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Formulation and Evaluation of Grape Seed Oil (Vitis Vinifera, L) Facial Cream with Variations in The Concentration of Stearic Acid as an Emulsifier

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ABSTRACT

Ageing is a complex biological process resulting from intrinsic ageing and extrinsic ageing. The most influential factor in ageing is free radicals because they can cause oxidative stress, which plays an essential role in the ageing process. Oligomeric proanthocyanidins (OPC) in grapeseed oil have the highest antioxidant effect against free radicals. These antioxidants are used in cosmetic products to maximize the treatment of ageing. This study aims to optimize the formula for grape seed oil cream by varying the concentration of stearic acid as an emulsifier. Grape seed oil is formulated into cream preparations which are divided into three formulas, namely formula one (F1), which uses 10% stearic acid concentration, formula two (F2) 12% stearic acid concentration, and formula three (F3) 14% stearic acid concentration. Determination of optimum stability and formula is obtained from the evaluation of cream preparations, including organoleptic, physical homogeneity, pH, dispersion, viscosity, and emulsion type tests. The results showed that the concentration of stearic acid emulsifiers affected the spreadability and viscosity. And does not affect the pH, homogeneity, and type of emulsion.

Keywords
Ageing
Facial cream
Emulsifier
Stearic Acid

Introduction

Ageing is a complex biological process due to intrinsic factors (from within the body, such as genetics) and extrinsic factors (from the environment). Extraneous factors that play the most role in ageing are free radicals. Free radicals can majorly impact the ageing process because they can cause oxidative stress [1]. Psychosocial effects have created a demand to fight ageing on the skin, one of which is anti-ageing cosmeceutical products. Anti-ageing products used to fight age caused by free radicals contain antioxidants as their active ingredients [2]. Antioxidants from grapes (Vitis vinifera, L) are considered a good source of antioxidants. It mainly due to oligomeric proanthocyanidins (OPC) in grapeseed oil.

Wine is a commodity that provides added value. It can be consumed as fresh fruit, grape juice, drinks (wine), raisins, and others [3]. Grapes contain various vitamins and minerals, such as calcium, magnesium, potassium, vitamins B1, B2, B3, B5, B6, and C and contain flavonoid compounds. The skin of grapes is rich in resveratrol, a substance that can delay the appearance of wrinkles on the skin. Research on the use of red grape juice (Vitis vinifera L.) in cream preparations showed that red grape juice with a concentration of 3% was able to reduce evaporation of water from the skin by 9.79% - 10.68 [4].

Grapeseed oil contains antioxidants that are beneficial in treating the skin. The antioxidants contained in it are vitamin E and oligomeric proanthocyanidins (OPC). This OPC serves to ward off free radicals that damage skin tissue. OPC can repair collagen damaged by free radicals, thus preventing wrinkles on the skin. The content of vitamin E in grape seed oil is also beneficial for the skin, where vitamin E helps moisturize the skin, improve skin elasticity and reduce the appearance of wrinkles [5].

The presence of antioxidant activity in grape seed oil can be used to properly utilize grape seed waste and potentially make cosmetics for skin care, for example, on facial skin. Currently, the use of cosmetics is increasing, especially with antioxidants that prevent premature ageing and neutralize free radicals on the skin [6]. Based on its ability as an antioxidant, grapeseed oil can be formulated in the form of cream preparations. Creams in the form of antioxidants protect the skin from environmental influences (sun exposure and pollution) by inhibiting skin damage and premature ageing [7].

Cream preparations are widely chosen as topical preparations because they are easy to use, formulate and function as good protectors, comfortable, and evenly distributed to the skin [8]. Many patients and doctors prefer creams over ointments because creams are easier to spread and clean [9]. The type of cream is divided into two: the type of cream in oil in water (W/A) and water in oil (W/W). Oil (W/M) is more accessible to wash with water and is not slippery when applied to the skin, such as on the face and is indicated for cosmetics [10]. Stearic acid has an essential role in cream formulations, namely as an emulsifier with a concentration of 1-20%. The use of stearic acid in oil-in-water (W/W) cream-type formulations [11].

Based on the above explanation, a cream formulation for the face with type M/A was made, which contains the active substance of grape seed oil as an antioxidant. This grape seed oil W/A cream formula uses stearic acid emulsifier concentration with several variations, and the cream formula is good and physically stable.

