

COMPARISON OF FORMULATIONS OF CREAM SCRUB COFFEE (*Coffea canephora* P.) WITH ANIONIC AND NONIONIC EMULATORS ON PHYSICAL CHARACTERISTICS

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Abstract. One of the plants that can be used as an active ingredient in cosmetics is coffee. Coffee, which is a natural ingredient that is generally consumed, turns out to have a myriad of benefits for the face. The ingredients are very good for facial care, mixing an ointment base with coffee grounds with some natural ingredients can produce good results. Coffee contains hydroxycinnamic acid which has antioxidants so it can be used as cosmetic skin care. Based on the above background, research was conducted on the formulation of coffee scrub preparations with anionic and nonionic emulsifier variations and then tested the physical characteristics of the scrub preparations. This study aims to determine the effect of an anionic emulsifier on the physical characteristics of coffee cream scrub preparations, the physical characteristics of coffee cream scrub preparations, and which emulsifier can produce coffee scrub preparations with good physical characteristics. This study uses experimental research, namely by making cream preparations with new formulations. Coffee is the independent variable which is then subjected to organoleptic tests, creaming, phase inversion, pH tests, adhesion tests, homogeneity tests, and spreadability tests. The results showed that the anionic emulsifier affected the physical characteristics of the coffee cream scrub preparation, namely, there was no phase inversion, the pH tended to be more alkaline, the adhesion of the cream with anionic emulsifier was lower and the spreadability was higher compared to the nonionic emulsifier. The results of the physical characteristics of the coffee cream scrub preparation were phase inversion, the pH tended to be acidic, the adhesive power of the cream with nonionic emulsifier was higher and the spreading power was lower compared to the anionic emulsifier and anionic emulsifier resulting in coffee cream scrub with better physical characteristics than cream with emulsifier nonionic.

Keywords: [Coffee, cosmetic skin care, cream scrub, emulsifier nonionic]

INTRODUCTION

In the past, people used medicinal plants and spices as traditional cosmetics, but over time, the development of medicinal and cosmetic products was influenced by synthetic ingredients. In recent decades many manufacturers have preferred ingredients from natural sources, so various countries have researched exotic plants, fruits, and flowers to find new cosmetic ingredients (Thornfeldt & Bourne, 2010). One of the plants that can be used as an active ingredient in cosmetics is coffee.

Coffee, which is a natural ingredient that is generally consumed, turns out to have a myriad of benefits for the face. The ingredients are very good for facial care, mixing an ointment base with coffee powder (*Coffea canephora* P.) with some natural ingredients can produce good results. Coffee contains hydroxycinnamic acid which has antioxidants so it can be used as a skincare cosmetic (Duangjai et al., 2016). Meanwhile, according to (Harahap, 2017), coffee contains secondary metabolites in the form of caffeine, chlorogenic acid, alkaloids, flavonoids, tannins, and saponins as antioxidants which are good for preventing and treating various kinds of skin problems, including dull skin. Interestingly again,

Based on research on coffee, caffeine, and chlorogenic acid, antioxidant activity has been tested using the DPPH method. The EC50 value (efficiency concentration) produced by caffeine is 21.41 ppm while chlorogenic acid is 5.86 ppm. The EC50 value is a parameter used to indicate antioxidant activity that provides 50% inhibition. Substances that have high antioxidant activity will have a low EC50 value. The EC50 value of chlorogenic acid is lower than caffeine, so it can be concluded that chlorogenic acid has higher antioxidant activity than caffeine. This is because chlorogenic acid has many hydroxyl groups which affect antioxidant activity (Sukohar et al., 2011).

Skin care cosmetics consist of cosmetics for cleaning the skin (cleansing cream, soap, cleansing milk, and skin fresheners). Cosmetics to moisturize the skin/moisturizer (night cream), protective cosmetics (sunscreen cream, sunblock cream/lotion). Cosmetics for skin thinning/peeling. (Tranggono & Latifah, 2007).

