange oil and stirring again until homogeneous and put into transparent soap molds.

2.2 Validation analysis method[2].

2.2.1 Specificity test

the Specificity test was carried out by scanning the maximum absorption wavelength with the maximum absorption wavelength of the compound reaction between quercetin and Aluminium chloride. Observations of the spectrum produced and then compared are carried out.
2.2.2 Test Of Linearity
Measurement linearity of raw kuarsetin made in the solution of some of the concentration range. From raw kuarsetin stock solution is then made 7 series kuarsetin dilution in a flask 5 ml of tentukur and then added to mark the boundary with 5% acetic acid in methanol, whisk until homogeneous. Do replication 3 times. Then measured using a spectrophotometer at a wavelength of UV maximum. The correlation coefficient (r) was calculated from the linear regression analysis of $Y = a + bx$ on the raw curve

2.2.3 Presisition Test
Precision testing done with kuarsetin solution with 6 replication of the same concentration. Precision test is determined by the parameter at (Relative Standard Deviation) [14], with the formula:

$$ \text{RSD} = \frac{SD}{\bar{x}} \times 100\% $$

Description: $SD = \text{Standard Deviation}$$
$x = \text{average sample Levels}$

2.2.4 Acuration Test
the Determination of accuracy is done by a simulation method (spike-placebo recovery). In the method of simulating several some many pure material analytes added to the mixture of pharmaceutical preparation carrier materials [14].

2.3 Analysis of Total Flavonoids on Banyuru and Galangal Extract Soap

| Extract     | Formula 1 | Formula 2 | Formula 3 | Formula 4 | Formula 5 | Formula 6 |
|-------------|-----------|-----------|-----------|-----------|-----------|-----------|
| Banyuru     | 1         | 1         | 3         | -         | 1         | -         |
| Lengkuas    | 3         | 1         | 1         | 1         | -         | -         |

Ten grams of each formula soap weighed carefully and in accordance with the preparasikan sample preparation test on test accuracy that had been in the validation. The result of preparation then take 1 ml then incorporated into the next 5 ml tentukur flask added aluminium chloride 2% as much as 200 µl and and sodium citrate 100 µl. And then diluted with 5% acetic acid in methanol to limit sign. Absorbance is measured at a wavelength of maximal on the operating time accordingly. The absorbance obtained further processed to find out the levels of total flavanoid in preparations sops extracts banyuru and lengkuas.

3. Result and Discussion
3.1 Extraction results

| Extract     | Colour       | aroma | Weight of simplisia (g) | Weight of extract (g) | Persen Rendamen |
|-------------|--------------|-------|-------------------------|-----------------------|-----------------|
| Lengkuas    | Yellow-brown | specific | 300                     | 20,32                 | 6,7%            |
| Banyuru cortex | Red-brown   | specific | 500                     | 46                    | 9,2%            |
3.2 Test for the number of saponification of VCO and Olive Oil

Table 6. Results of measurement of the number of saponification of VCO and olive oil

|             | VCO       | Olive oil  |
|-------------|-----------|------------|
| Replication 1 | 224,4456  | 206,6162   |
| Replication 2 | 225,0569  | 208,2401   |
| Replication 3 | 223,4940  | 206,8124   |
| Mean         | 224,3322  | 207,2229   |
| Standard Deviation | 0.7876    | 0.8864     |

3.3 Organoleptic Observation of Solid Transparent Soap

Table 7. Observations organoleptik preparations of solid transparent SOAP

| Formula | Parameter | Before Storage | After Storage |
|---------|-----------|----------------|---------------|
|         | Color     | aroma          | Texture       | color         | Aroma | Texture |
| F0      | White-transparent | Orange oil   | Solid         | White-transparent | Orange oil | Solid |
| F1      | Brownish-yellow transparent | Orange oil | Solid         | Brownish-yellow transparent | Orange oil | Solid |
| F2      | Brown, transparent         | Orange oil   | Solid         | Brown, transparent         | Orange oil | Solid |
| F3      | Brownish-black transparent | Orange oil | Solid         | Brownish-black transparent | Orange oil | solid  |
| F4      | Brownish-black, almost transparent | Orange oil | Solid         | Brownish-black, almost transparent | Orange oil | solid  |
| F5      | Brownish-black, opaque     | Orange oil   | Solid         | Brownish-black, opaque     | Orange oil | solid  |

