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Research Article

Formulation and test of physical properties of eyeshadow cream with natural dyes of Kasumba turate flower extract (*Carthamus tinctorius* L.)

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ABSTRACT

Safflower (*Carthamus tinctorius* L.) contains two natural pigments,, namely yellow carthamin and orange-red carthamin dye which are used as natural dyes. The research aimed to see whether safflower extract could be used as a natural coloring in eyeshadow cream and the physical properties of eyeshadow cream. The 70% ethanol extract of safflower was formulated into lip cream preparations, with varying concentrations of 0% (K-), 1% (F1), 2% (F2), and 3% (F3). Testing the physical properties of the preparation, namely the organoleptic test, homogeneity test, pH test, type of cream test, spread test, spreadability test, viscosity test, and hedonic test. The formulation of the lip cream of safflower extract showed a distinctive smell of orange essence, yellow to brownish yellow color, semi-solid form, homogeneous preparation until the 28th day, easy to apply, had a pH (4.5-7.0), type cream O/W, has a viscosity of 10,000-20,000 cPs and spreadability of 5-7 cm. This study concludes that Safflower extract can be used as a natural dye in Eyeshadow cream preparations and meets the requirements of good physical properties of Eyeshadow cream preparations.

1. Introduction

The variety of colors found in eyeshadow can be used to give an interesting shadow to the eye. Eyeshadow usually comes in pink, brown, silver, blue, green and dark red (Barus and Kaban, 2019). Dyes are the most important ingredients in eyeshadow preparations. Colorants consist of synthetic dyes and natural dyes (BPOM, 2013). The use of synthetic dyes for a long time will cause health problems because

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these materials are carcinogenic. Rhodamine B can irritate the respiratory tract, can cause liver damage and is a carcinogenic substance (Ulfa and Hardianti, 2017).

Kasumba turate contains 2 large groups of water-soluble pigments, namely yellow carthamidine and dye carthamin, which are orange-red in color and soluble in alkaline solutions. The flower content has 0.3-0.6% carthamin (Vossen, 2001). *Eyeshadow* in the form of a cream has the advantage of being able to adhere to the surface where it is used for a long time before this preparation is washed or removed, the form of a cream can give a shiny, oily, moisturizing effect and is easily spread evenly, easy to wipe and easy to wash off with water (Ulfa and Hardianti, 2017). The ethanol extract of kasumba turate flower (*Cartamus tinctorius* L.) showed a blackish red color and secondary metabolites contained in kasumba turate flower were alkaloids, flavonoids, saponins, terpenoids, tannins and anthraquinones (Hamsidi et al., 2019a). Researchers desire to further study the formulation and physical properties of eyeshadow cream prepared with natural kasumba turate flower extract.

2. Materials and Methods

This type of research is a laboratory experiment, namely 70% ethanol extract of kasumba tarate flowers which has been obtained from several tests, then formulated as a natural dye in the form of eyeshadow cream with concentrations of 0%, 1%, 2%, and 3% and Eyeshadow cream used as a comparison. Then a physical evaluation of the eyeshadow cream preparation was carried out in the form of organoleptic testing, homogeneity, spreadability, pH, viscosity, and cream type testing.

This research was carried out from January 2021 to March 2021 at LIPI Cibinong Bogor to determine plants and at Muhammadiyah University A.R. Fachruddin for the extraction process, making eyeshadow cream preparations and evaluating the preparations. The process of testing the water content of simplicia and extracts, as well as the remaining solvent extract tests were carried out at the Health Laboratory of the Special Capital Region of Jakarta.