One example of cosmetic skin care that functions to thin the skin/peeling is scrub. Scrub is one example that is used for skin care. Scrub is a traditional preparation used to remove dead skin cells, dirt,

and open pores so that air exchange is free, making the skin brighter. Scrubs are divided into several dosage forms, namely powder scrubs, cream scrubs, or whipped/liquid scrubs (Tranggono & Latifah, 2007).

Body scrub in the form of cream requires an emulsifier as an emulsion stabilizer. Synthetic emulsifiers can be grouped into three, namely anionic emulsifiers which are good O/W emulsifiers, cationic emulsifiers which are included in normal pH, and nonionic emulsifiers which are not affected by pH and the addition of electrolytes (Sari, 2012). Each emulsifier has different physical and chemical properties. In cream preparations, the selection of the appropriate emulsifier must be considered to obtain preparations that have good stability and effectiveness. The stability of the cream is determined by the ability of the emulsifier to be at the oil-water interface and lower the interfacial tension. The emulsifier must be able to form a cream that is physically and aesthetically stable, but not strong enough to hold the active ingredient in the preparation.

Based on the above background, research was conducted on the formulation of coffee scrub preparations with anionic and nonionic emulsifier variations and then tested the physical characteristics of the scrub preparations.

METHODS

Tools & Materials

The tools used are stirring sticks, porcelain dishes, 25 ml pyrex measuring cups, glass objects, mortar and stamfer, horn spoons, bowls, pH indicator paper, dropping pipettes, spatulas, analytical balances/balances, thermometers, 44 mesh sieve, water bath, cream containers, stickiness test kits, spreadability test kits.

The materials used are coffee grounds, stearic acid (bratachem), cetyl alcohol (bratachem), propylene glycol (bratachem), anhydrous lanolin (bratchem), triethanolamine (bratachem), tween60 (brathem), span60 (bratachem), methylparaben (bratachem), propylparaben (bratachem), rose oil (bratachem), distilled water.

Plant determination

Determination of coffee plants (*Coffea canephora* P.) aims to avoid errors in the plants used and determine the morphological characteristics of the plants to be studied. Thus errors in the collection of materials to be used for research can be avoided. Coffee grounds used for research were determined at the Biology Laboratory, Faculty of Mathematics and Natural Sciences, Diponegoro University, Semarang. Based on the results of the determination, it can be ascertained that the plants used in this study were the true coffee tree samples from (*Coffea canephora* P.).

Cream preparation

The coffee cream scrub formulation comes from Diah's formula (2017).

Table 1. Formula design for coffee cream scrub (*Coffea canephora* P)

Material	Material concentration (gr/mL)					
	F1	F2	F3	F4	F5	F6
Coffee grounds	10	10	10	10	10	10
Stearic acid	3	3	3	3	3	3
Cetyl alcohol	1.5	1.5	1.5	1.5	1.5	1.5
Propylene glycol	5	5	5	5	5	5
Lanolin anhydrous	1	1	1	1	1	1
Triethanolamine	0.5	1	1.5	-	-	-
Tweens 60	-	-	-	1,221	1.325	1.5
Span 60	-	-	-	0.2795	0.1325	0
Methyl paraben	0.1	0.1	0.1	0.1	0.1	0.1
Propyl paraben	0.01	0.01	0.01	0.01	0.01	0.01
Rose oil	0.05	0.05	0.05	0.05	0.05	0.05
Aquadest ad	50	50	50	50	50	50

Source: Diah, (2017)

Making coffee cream with an anionic emulsifier is done by weighing each ingredient according to the calculation, then the oil phase consisting of stearic acid, cetyl alcohol, anhydrous lanolin, propyl paraben is melted while the water phase consisting of TEA, nipagin, propylene glycol, aquadest heated at 70°C. Emulsions are made by adding the oil phase to the water phase while stirring until a homogeneous emulsion is formed. The coffee grounds are put into the mortar then add the rose oil stir until homogeneous, add the cream base little by little at 45°C - 55°C, and stir until homogeneous then add the remaining cream base and stir. The cream is packaged in a container protected from sunlight (Diah, 2017).

