

FORMULATION AND DETERMINATION OF TOTAL FLAVONOID CONTENT OF FACE WASH GEL COCOA FRUIT EXTRACT (THEOBROMA CACAO L.) USING UV-VIS SPECTROPHOTOMETRY

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ABSTRACT

Cosmetic products for skin care have recently been very popular among consumers, especially women. One type of face wash preparation that is used every day is face wash gel, this preparation is very suitable for dealing with facial skin problems. Plants or natural ingredients that can be used to formulate face wash gel preparations are cocoa fruit (*Theobroma cacao* L.) These compounds can be grouped into secondary metabolite compounds which are biomolecules that are used as new discovery materials in the development of herbal cosmetics. This research aims to formulate a face wash gel preparation and determine the total flavonoid content of the face wash gel from cocoa fruit extract (*Theobroma cacao* L.). This research uses a laboratory experimental research design by extracting cocoa pods using the maceration method with methanol solvent. Cocoa fruit extract face wash gel was formulated with a concentration of 2.5%, 5%, 7.5%, the formula was tested for the physical properties of the preparation, namely organoleptic test, homogeneity test, spreadability test, foam height test, pH test and viscosity test. Then the total flavonoid content of formula 3 face wash gel was determined using UV-Vis spectrophotometry. The results of the physical properties of the face wash gel preparations with concentrations of 2.5%, 5% and 7.5% met the test parameter requirements, while the results of determining the total flavonoid content of the formula 3 face wash gel received a value of 0.0005/mg/QE/g. Test of the physical properties of the face wash gel preparation from cocoa fruit extract (*Theobroma cacao* L.) meets the requirements and the face wash gel preparation in formula 3 cocoa fruit extract (*Theobroma cacao* L.) contains total flavonoid levels of 0.0005/mg/QE/ g.

Keywords: cocoa fruit; face wash gel, UV-Vis spectrophotometry; *Theobroma cacao* L.;



INTRODUCTION

Cosmetic products for skin care are very popular among customers, especially women, these days. Skin care is very important to keep your skin healthy and beautiful *face wash* is the most frequently used skin care product every day. One type of preparation *face wash* which is used every day, namely gel *face wash*, this preparation is very suitable for dealing with facial skin problems, especially choosing a gel preparation because the gel preparation has thixotropic properties *that is* solid when stored and will melt immediately when shaken, gel only leaves a thin layer on the skin of the face and is easily washed off with water and leaves a cool sensation after use, this preparation is also able to penetrate further than cream. Products sold on the market are mostly made from chemicals such as *asniacinamide*, *salicylic acid*, *lactid acid*, *retinol*, for now the preparation of the preparation *face wash* has begun to be developed using natural ingredients as active ingredients in formulations *face wash*. Therefore, in this study, researchers formulated a gel *face wash* from natural ingredients because they are healthier and of course safe for facial skin formulation of the preparation *face wash* use *gelling agent* in this formulation, the physical properties of the gel produced are greatly influenced by the base used in *face wash* is carbopol-940 which is a synthetic polymer that is easily dispersed in water and functions as a gel base with fairly good viscosity (Rasydy *et al.*, 2020).

Plants or natural ingredients that can be used to formulate gel preparations *face wash* namely cocoa fruit (*Theobroma cacao* L.). Cocoa fruit contains compounds such as polyphenols, flavonoids and tannins. These compounds can be grouped into secondary metabolite compounds which are biomolecules used as new discovery materials in the development of herbal cosmetics. One of the secondary metabolite compounds contained in cocoa fruit (*Theobroma cacao* L.) is a flavonoid. The mechanism of action of flavonoids as antibacterials is to form complex compounds with dissolved proteins so that they can damage the bacterial cell membrane followed by the release of intracellular compounds. To determine the concentration or total flavonoid levels, quercetin is used as a comparative standard because it is a type of flavonoid compound (flavonol) with a keto group at C4 and the hydroxyl group at C3 or C5 which can help to provide complex colors with $AlCl_3$. While for the formulation of the preparation *face wash* use *gelling agent* In this formulation, the physical properties of the gel produced are greatly influenced by the base used in *face wash* is carbopol-940. Therefore, this study aims to formulate and determine the gel content *face wash*. Cocoa fruit extract with a good understanding of the physicochemical properties of cocoa fruit extract and the interaction of the extract with other components in the formulation, is expected to produce a product that is safe, effective and meets consumer needs (Nurkholis *et al.*, 2021).