Methods

A. Context

The tools used in this study include analytical balance (Wiggen Hauser), measuring cup (Pyrex), stirring rod, beaker (pyrex), mortar, stemper, evaporating dish, spatula, and object glass, viscometer (Brookfield), pH indicator. The materials used in this study include Grapeseed oil, stearic acid, cetyl alcohol, stearyl alcohol, triethanolamine (TEA), methyl paraben, propyl paraben, glycerin and aquadest.

The type of research conducted is experimental research. Several steps were carried out. It included searching for grapeseed oil samples, formulation of grape seed oil cream, physical evaluation of the finished cream including organoleptic testing, testing pH, viscosity, homogeneity, spreadability and emulsion type. The manufacture of grapeseed oil is carried out by Lansida Herbal Technology, Yogyakarta, Central Java.

A. Making Procedure

Weigh all the ingredients that will be used in making the cream. Enter the components included in the oil phase (grapeseed oil, stearic acid, cetyl alcohol, stearyl alcohol) into a cup and then heat in a water bath, stir until homogeneous with stirring until it reaches a temperature of 70-75°. Then enter the propyl paraben and methyl paraben into the oil phase and stir until homogeneous. The solubility of propyl paraben and methyl paraben is very slightly soluble in water, in 3.5 parts of ethanol (95%), in 3 parts of acetone, and 140 parts of glycerol, and 40 parts of fatty oils, easily soluble in alkaline hydroxide solutions. Melting Temperature 95° to 98° [12].

Mix the aqueous phase (glycerin, triethanolamine and hot aquadest) with heating until the temperature reaches 70-75°. Then mix the two phases, namely the oil phase and the water phase, into a hot mortar. Stir until a good cream preparation is formed (see Table 1).

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Table 1. Grapeseed Oil Cream Formulation

Ingredient	Concentration (% weight per cent)		
	FI	F2	F3
Grapeseed oil	5	5	5
Stearic acid	10	12	14
Cetyl alcohol	1	1	1
Stearyl alcohol	1	1	1
TEA	2	2	2
Glycerin	10	10	10
Nipagin	0.1	0.1	0.1
Nipassol	0.05	0.05	0.05
Aquadest up to	100	100	100

Description: F1 = Formula 1; F2 = Formula 2; F3 = Formula 3
(Source: Yadav, NP, et al, 2014; Setiawati, 2014 with modifications)

B. Preparation Evaluation

Physical evaluation test of the preparation was carried out for 28 days on days 0, 7, 14, 21 and 28. It included organoleptic observation, viscosity testing, pH measurement, homogeneity testing, spreading power testing and cream type testing. Organoleptic observations of cream preparations include observations of the colour, texture, and odour of cream preparations [13]. Measurement of pH using a pH indicator. Weighed as much as 0.5 g and dissolved in 50 mL of distilled water, then measured the pH. The tolerance for cream pH ranges from 4.0 to 8 (SNI 16-4399-1996). Viscosity measurements were carried out using a viscometer (Brookfield). The preparation is stored in a 100 ml glass beaker as much as 100 grams, then attached to a spindle size four, and the rotor was run at a speed of 100 rpm; after showing a stable number, read and record the results [11]. Homogeneity testing is done by taking 1 gram of cream on the top, middle and bottom, then applying it to a piece of transparent glass (object glass). Observe if there are coarse grains visually [12].

Cream weighing 1 gram was placed in the middle of a round glass scale. On top of the cream is placed another round glass and ballast. So, the round glass and ballast weight is 150 grams, left for 1 minute. Then the diameter of the distribution was recorded [12]. The dispersion requirement for topical preparations is about 5-7 cm [14]. Good dispersion causes the contact between the drug and the skin to be broad so that the absorption of the drug into the skin takes place quickly.

The type of emulsion preparation was determined by placing a certain amount of preparation on an object glass, adding one drop of methyl blue, and stirring with a stirring rod. If the methyl blue is evenly distributed, the preparation has an o/w emulsion type, but if only blue spots are present, the preparation has a w/o emulsion type [15].

C. Statistic analysis

Analysis of research data using SPSS 21.0. From the results of the research on pH, dispersibility and viscosity, analysis was carried out using the Shapiro-Wilk test to state that the data were normally distributed. If the data is not normally distributed, statistical analysis using the Kruskal-Wallis test is used to determine the effect of variations in the concentration of stearic acid on grape seed oil cream preparations. Meanwhile, the organoleptic test results' data, homogeneity and emulsion type were analyzed descriptively.