Equipments

Stirring rod, beaker glass, porcelain cup, Funnel, Erlenmeyer, Measuring cup, Mortar, Analytical Balance, Glass slide, pH Meter, Dropper, Rotary Evaporator, Spoon Horn, Spatula, Stamper, Dipper, Waterbath,

Materials

Stearic acid (Wilmar Nabati, Indonesia), aquadest (Indonesia), Alcohol (Palapa, Indonesia), butyl hydroxy toluene (Making Cosmetic, US), erythrosine, methylparaben, petrolatum (Raj Petro, India), propylene glycol (Dow, Singapore), propylparaben (Alpha Chemika, India), adeps lanae (Fagron, US) , oleum citri (Choice,US) , triethanolamine (Making Cosmetic, US), cetyl alcohol (Akoma, UK) Pharma grade.

Method

1. Sample collection

   Kasumba turate flowers (*Carthamus tinctorius* L.) were taken from Kajuara Village, South Sulawesi

2. Plant determination.

   Determination of kasumba tarate flowers is done to ensure that the plants used are correct.

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Sample making

The samples were sorted by wet sorting by separating foreign objects such as impurities that were still left in the sample. Then the sample was washed with running water until no dirt remained, then drained in a container and placed in a clean container. Then it was dried in a drying cabinet at a temperature of 50^{o}C, after that it was sorted dry. then the dried kasumba turate flowers were mashed and sieved with a 40 mesh, The dry weight of the simplicia was weighed and the simplicia was put in a tightly closed container.

Simplicia quality parameters

Nonspecific Parameters

Drying loss: weigh 1g of sample, put it in the moisture analyzer at 105^{o}C for 30 minutes, replicate 3 times and record in percent form (Kementerian Kesehatan RI, 2017).

Moisture content: weigh 0.5 g of sample, put it in the Karl Fischer tool, and record the results.

Ash content: weigh 2g sample using a constant cup. Heat the sample on a hotplate at low temperature until the sample ash disappears. Put it in a furnace at a temperature of ±550ºC, leave it for 5 hours, and weigh it at a constant weight (repeat ashing if the weighing value is not constant) (Kementerian Kesehatan RI, 2017).

Specific parameters

Organoleptic test: then weigh the sample which is already constant (repeat ashing if the weighing value is not constant) (Depkes RI, 2000).

Extract manufacture

Maceration: 288 g of dry Simplicia powder was put into a macerator. Then, 2880 ml of 70% ethanol solvent was added (1:10) and soaked for 24 hours before being filtered (Hamsidi et al., 2019b).

Extract Quality Parameter Test

Specific parameters

Organoleptic test: Form (solid, dry powder, viscous, liquid), Color (yellow, brown, etc.), Odor (aromatic, odorless, etc.).

Non-specific parameters

Water content: Weigh 5g of sample, put it in the Karl Fischer tool then get the results

Ash content: Weigh 1g of sample using a constant cup. Heat the sample on a low-temperature hotplate until the sample ash disappears. Place the sample in a furnace with a temperature of ±550ºC and leave for 5 hours, weighed after constant (repeat ashing if the weighing value is not constant) (Kementerian Kesehatan RI, 2017).

Remaining solvent: Prepare a standard series of ethanol (0.001; 0.03; 0.05; 0.1; 0.3; 0.5) %. The sample was weighed ± 0.5000 grams in a 10 mL volumetric flask. Then dissolve it with aquabidest Condition the instrument with temperature column and injector at 170°C, and detector at 200°C. Then the standard series was injected 1 µl into the injection column on the gas chromatograph. calculated the linearity of the standard series (r > 0.99). Then, 1 µl of the prepared sample was injected

using a micro syringe into the injection column on a gas chromatograph. Calculate the peak area of ethanol and then look for the ethanol content with the following calculations.

Phytochemical Screening

Flavonoid test: 5 ml of liquid extract which is put into a test tube. Mg powder, 2 ml of 2 N HCl and 5 ml of amyl alcohol were put into a test tube. Then shaken until it becomes 2 phases, positive results are indicated by the formation of an orange color on the amyl alcohol layer (Handayani et al., 2017).

Tannin test: put 3 ml of liquid extract in three test tubes, the first tube is dropped with 10% FeCl3 the positive result is green, blue or black. The second tube was dripped with 1% gelatin, a positive result formed a white precipitate. The third tube was dripped with steasny, a positive result formed a red precipitate. Then filter the three tubes and the filtrate is added with Na acetate until it is clear and drops of 10% FeCl solution. A positive result shows the blue color of the ink (Handayani et al., 2017).