METHOD

The time and place of the research were carried out from March to May 2024 at the Dr. Soebandi University Laboratory, Jember. This research design uses a laboratory experimental study with data collection techniques.



namely the process of formulating gel preparations *face wash* cocoa fruit extract (*Theobroma cacao* L.), physical properties test of the preparation, and determination of total flavonoid content of the gel *face wash*. The population of this study was cocoa plants. (*Theobroma cacao* L.), and the sample in this study was cocoa fruit extract (*Theobroma cacao* L). Sampling technique by extracting cocoa fruit (*Theobroma cacao* L.) until it becomes a thick extract that can be formulated into a gel *face wash* cocoa fruit extract (*Theobroma cacao* L.).

Tools and materials

The tools used in this research process are maceration containers, blenders, analytical scales, filter paper *whatman* No.1, aluminum foil, *glasswear*, funnel, mortar and pestle, analytical balance, measuring cylinder and funnel, test tube, watch glass, digital pH meter (DrGray), viscometer (*riion* VT-06), UV-Vis spectrophotometry (pharo 300).

The materials used in this research process are filter paper and dry cocoa fruit samples (*Theobroma cacao* L), methanol, cocoa fruit extract, glycerin, sodium lauryl sulfate, methyl paraben, carbopol, TEA, gelface wash $AlCl_3$, quercetin (*sigma*).

Plant Determination

Determination of cocoa plants was carried out by the Batu Herbal Materia Medica Laboratory UPT, Batu City, to determine the identification of the plants to be studied.

Cocoa Fruit Extraction Process (*Theobroma cacao* L.)

Cocoa fruit sample (*Theobroma cacao* L.) in powder form as much as 2000 grams was extracted by maceration using methanol with a ratio of 1:5 for 24 hours. After the sample was filtered, the extract was evaporated using *rotary evaporator* until it produces a thick extract.

Formulation Gel Face Wash

Preparation of gel preparations *face wash* from cocoa fruit extract (*Theobroma cacao* L.) is done by mixing distilled water, methylparaben and glycerin, then homogenizing it with *magnetic stirrer*. After that add Na-lauryl sulfate, heat the solution to a temperature of 40°C, transfer to a hot mortar, then add the thick cocoa fruit extract (*Theobroma cacao* L.) little by little on a hot mortar and stir until homogeneous, then add carbopol 940 little by little while stirring until all mixed and finally add TEA and stir until homogeneous. Do the same for formulas F2 and F3 (Junaidi *et al.*, 2020) .

Preparation of Quercetin Standard Solution

Quercetin as much as 25 mg was weighed and dissolved in 25 mL of ethanol as a 1000 ppm stock solution. The standard quercetin solution was made by diluting it with a concentration of 100 ppm as much as 50 (Ramadhani, *et al.*, 2023).

Determination of Total Flavonoid Levels

The maximum wavelength for determining the content is 443nm, the content determination is carried out by making a gel solution. *face wash* concentration of 1800 ppm into 10 mL of ethanol. Replication was done 3 times. Pipette 1 mL then put into a 10 mL measuring flask, add 0.1 mL of 10% $AlCl_3$, 0.1 mL of 1 M sodium acetate, 2.8 mL of distilled water, and 96% ethanol to the limit mark. Flavonoid levels were determined based on absorbance



interpolation into the standard linear regression equation of quercetin to obtain the sample concentration (Handoyo *et al.*, 2020).

$$\text{Total Flavonoid} = \frac{\text{Consertation } \mu\text{g} \times \text{Absorbance mL}}{\text{Weight sample}} \times \text{fp}$$

Evaluation of Gel Preparations *Face Wash*

Organoleptic Test

Organoleptic tests observe gel preparations including shape, color and odor analyzed using our five senses, namely with the help of the eyes and nose (Syamsul *et al.*, 2020).