Results

The manufacture of grapeseed oil is carried out by Lansida Herbal Technology, Yogyakarta, Central Java. The cream was made using grapeseed oil as the active ingredient and several other ingredients according to a predetermined formula. The prepared cream was evaluated by organoleptic, pH, spreadability, homogeneity, viscosity, and emulsion type tests.

The physical evaluation of cream preparations aims to determine the effect of variations in stearic acid concentration on the physical properties of the preparation. It includes cream organoleptic, pH, dispersibility, homogeneity, viscosity, and cream type tests.

The organoleptic examination includes the shape, colour and odour of the cream. The results of the organoleptic test can be seen in Table 2.

Table 2. Organoleptic observations

Days to-	F1	F2	F3
0	White No smell Gentle	White No smell Gentle	White No smell Gentle
7	White No smell Gentle	White No smell Gentle	White No smell Gentle
14	White No smell Gentle	White No smell thick-solid	White No smell thick-solid
21	White No smell Gentle	White No smell thick-solid	White No smell thick-solid
28	White No smell Gentle	White No smell thick-solid	White No smell thick-solid

Description: F1 (Formula 1), F2 (Formula 2), F3 (Formula 3).

The purpose of testing the pH of this cream preparation is to determine whether the cream has met the pH requirements for topical preparations, which are between 4.5-7.5. pH preparations that are too acidic can irritate the skin, while if the pH value is too alkaline, it can make the skin dry and scaly [16]. The pH test is carried out using pH indicator paper. The results of the pH measurement test can be seen in Table 3.

Table 3. PH. Measurement Results

PH	Days to				
	0	7	14	21	28
F1	7	6	6	6	6
F2	7	6	6	6	6
F3	7	6	6	6	6

Description: F1(Formula 1), F2(Formula 2), F3(Formula 3).

The homogeneity test determines the mixture of cream preparation ingredients [17]. In this evaluation, the homogeneity test of the cream was carried out visually by observing the colour of the cream and the presence or absence of parts that were not well mixed. The results showed that the cream preparation with stearic acid emulsifier met the homogeneity requirement. That is, no coarse particles were seen. The condition for the preparation of the cream is that if it is applied to a piece of glass, no separation between the components make up the emulsion. The results of the homogeneity test can be seen in Table 4.

Table 4. Results of Homogeneity Test

Cream	Homogeneity Day-				
	0	7	14	21	28
F1	-	-	-	-	-
F2	-	-	-	-	-
F3	-	-	-	-	-

Description: (-) Homogeneous, (+) Not Homogeneous

The cream's spreadability was evaluated to determine the extent of the spread of the cream when applied to skin. So, the ease of laying the preparation to the skin could be seen. The dispersion surface produced by increasing the loading is intended to describe the dispersion characteristics. The resulting surface area is directly proportional to the increase in the added load [18]. The dispersion requirement for topical preparations is about 5-7 cm [14]. The results of the dispersion test can be seen in Table 5.

Table 5. Results of Spreading Power Test

Cream	Spreading the power of the day-				
	0	7	14	21	28
F1	8.3	8.1	8.1	8	7.9
F2	5.6	5.1	5.1	5	4.9
F3	5.2	5.1	5	4.8	4.7

Description: F1(Formula 1), F2 (Formula 2), F3(Formula 3).

A viscosity test is carried out to determine the viscosity level of the resulting preparation. Viscosity is a statement of a liquid to flow. The higher the viscosity, the more difficult it is to flow/ the greater the resistance [9]. The viscosity required by SNI 16-4399-1996 is 2.000cp-50.000cp. Viscosity test results can be seen in Table 6.

Table 6. Viscosity Test Results

Viscosity Day-	**Cream**		
	F1	F2	F3
0	47,500	80,100	91,100
7	48.000	80,800	91,500
14	48,800	80,800	91,600
21	48,900	80,500	91,600
28	48,900	81,100	91,800

Description: F1(Formula1), F2(Formula 2), F3(Formula 3).

The emulsion type test was conducted to determine whether the cream made was classified as oil-in-water (O/W) cream or water-in-oil (W/O) cream. Determination of the type of emulsion preparation is carried out by placing a certain amount of preparation on an object glass, adding one drop of methyl blue, and stirring with a stirring rod. If the methyl blue is evenly distributed, the preparation has an o/w emulsion type, but if only blue spots are present, the preparation has a w/o emulsion type [15]. The results of the emulsion type test can be seen in Table 7.