Description:
F0: SOAP Base without the content of the extract
F1: Soap with 1% concentration of extracts of Galangal
F2: the SOAP with a concentration of 1% extract of bark Banyuru
F3: Soap with extract concentration 1% White and 1% Banyuru bark extract
F4: Soap with 1% concentration of extracts of Galangal and 2% Banyuru bark extract
F5: Soap with 1% concentration of extracts of Galangal and 3% Banyuru bark extract
Evaluation of the organoleptic soap shows that use of galangal and extract extract of bark Banyuru effect on colors and levels of transparency transparent solid soap, due to the addition of extracts of Galangal and Banyuru on bark extract Soap.

3.4 Transparent Solid Soap Hardness Test Results

![Figure 1. Histogram of hardness test solid soap](image)

An examination of hardness looks solid soap that hardness increases each week, where the higher the concentration of the extract used the greater the hardness level SOAP anyway. In addition because the influence of the addition of extracts of galangal and Banyuru, the degree of the hardness soap was also influenced by the high concentration of stearic acid and sucrose are used, where the higher the concentration of stearic acid and also the greater the sucrose the resulting SOAP violence, while increasing the hardness of SOAP every week after storage due to the reaction of the saponifikasi began to react perfectly after 3-4 weeks storage [15].

Table 8. Results of measurement Total Flavanoid soap of Banyuru and Lengkuas Extract

| Formula | Absorbancy samples (a) | Average (a) | Absorbancy Blanko (b) | (a-b) | Levels of Flavanoid | Flavanoid Concentration |
|---------|------------------------|-------------|-----------------------|-------|--------------------|------------------------|
| 1 : 3   | 0.51578                |             | 0.51241               | 0.34075 | 0.17166          | 20.25                  | 0.0814%               |
| %RSD    | 0.51118                | 0.51028    | 0.575                 |       |                   |                        |                      |
| 1 : 1   | 0.38691                |             | 0.38405               | 0.31117 | 0.07288          | 20.20                  | 0.0448%               |
| %RSD    | 0.38027                | 0.38498    | 0.889                 |       |                   |                        |                      |
| 3 : 1   | 0.45670                |             | 0.45806               | 0.33230 | 0.12576          | 20.40                  | 0.0639%               |
| %RSD    | 0.45896                | 0.45853    | 0.261                 |       |                   |                        |                      |
| 0 : 1   | 0.31231                |             | 0.31259               | 0.27694 | 0.03565          | 20.85                  | 0.0299%               |
| %RSD    | 0.31214                | 0.31334    | 0.207                 |       |                   |                        |                      |
| 1 : 0   | 0.21048                |             | 0.21494               | 0.20859 | 0.00635          | 20.80                  | 0.0194%               |
| %RSD    | 0.21380                | 0.21615    | 1.334                 |       |                   |                        |                      |
3.5 High-foam solid transparent soap

![Weight of foam result](image1.jpg)

**Figure 2.** Histogram high test result foam solid transparent soap

High check foam solid transparent soap, the height of foam is increasing every week, this is because the response will be formed perfectly after 3-4 weeks of storage. The addition of Lengkuas extracts and Banyuru stem bark extracts influences the high-foam soap, where the foaming ability of soap decreases with the addition of Lengkuas and Banyuru extracts, in soaps with the combination of Lengkuas and Batang Bark extract Banyuru increasingly higher concentration of Banyuru extract is decreasing also high soap foam. In all formulas, after five minutes of testing, the height of foam is not reduced, this is due to the use of surfactant cocobetain and the use of TEA capable of delivering a stable foam after five minutes of testing [16].