Making coffee cream with a nonionic emulsifier is carried out by weighing each ingredient according to the calculation, then the oil phase consisting of stearic acid, cetyl alcohol, anhydrous lanolin, propylparaben, Span 60 is melted while the water phase consisting of Tween 60, nipagin, propylene glycol, distilled water heated at 70°C. Emulsions are made by adding the oil phase to the water phase while stirring until a homogeneous emulsion is formed. The extract was crushed in a mortar, then rose oil was added, and stirred until homogeneous, then added the cream base little by little at 45-55°C and stirred until homogeneous, then added the remaining cream base and stirred. The cream is packaged in a container protected from sunlight (Diah, 2017).

Test the Physical Properties of Preparations

1. Organoleptic test

The organoleptic examination includes shape, color, and smell which are observed visually. The specifications of the cream that must be met are having a soft consistency without coarse grains, a homogeneous color of the preparation, a fragrant smell, and no phase separation (Pramuditha, 2016).

2. Creaming test

The measurement of creaming volume was carried out by taking 10 ml of cream scrub, putting it in a measuring cup, and then observing the creaming volume. Calculated in % with the formula:

$$\text{Volume creaming} = \text{Hu}/\text{Ho} \times 100\%$$

(Pramuditha, 2016).

Where: Hu = the volume of the creaming emulsion
Ho = total volume of creaming

Creaming test of coffee cream scrub aims to determine the occurrence of layers with different concentrations in the emulsion (Sirait, 2018). The results of observing the creaming volume using an anionic emulsifier did not show any creaming in the coffee cream scrub. Whereas preparations with nonionic emulsifiers experienced creaming, namely 10%. This is due to the influence of the gravitational factor, and the determination of HLB which must be by the concentration, so coffee cream scrub with nonionic emulsifier experiences creaming or separation. In line with Nonci's research, Tahar & Aini (2016) stated that creams with anionic emulsifiers are more stable, with no creaming even after accelerated storage conditions.

3. Phase Inversion

Determination of the type of preparation emulsion is done in two ways, namely dilution with water and painting or coloring. Dilution with water is done by diluting 100 mg of cream scrub with 10 mL of water, if the emulsion is easily diluted with water, then the emulsion is of the w/a type (Sirait, 2018)

Staining or coloring is done by adding 1 drop of methylene blue solution to 500 mg of the preparation on a glass object. Cover with cover glass. If methylene blue is spread evenly, it means that the preparation is an oil-in-water emulsion type, but only blue spots show that the preparation is a water-in-oil emulsion type (Sirait, 2018).

4. pH test

The pH of the preparation was measured using indicator paper. Dip the indicator paper with the colored part into the sample, then after that, you will see a color change, this color change is compared to the color on the pH stick box. So in the box match which color is the most suitable and suitable.

5. Stickiness test

The ointment that has been weighed at 0.5 g is placed on an object glass whose area has been determined, then another object glass is placed on top of the ointment and pressed with a 1 kg load for 5 minutes, then the object glass is placed on the adhesive test equipment. The 80 gram weight of the ointment was removed, and the time was recorded until the two glass objects were released. The requirement for good adhesion time for topical preparations is less than 4 seconds (Mukhlisah, Sugihartini & Yuwono, 2016).

6. Homogeneity test

Homogeneity testing is carried out by applying 0.1 gram of ointment on the surface of the object glass, the ointment preparation is said to be homogeneous if there are no coarse grains on the object glass (Mukhlisah, Sugihartini & Yuwono, 2016).

7. Spread Power

A total of 0.5 g of cream was weighed and placed in the middle of the glass apparatus, and the cover glass which had been weighed first and then placed on the base, was left for 1 minute. The diameter of the spread of the cream is measured after one minute by taking the average length of the diameter from several sides, the weight is added to the weight of 50g then the measurement is carried out again after one minute, and the weight is added every 50g until the weight is added to 150g until it doesn't move at all, the diameter is recorded distribution (Mukhlisah, Sugihartini & Yuwono, 2016).