Spread Power Test

A total of 0.5 grams of gel preparation *face wash* placed on top *object glass* and closed, then given a load of 50 grams, 100 grams, 150 grams on top of each load, given a time of 1 minute and measure the diameter for each additional load, then average the diameter. The requirement for a good spreadability test is 5-7 cm (Lestari G *et al.*, 2020).

pH Test

A total of 1 gram of preparation *face wash* diluted with distilled water up to 10 ml. Insert the digital pH meter into the gel solution and then observe the pH value on the pH meter monitor. The requirements for a good pH gel test are 4-8 (Supriyani *et al.*, 2020)

Viscosity Test

Viscosity test is carried out using a viscometer *ronby* putting 100 mL of gel into a tube-shaped container and then installing it *spindle* No.2, the spindle must be immersed in the test preparation. The viscometer is turned on and the rotor is ensured to rotate at a speed of 60 rpm. The viscometer pointer is observed pointing to the number on the viscosity scale, then recorded and multiplied by a factor of 100. The viscosity of the gel preparation meets the requirements, namely 3,000-50,000 cP or 30-500 dPa.s (Fitriyanti *et al.*, 2019).

Homogeneity Test

Gel preparation *face wash* applied to a glass preparation, the preparati on is observed carefully and then felt to see whether the preparation is homogeneous, that is, there is no solid material or lumps felt on the glass and it is spread evenly (Hasanah, *et al.*, 2023).

Foam Height Test

Measuring the ability to form foam is by dissolving the gel *face wash* in water in a measuring cup and then shaken manually up to ten times. The foam height is measured, and the foam stability is measured by calculating the time the foam starts to disappear. The foam height test requirement is 12-220 mm (Rahmadinah *et al.*, 2023)



RESULTS AND DISCUSSION

The results of plant determination carried out by the UPT Herbal Materia Medica Laboratory, Batu City, with letter number 000.9.3/2080/102.2 024, show that cocoa fruit is a type of *Theobroma cacao* L. and Sterculiaceae family. The results of plant determination letters can be seen in attachment.

Cocoa fruit extraction (*Theobroma cacao* L.) carried out at the UPT Herbal Materia Medica Batu Laboratory, Batu City using the maceration method, because the manufacturing process and equipment used are simple and do not go through a heating process, namely by soaking the herbal medicine for a specified time to allow the solvent to draw out the components of the substances in the extract (Kusuma *et al.*, 2022).

The extract result obtained was 126 grams, so the value obtained was The yield of the extraction result (maceration) was 6.3% w/w. The yield value of the cocoa fruit extract was below 10% w/w, meaning that the yield was not good, this was because the duration of the maceration was short, namely 24 hours. In previous studies, the yield of cocoa fruit extract was also obtained as much as 7.05% (less than 10%) (Widyasanti *et al.*, 2020). Thus, it is necessary to increase the duration of extraction using maceration.

Gel Preparation Evaluation Results Face Wash

Organoleptic Test

Organoleptic tests were carried out after obtaining the thick extract and gel formulation. *face wash* cocoa fruit extract (*Theobroma cacao* L.) with extract concentration 2.5%, 5%, 7.5%. The following organoleptic test results can be seen in the table below.

Organoleptic tests include the color, odor, and consistency of the preparation produced from the gel preparation formulation. *face wash* cocoa fruit extract. The organoleptic test requirements for gel preparations are that the color of the preparation must match the extract/active substance, the strong smell of chocolate, and the consistency of the preparation must be thick. Gel test results *face wash* cocoa fruit extract (*Theobroma cacao* L.) shows that the gel preparation with concentrations of 2.5%, 5%, and 7.5% has a thick form while the smell has a distinctive chocolate smell and a color that follows the cocoa fruit extract (*Theobroma cacao* L.). Based on the results obtained from the differences in the concentration of active substances in each formula, the higher the concentration of extract used, the better the gel preparation. *face wash* the darker/thicker the color of the preparation will be.