Table 7. Emulsion Type Test Results

Cream	**Emulsion Type**				
	0	7	14	21	28
F1	O/W	O/W	O/W	O/W	O/W
F2	O/W	O/W	O/W	O/W	O/W
F3	O/W	O/W	O/W	O/W	O/W

Description: F1(Formula 1), F2 (Formula 2), F3(Formula 3), O/W (Oil in Water)

Discussion

The manufacture of grapeseed oil is carried out by Lansida Herbal Technology, Yogyakarta, Central Java. Cream manufacture is divided into three formulas, where each formula differs only in adding the emulsifier concentration. In the first formula (F1), the added formula was stearic acid as much as 10% of the total formula. In the second formulation (F2), the emulsifier was added to 12% of the complete formula. And the third formula (F3) emulsifier added is 14% of the total formula. The procedure for making cream begins with heating the mortar and stamper by inserting hot water into the mortar and stamper, which is then allowed to stand for a while until the mortar is prepared for the mixing process of the two phases.

The melting of the oil phase, namely 5% grapeseed oil, 10% stearic acid (F1), 12% stearic acid (F2), 14% stearic acid (F3), 1% cetyl alcohol, 1% stearyl alcohol was carried out on a water bath at a temperature of ± 70˚C. Heating is carried out at a temperature of ± 70˚C because this temperature is the highest melting point for melting materials in the oil phase, namely stearic acid with a melting point of 69-70˚C, cetyl alcohol with a melting point of 45-52˚C. After the dissolved oil phase was added, propyl paraben and methyl paraben into the oil phase and stirred until homogeneous. The solubility of propyl paraben and methyl paraben is very slightly soluble in water, soluble in 3.5 parts of ethanol (95%), in 3 parts of acetone, and 140 parts of glycerol and 40 parts of fatty oils, easily soluble in alkaline hydroxide solutions. Melting Temperature 95° C to 98°C [12].

At the same time, in the aqueous phase, 2% triethanolamine functioning emulsifier, and 10% glycerin which functions as a humectant and emollient, were mixed into distilled water at a temperature of ± 70˚C until all ingredients were dissolved. Added methylparaben stir until homogeneous. Mixing the oil and water phases is carried out at the same temperature of ± 70˚C to avoid separation between the two steps. Then the two phases are combined in a hot mortar and stirred using a stamper until a creamy mass is formed. After the cream mass is created, the preparation is put into a cream container.

The finished cream preparation was stored at room temperature. Physical evaluation included organoleptic observation, pH measurement, viscosity testing, homogeneity testing, spreadability and emulsion type. Physical evaluation of cream preparations aims to determine the effect of variations in stearic acid concentration on the physical properties of the preparation. It includes cream organoleptic, pH, dispersibility, homogeneity, viscosity, and cream type tests.

The organoleptic observations of F1, F2 and F3 creams on day 0 showed that the cream was white, odourless, and soft (Table 2). In F1, the cream did not change in colour, smell or texture after 28 days of storage. While in F2 and F3, the texture varies to become thick and solid on the 14th day of storage until the 28th day. It is influenced by the high viscosity of the cream preparation, which affects the viscosity of the preparation.

The pH value of cream F1, F2 and F3 on the 0th day in a row was 7. On the 7th day of storage, F1, F2 and F3 decreased the pH to 6 (Table 3). Changes in the pH of the preparation during storage indicate that the preparation is less stable during storage. This instability may damage the product during storage or use. Changes in the pH value will be affected by the medium decomposing by high temperatures during manufacture or storage that produce acids or bases. These acids or bases affect pH. Besides, changes in pH caused by environmental factors such as temperature, poor storage, and extracts that are less stable in preparations due to oxidation. The results of pH measurements at F1, F2 and F3 met the physiological pH range.

Based on the results of the homogeneity test (Table 4) that the test observations on day 0 to day 28 of storage, there is no presence of coarse particles and no separate constituent components. So F1, F2 and F3 creams indicate that the preparation of grapeseed oil cream is homogeneous.

Table 5 expresses the results of evaluating the spreadability of grape seed oil cream. Each formula shows a different spreadability when given a load of 150 g above the coverslip of the cream preparation. There was a decrease in dispersion until the 28th day of storage. F1 has a higher dispersion value than F2 and F3. It shows that the greater the concentration of stearic acid, the smaller the spread area produced due to an increase in viscosity. The wider the spread area created by a cream, the cream will have a better spreading ability when applied. However, the results showed that F1 did not meet the dispersion test requirements, while F2 and F3 met the criteria for the dispersion test.