3.6 pH measurement for Transparent Solid Soap

![pH measurement result](image2.jpg)

**Figure 3.** Histogram results from a pH test for transparent solid soap

High check foam solid transparent soap, the height of foam is increasing every week, this is because the response will be formed perfectly after 3-4 weeks of storage. The addition of Lengkuas extracts and Banyuru stem bark extracts influences the high-foam soap, where the foaming ability of soap decreases with the addition of Lengkuas and Banyuru extracts, in soaps with the combination of Lengkuas and Batang Bark extract Banyuru increasingly higher concentration of Banyuru extract is decreasing also high soap foam. In all formulas, after five minutes of testing, the height of foam is not reduced, this is due to the use of surfactant cocobetain and the use of TEA capable of delivering a stable foam after five minutes of testing [1].
The pH inspection of transparent soap extracts of Lengkuas and bark extract of Banyuru stem is carried out every week until the fourth week of storage time. Can be seen that the pH of soap ranges between 8.83 – 9.23. The base Formula (F0) of the pH obtained is quite alkaline ranging from 8.83 – 9.87. According to Febriyenti [16]. A pH soap that is sufficiently alkaline when used will increase the pH of the skin, but the skin has the ability to restore the skin's pH as it was originally immediately after rinsed within 15-30 minutes. This buffer effect is due to the amino acid content contained in the skin components. In F1 there is a decrease in soap pH, this is due to the addition of Lengkuas extracts that have a pH of acids, whereas in F2, F3, F4, and F5 occur increased dosage of the dosage due to the addition of the bark extract of Banyuru stem which is alkaline, which The pH of Banyuru stem bark extract used is 10.2. According to the research results Febriyenti, Dkk, 2014, pH of transparent solid soap circulating in the market range 9.45-9.59 and the requirement of solid soap pH required by SNI 3532-2016 is around 8-11, so based on the results of pH measurements, all formulas Transparent soap meets the requirements of the soap quality standard SNI 3532-2016.

3.7 Transparent solid SOAP Moisture content

| Moisture content test results |
|-----------------------------|
| F0  | F1  | F2  | F3  | F4  | F5  |
| 40  | 35  | 30  | 25  | 20  | 15  |
| 10  | 5   | 0   | 10  | 15  | 20  |
| 0   | 5   | 10  | 15  | 20  | 25  |
| Minggu ke 1 | Minggu ke 2 | Minggu ke 3 | Minggu ke 4 |

Figure. 4. Histogram solid transparent SOAP water content test result

3.8 Alkaline Free Solid Soap Transparent

Moisture content in solid soaps affects the quality of the dosage. Water added in soap products can affect the solubility of soap in water. According to Hambali [15]. The more water is contained in the soap, the soap will be easily shrinking and quickly depleted at the time of use. In Figure 4, the water content of the transparent solid soap formula is reduced with the addition of Lengkuas extract and Banyuru stem bark extract, in the soap with the combination of Lengkuas extracts and the bark extract of Banyuru rod also shows a Less transparent if the concentration of stem bark extract Banyuru left. Soap water content in the fourth week ranged from 9.80%-13.87%. The quality requirement of solid soap set by SNI 3532-2016 is ≤ 15% thereby all formulas meet the requirements stipulated by SNI.
Free alkaline examination of transparent soap extract Lengkuas and bark extract of Banyuru stem is carried out on the fourth week of storage time. Soap-free alkaline levels range between 0.0409% – 0.0848%. In the free alkali number testing, the higher the concentration of the extract the lower the liquid-free alkaline solid soap is produced. A solid soap-free alkaline level based on SNI 3532-2016 is < 0.1%, so all formula of transparent soap meets the standard of free alkaline levels required by SNI.

4. Conclusion
Based on the research results it can be concluded that:

a) Banyuru Bark extract and galangal extract and can be formulated in transparent solid soap.

b) The method of Spectrophotometry visible with the use of AlCl3 reagents as a complex forming can be used in the analysis of total flavanoid levels in soap, evidenced by looking at the results of the validation testing parameters such as, accuracy, precision, Specificity, linearity and range.

c) The method of analysis of the total amount of flavanoid obtained can be applied to the soap of bark extract Banyuru rod (*Pterospermium Celebicum*, Miq) and Lengkuas (*Alpinia Galanga*) with the results and spread of data is quite good.

Acknowledgement
It is recommended that advanced testing related to soap irritation test and advanced test related to the influence of VCO use, and olive oil to Banyuru root extract.

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