Data analysis

The data obtained is in the form of descriptive and quantitative data. Descriptive data were obtained from the observation of organoleptic homogeneity, pH test, and cream-type test. Quantitative data were obtained from the spreadability test and the adhesion test. The analysis will be carried out by comparing the results with related literature and by means of a statistical approach, namely by using the SPSS program. Data were analyzed with normal distribution and homogeneity. If the data is normally distributed and homogeneous then proceed with One Way Anova analysis at the 95% confidence level then proceed with the Tukey test. If the data is not normally distributed and not homogeneous, then continue with the Kruskal Wallis test and the Man Whitney advanced test.

RESULTS AND DISCUSSION

Determination Result

The coffee plant used in the study was obtained from the holy city, and the determination was carried out at the Ecology and Biosystematic Laboratory of the Department of Biology, Faculty of Science and Mathematics, Diponegoro University. The purpose of the determination is to ensure the correctness of the plant used. Based on the results of the determination, it can be seen that the plant used belongs to the Rubiaceae family with the species name *Coffea canephora* Pierre ex Froehner.

Organoleptic test results

Organoleptic test of coffee cream scrub was carried out visually including shape, color, smell, and consistency. This test aims to determine that the physical characteristics of the coffee cream scrub match the expected criteria. The organoleptic test results for coffee cream scrubs can be seen in table 2.

Table 2. Organoleptic test results for coffee cream scrub (*Coffea canephora* P.)

Formulas	Form	Color	Smell	Consistency
F1				Thick
F2				Less viscous
F3	Cream	Chocolate	Typical coffee	Less viscous
F4				Thick
F5				Thick
F6				Thick

Based on organoleptic observations coffee cream scrub is brown, has coarse cream granules, and has a distinctive coffee smell. Then on F2 and F3, the consistency is less thick because the ratio of the higher emulsifier concentration results in a higher cream consistency

Creaming Test

The results of the creaming test can be seen in table 3.

Table 3. Creaming test results for coffee cream scrub (*Coffea canephora* P.)

Formulas	Average % creaming volume
F1	0
F2	0
F3	0
F4	10
F5	10
F6	10

Inversion Phase

Phase inversion testing of coffee scrub aims to determine the type of cream, namely O/AW or W/O type. Cream-type testing uses two methods, namely the dilution method and the coloring method. The dilution method is carried out by diluting the cream with a certain amount of water, then observing whether dilution occurs or not. If the cream does not experience dilution, it is a W/O type cream, but if the cream experiences dilution, it is an O/W type cream (Pramuditha, 2016).

The coloring method is done by coloring the cream using methylene blue then stirring and observing, if the cream is fully colored then it is an O/W type cream. Conversely, if the cream is not completely colored, then the cream is W/O cream.

The results of the cream type test were O/W cream which can be seen from the results of the dilution test, that all coffee creams scrub can experience dilution or can be diluted after being given water. The results of the cream type test using staining were then stirred and observed, all coffee creams scrub had the O/W type which could be seen in an even blue color. These results are consistent with the initial formulation objective of formulating an oil-in-water (O/W) type cream. This is because the amount of dispersed phase used in the cream is smaller than the dispersing phase (water phase). The results of the cream-type test can be seen in table 4.

Table 4. Results of phase inversion test of coffee cream scrub (*Coffea canephora* P.)

Formulas	Dilution with water	Dilution with methylene blue
F1	Diluted	Spread evenly
F2		
F3		
F4		
F5		
F6		

pH Test Results

The pH test aims to determine whether the cream preparation is acidic or alkaline. Cream preparations should not be too sour and too alkaline. A good cream is a cream that has a pH that corresponds to the physiological pH of the skin 4.5-6.5 (Kalangi, 2013). If the pH of the cream scrub is too acidic it will irritate the skin and if it is too alkaline it will cause scaly skin. From the test results it can be seen that coffee cream scrub has a pH that is by the physiological pH of the skin, cream preparations with anionic emulsifiers tend to be less acidic and stable. Anionic emulsifier (TEA) is used as an emulsifier as well as an alkalizing agent so that the pH of the preparation is more stable and not acidic. This is in line with the research of Sehro, Luliana & Desnita (2016), that the addition of TEA in the base lotion can affect the pH of the base and the stability of the base. Coffee powder cream scrub formula with a nonionic emulsifier has a pH that tends to be acidic. The results of the pH test on the

coffee cream scrub formulation can be seen in table 5.