From the results of the evaluation of the viscosity of the three formulas, Table 6 shoes the evaluation results of F1 meet the viscosity requirements. While the evaluation results for F2 and F3 did not meet the viscosity requirements. It is due to variations in the concentration of the stearic acid emulsifier. The greater the emulsifier content, the greater the resulting viscosity. Likewise, the results obtained in F2 and F3 with concentrations of stearic acid F2 (12%) and F3 (14%) were compared to stearic acid concentrations at F1 (10%).

From the evaluation of the emulsion type (Table 7), the three formulas were classified as oil-in-water (O/W) cream even though they had been stored for 28 days. Judging from the results of the statistical test of pH values that there was no significant difference in pH values between F1, F2 and F3 on day 0 (Asymp. Sig. =1,000; $p > 0.05$), day 7 (Asymp. Sig. =1,000; $p>0.05$), day 14 (Asymp. Sig. =1,000; $p>0.05$), day 21 (Asymp. Sig. =1,000; $p>0.05$), and the 28th day (Asymp. Sig. =1,000; $p>0,05$). Thus, the level of stearic acid does not affect the pH of F1, F2 and F3 preparations.

From the Shapiro-Wilk normality test, all F1 Spreading Power data on days 0 and 21 show a P value> 0.05. It means that the data is normally distributed, as well as on F2 on the 7th, 21st and 28th days, and F3 on day 0 and 21 showed a P value> 0.05 which means that the data is normally distributed. While on the 7th, 14th and 28th day, F1 showed a P value <0.05, which means that the data are not normally distributed. Likewise, on the 0th and 14th days F2, and F3 on the 7th, 14th and 28th days, the P value <0.05 means that the data are not normally distributed. So, the Kruskal-Wallis statistical test was carried out.

Judging from the results of the Kruskal-Wallis test it can be concluded that there is a significant difference in the dispersion value between F1, F2 and F3 on day 0 (Asymp. Sig. = 0.027; $p<0.05$), day 0 14 (Asymp. Sig. = 0.038; $p < 0.05$), day 21 (Asymp. Sig. = 0.032; $p < 0.05$), and day 28 (Asymp. Sig. = 0.030; $p < 0.05$) Thus it can be concluded that the level of stearic acid affects the dispersion.

From the Shapiro-Wilk normality test, all F1 viscosity data on the 0, 7th, 21st and 28th days showed a P value> 0.05. It means that the data was normally distributed, as well as on the F2 day, 7th and 7th days. 21st and F3 on the 7th day showed a P value> 0.05 which means that the data is normally distributed. Meanwhile, on the 14th day of F1, the P value <0.05 means that the data are not normally distributed. Likewise, on the 0, 14th, 21st and 28th days F2, and F3 on the 0, 14th, 21st and 28th day, the p value <0.05. It means that the data is not normally distributed. So, the Kruskal-Wallis statistical test was carried out.

Judging from the Kruskal Wallis statistical test, it can be concluded that there is no significant difference in viscosity values between F1, F2 and F3 on day 0 (Asymp. Sig. = 0.026; $p<0.05$), day 7 (Asymp. Sig. =0.027; $p<0.05$), day 14 (Asymp. Sig. =0.025; $p<0.05$), day 21 (Asymp. Sig. =0.027; p<0, 05), and the 28th day (Asymp. Sig. = 0.026; $p<0.05$). Thus, the level of stearic acid affects viscosity.

Conclusion

Grape Seed Oil can be formulated into O/W type cream preparations or oil in water. The physical evaluation requirements meet many test. It includes organoleptic test parameters, homogeneity, pH, spreadability, viscosity and emulsion type test. Grapeseed oil cream formula with various concentrations of stearic acid emulsifier 10% (F1) can meet the physical evaluation test parameters of the cream except for the spreadability test. F2 (12% stearic acid) and F3 (14% stearic acid) can meet the cream physical evaluation test parameters except for the viscosity test. Variations in the concentration of stearic acid significantly affect the physical evaluation of dispersion and viscosity. Further research is needed on the antioxidant activity test in the formulation of grapeseed oil cream. It is necessary to do further testing regarding irritation test, adhesion and total microbial test of grape seed oil cream preparations.

Conflict of Interest

We declare that there is no conflict of interest.

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