Spread Power Test

The spreadability test was carried out to determine the speed of gel spread. *face wash* when applied to the skin. The following are the results of the spreadability test conducted with extract concentrations of 2.5%, 5%, 7.5% can be seen in the table below.

The range of the gel preparation spreadability test is 5-7 (Berliana *et al.*, 2020). Based on the results of the spreadability test, it can be seen that F2 is the best formula compared to F1 and F3. This is because the concentration of extract and additional ingredients in F2 interact better, making it easier to spread. High viscosity will reduce the gel's ability to spread because the components or particles in the gel are more difficult to move.



In addition, it can also be caused by *gelling agent* or gel-forming agents that can increase gel strength thereby reducing the spreadability test.

Homogeneity Test Results

Homogeneity test is carried out to determine the gel preparation formulation *face wash* mixed evenly or homogeneously. The following are the results of the homogeneity test with concentrations of 2.5%, 5%, 7.5%. In the homogeneity test to determine the gel preparation *face wash* has been dispersed evenly or not. The results of the test carried out for the homogeneity test on formulations 1-3 meet all requirements, which means that the gel preparation is dispersed evenly. The test parameter is if the gel preparation does not contain coarse granules or powder.

pH Test Results

The pH test was carried out because the gel preparation *face wash* is a topical preparation applied to the face. The following are the results of the pH test with different extract concentrations of 2.5%, 5%, 7.5% can be seen in the table below.

pH test to determine the acidity and alkalinity of a preparation. If the pH is too high or too low it can cause skin irritation and also cause dry skin. The pH test is carried out to determine whether it is in accordance with the pH of facial skin with a pH range between 4 - 6 and so that the gel preparation *face wash* safe to apply on the face (Purwaningtyas, *et al.*, 2020). The results of the pH test carried out showed that pH F1 and F2 did not meet the pH test requirements, which means that the gel preparation *face wash* in F1 and F2 are not safe to use, while in F3 it meets the requirements of pH testing and of course the preparation is safe to use. This is because the cocoa fruit extract (*Theobroma cacao* L.) has acidic properties, namely pH 5. The acidic properties of the extract, especially as the concentration increases, the amount of acidic compounds released into the solution also increases, thereby lowering the pH of the preparation.

Foam Height Test Results

Foam height test is one way to determine the surfactant in the preparation has the ability to produce foam. The following results of foam height with different extract concentrations of 2.5%, 5%, 7.5% can be seen in the table below.

The foam height test was carried out to see the foam power produced by the gel *face wash* with a range of soap foam heights set by the Indonesian National Standard (SNI) namely 1.3 cm-22 cm (Septia *et al.*, 2020). From the results of the foam height test F1, F2, F3 meets the SNI requirements. It can be seen from the table above that the higher the concentration of extract in the preparation, the lower the foam height produced, this is because the cocoa fruit extract (*Theobroma cacao* L.) which is acidic while soap is basic. In acidic conditions, proteins and surfactants that play a role in the formation and stability of foam can be denatured or lose their effectiveness, thereby reducing the ability of the liquid to form and maintain foam. Therefore, extracts that have an acidic (low) pH tend to produce lower or less stable foam compared to neutral or basic pH liquids (Hidayah *et al.*, 2020).



Viscosity Test Results

Viscosity test is the main characteristic related to the ease of use of gel preparations. *face wash*. The following results of foam height with different extract concentrations of 2.5%, 5%, 7.5% can be seen in the table below.

Viscosity test using a viscometer *which* is used to see the viscosity of the gel preparation *face wash*. The viscosity range of a good gel preparation is 3000-40000 cps, the results of the gel preparation viscosity test *face wash* cocoa fruit extract (*Theobroma cacao* L.) has met the requirements according to the viscosity range of the gel preparation. The difference in viscosity values in the table above, namely formula 1 (2.5%), formula 2 (5%), formula 3 (7.5%) shows that the higher the concentration of extract given to the gel preparation, the higher the viscosity value of the gel preparation *face wash*.