Table 5. pH test results of coffee cream scrub (*Coffea canephora* P.)

Formulas	The average pH test results	SNI	Information
F1	6.0±0.664	4.5 – 6.5	Fulfill
F2	6.0±0.664	4.5 – 6.5	
F3	6.5±0.664	4.5 – 6.5	
F4	5.0±0.664	4.5 – 6.5	
F5	5.0±0.664	4.5 – 6.5	
F6	5.0±0.664	4.5 – 6.5	

Stickiness Test Results

The results showed that anionic emulsifiers had low adhesion and did not meet the requirements for topical preparations, this was because anionic emulsifiers had low viscosity. Anionic emulsifier is a surfactant that has a negative charge on its hydrophilic part (Voight, 1995), and has properties that are compatible with a cationic compound, namely a positively charged compound found in coffee grounds, namely flavonoid compounds. So that the base and the coffee grounds react to attract each other which causes the base to become less viscous due to the reduced emulsifier potential.

In formulas with nonionic emulsifiers, namely F4, F5, and F6, the viscosity is higher because the nonionic emulsifier is an emulsifier that has no charge or is neutral (Voight, 1995), this emulsifier is compatible with cationic and anionic compounds contained in coffee grounds. So that the cream base has a thick consistency which affects the stickiness of the cream to be longer. The results of the adhesiveness test of the coffee cream scrub can be seen in table 6.

Table 6. The results of the adhesiveness test of coffee cream scrub (*Coffea canephora* P.)

Formulas	Average adhesion test results (seconds)	SNI	Information
F1	1.31 ± 0.017	Not less than 4 seconds	Does not meet the
F2	1.29 ± 0.061		
F3	1.14 ± 0.045		
F4	1.81 ± 0.106		
F5	1.88 ± 0.015		
F6	1.93 ± 0.047		

From the results of the adhesion test then analyzed with the normality test, this test was carried out using the Shapiro-Wilk test. The results of the analyzed data show that the data is not normally distributed which can be seen in the appendix. These results are indicated by a significance value of $P < 0.05$ which indicates that the data is not normally distributed.

After the data is not normally distributed, it is then followed by a variant homogeneity test using Levene Statistics. Based on the data analyzed by the homogeneity test, it shows that the variance of the data obtained is homogeneous. The results of the homogeneity test can be seen in the appendix. These results are indicated by a significance value of $P > 0.05$ which indicates that the data is homogeneous.

After the data is not normally distributed and homogeneous, it is then followed by the Kruskal-Wallis test which is used to see whether or not there is a significant difference between the formulas. The results of the Kruskal Wallis test can be seen in Appendix No. 2. These results show a significance value of $P < 0.05$ which indicates a significant difference between groups.

Then proceed with the Mann-Whitney test which is used to determine the differences in each formula. It is known that between F1&F2, F1&F3, F2&F3, $P > 0.05$ means that it is not significant where there is no difference between the formulas. This is because F1, F2, and F3 have the same emulsifier, namely anionic emulsifier. Based on these data, F3 has the lowest adhesive power compared to F1 and F2, although statistically there is no significant difference. This is because F3 has the highest concentration of anionic emulsifier which causes the F3 cream formula to be the most dilute and has the lowest stickiness. In F4 and F5, F5 and F6 it is known that $P > 0.05$ means it is not significant and there is no difference in each formula because in formula F4,

Based on the data it is known that F6 has the highest adhesive power compared to F4 and F5

although statistically there is no significant difference, this is because F6 has a nonionic emulsifier concentration with an HLB of 16. Between F1&F4, F1&F5, F1&F6, F2&F4, F2&F5, F2&F6, F3&F4, F3&F5, F3&F6 (between cream formulas with anionic emulsifiers and nonionic emulsifiers) were found to be $P < 0.05$, meaning that there was a significant difference in each formula. This is because F1, F2, and F3 which contain anionic emulsifiers will produce a runny cream with low adhesion. While on F4, F5.

Homogeneity Test

The homogeneity test of coffee cream scrub aims to determine whether the cream scrub has been distributed homogeneously in the preparation or not. The homogeneity test is important because homogeneity affects the therapeutic effect of the preparation of the material. Cream preparations that are not homogeneous can cause medicinal properties to not be achieved (Yumas, 2016). Coffee cream scrub in all formulas produces cream preparations that contain little fine granules due to the presence of coffee grounds as an active substance. However, the slightly textured cream preparations in the cream scrub formula will make it easier to remove dirt from the skin by scrubbing.

Spreadability Test

In the spreadability test, it is known that cream with an anionic emulsifier has greater spreading power than cream with a nonionic emulsifier. Cream with anionic emulsifier spreadability increased with increasing concentration of anionic emulsifier sequentially from the lowest spreadability at F1, F2, and F3. Whereas the cream with nonionic emulsifier at F4, F5, and F6 has a relatively stable spreading power. The average spreading power of coffee cream scrub does not meet the requirements for topical preparations, the cream is classified as a preparation that is difficult to spread because its application as a scrub requires pressure when used to remove dirt from the skin.

The results of the spreading power test were then analyzed with the normality test. This test was carried out using the Shapiro-Wilk test, the results of the data analyzed showed that the data was not normally distributed which can be seen in the appendix. These results are indicated by a significance value of $P < 0.05$ which indicates that the data is not normally distributed.

After the data is not normally distributed, it is then followed by a variant homogeneity test using Levene Statistics. Based on the data analyzed by the homogeneity test, it shows that the variance of the data obtained is homogeneous. The results of the homogeneity test can be seen in the appendix. These results are indicated by a significance value of $P > 0.05$ which indicates that the data is homogeneous.

After the data were not normally distributed and homogeneous, then the Kruskal Wallis test was used to see whether or not there were significant differences between the formulas. The results of the Kruskal Wallis test can be seen in Appendix No. 3. These results show a significance value of $P < 0.05$ which indicates a significant difference between groups.

Then proceed with the Man Whitney test which is used to determine the differences in each formula. It is known that between F1&F2, F1&F3, and F2&F3 $P > 0.05$ means it is not significant where there is no difference between the formulas. This is because F1, F2, and F3 have the same emulsifier, namely anionic emulsifier based on spreading power. It is known that F3 has the highest spreading power compared to F1 and F2 although statistically there is no significant difference, this is because F3 has a concentration of emulsifier the highest which causes F3 to be the most dilute and has a wider spreadability. Between F4&F5, F4&F6, and F5&F6 it is known that $P > 0.05$ means it is not significant, there is no difference in each formula.

Based on the spreadability test data it is known that F4, F5, and F6 tend to be stable, between F1&F4, F1&F5, F1&F6, F2&F4, F2&F5, F2&F6, F3&F4, F3&F6 (between cream formulas with anionic and nonionic emulsifiers) $P < 0.05$ means there is a significant difference significant in each formula. This is because anionic emulsifiers can produce runny cream and have higher spreadability, while F4, F5, and F6 which contain nonionic emulsifiers produce thicker cream and lower spreadability compared to anionic emulsifiers, this is to Diah's research (2017).

CONCLUSION

In this study, the following conclusions can be obtained:

1. The anionic emulsifier affects the physical characteristics of the coffee cream scrub preparation (*Coffea canephora* P.), namely there is no phase inversion, the pH tends to be more alkaline, the adhesion of the cream with anionic emulsifier is lower and the spreadability is higher compared to the nonionic emulsifier.
2. The nonionic emulsifier affects the physical characteristics of the coffee cream scrub preparation (*Coffea canephora* P.) The cream has a phase inversion, the pH tends to be acidic, the adhesive power of the cream with the nonionic emulsifier is higher and the spreadability is lower compared to the anionic emulsifier.
3. Anionic emulsifier produces coffee cream scrub (*Coffea canephora* P.) with better physical characteristics than cream with a nonionic emulsifier.

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