Results of Determination of Total Flavonoid Content of Gel Face Wash

Based on the results of the evaluation of the preparation, it shows that F3 is the best formula based on the use of the preparation which is safe to use as seen from the pH test parameters. So that in determining the total flavonoid content of the gel *face wash* The researcher chose F3 because he saw the results of the pH test related to the use of safe preparations, because if using F1 and F2 it is not safe to apply to the face because the results of the pH test carried out do not enter the pH range of the facial skin. Thus, the researcher chose F3 for determining the levels of flavonoid gel *face wash* cocoa fruit extract (*Theobroma cacao* L.)

From the results of testing the total flavonoid content of the gel *face wash* obtained a flavonoid content value of 0.0005 mg QE/g. In previous research, the determination of flavonoid content of cocoa fruit extract obtained a value of 0.0597 mg/L [14]. It can be seen that the flavonoids contained in the gel preparation *face wash* is classified as low because the sample tested was not pure cocoa fruit extract but a gel preparation *face wash* cocoa fruit extract. There are factors that influence the difference in flavonoid results in previous studies, namely samples for flavonoid tests, in previous studies using pure cocoa extract (*Theobroma cacao* L.), while in this study we used gel samples *face wash* cocoa extract (*Theobroma cacao* L.) which is not pure cocoa extract (Kusuma Dewi *et al.*, 2020)

Factors that influence the content of secondary metabolites in plants are environmental factors such as temperature, light intensity, amount of water, and CO₂.

CONCLUSION

Testing the physical properties of gel preparations *face wash* cocoa fruit extract (*Theobroma cacao* L.) meets the requirements and gel preparations *face wash* in formula 3 cocoa fruit extract (*Theobroma cacao* L.) contains total flavonoid levels of 0.0005/mg/QE/g.

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TABLE

Table1. Formulation Gel Face Wash

Material	Function	F1 (%)	F2 (%)	F3 (%)
Cocoa Fruit Extra	Active Substance	2,5	5	7,5
Gliserin	Humektan	15	15	15
Na-Lauril Sulfat	<i>Foaming agent</i>	2	2	2
Metil Paraben	Preservative	0,18	0,18	0,18
Carbopol 940	<i>Gelling agent</i>	1	1	1
TEA	Emulsifier	2	2	2
Aquadest ad	Solvent	ad 100 mL	ad 100mL	ad 100 mL

Table 2. % Yield

Total Cocoa Fruit Simplex	Thick Ekstrakt	% Yield
2.000 gram	126 gram	6,3%

Table 3. Spreadability Test Results

Formula	Spreadability Test Results
F1 (2,5%)	5,7 cm $\pm$ 0,05
F2 (5%)	5,8 cm $\pm$ 0,06
F3 (7,5%)	5,3 cm $\pm$ 0,08

Table 4. pH Test Result

Formula	pH Test Result
F1 (2,5%)	7,42 $\pm$ 0,08
F2 (5%)	6,23 $\pm$ 0,09
F3 (7%)	5,9 $\pm$ 0,05



Table 5. Foam Height Test Results

Formula	Tall (cm)
F1 (2,5%)	10 cm $\pm$ 0,1
F2 (5%)	9 cm $\pm$ 0,1
F3 (7,5%)	8 cm $\pm$ 0,2

Table 6. Viscosity Test Results

Formula	Viscosity
F1 (2,5%)	15400 cps $\pm$ 100
F2 (5%)	15800 cps $\pm$ 100
F3 (7,5%)	16800 cps $\pm$ 200

Table 7. Results of Flavonoid Level Determination

Sample	Absorbance	Concentration	Flavonoid levels
F3	0.083	7,566	0.0005 mg QE/g
	0.082	7,473	
	0.083	7,566	
Average $\pm$	0.083 $\pm$	7,535 $\pm$	
SD	0.0057	0.0046	