Saponin test: 10 ml of liquid extract was shaken vertically for 10 seconds, let stand for 10 minutes. A positive result is the formation of foam with a height of 1-10 cm. Then 2 N hydrochloric acid was added, and positive results for saponins were shown to remain stable (Handayani et al., 2017).

Alkaloid test: 2 g of simplicia powder moistened with 5 ml of ground ammonia then add 10 ml of crushed chloroform, then filter and the extraction filtrate 2x with 10% HCl (1:2). The acid filtrate is taken and then put in a test tube and dripped with Mayer's reagent. Positive alkaloid formation of white precipitate (Handayani et al., 2017).

Triterpenoid test: A total of 2 g of powder was macerated using 20 ml of ether, then filtered and the filtrate was evaporated and the Liebermann-Burchard reagent was dropped. A positive result of steroids/triterpenoids is the formation of a blue green or red purple color (Handayani et al., 2017).

Eyeshadow Cream Preparation Formula Kasumba Turate Flower Extract

Table 1. Eyeshadow Cream dosage formula

Components	F1 (%)	F2 (%)	F3 (%)	Function
Kasumba turate flower	1	2	3	Aktif substance
Stearic Acid	10	10	10	Emulsifying Agent, Oil base
Cetyl alkohol	5	5	5	Emulsifying
Triethanolamin	0,5	0,5	0,5	Emulsifying
Petrolatum	15	15	15	Emollient
Propilenglikol	5	5	5	Humectant
Lanolin	5	5	5	Moisturizer
Butilhidroksi toluena	0,1	0,1	0,1	Antioxidant
Oleum citri	0,2	0,2	0,2	Fragrance
Metil paraben	0,018	0,018	0,018	Preservative
Propil paraben	0,02	0,02	0,02	Preservative
Aquadest ad	100 ml	100 ml	100 ml	Solvent

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Stage of making Eyeshadow Cream

The first stage is weighing the ingredients, which are separated into two phases, namely the oil phase and the water phase. Oil phase ingredients (stearic acid, cetyl alcohol, petrolatum, lanolin, and propylparaben) and water phase ingredients (triethanolamine, propylene glycol, methylparaben, and aquadest). The oil phase is preheated at 70^0C. Then, one by one, the oil phase ingredients were heated. Stearic acid, cetyl alcohol, petrolatum, lanolin, and propylparaben were put into a mortar and pestle until homogeneous (phase A was formed). Furthermore, making the water phase without being heated, input one by one the ingredients, namely triethanolamine, propylene glycol, methylparaben and aqua dest, put it in a mortar and pestle until homogeneous then add little by little the flower extract of kasumba turate with each concentration of FI (1%), F2 (2%), and F3 (3%) grind until homogeneous (phase B is formed). Next, enter phase B into phase A and grind until homogeneous then add oleum citri grind until homogeneous, and stir until a creamy mass is formed. Then put it in the eyeshadow cream container.

Table 2. Safflowers Extract Concentration

Materials	F1 (%)	F2 (%)	F3 (%)
Kasumba Turate Flower Extract	1	2	3
Cream Eyeshadow Base	99	98	97

Physical evaluation of Eyeshadow Cream preparations

Organoleptic test: Tests carried out include smell, taste, and color (Jessica et al., 2018).

Homogeneity test: Preparation of eyeshadow cream is applied in a certain amount on the slide (Jessica et al., 2018).

pH test: The instrument was calibrated with an acidic pH buffer solution of 4.0. 1g of sample was diluted with purified water. The electrode is immersed in the solution. Let the tool show a constant pH value

Spreadability test: weigh 1g preparation, Place it in the middle of two flat pieces of glass. Then add 125 g of weight and let stand for 1 minute and note the diameter of the eyeshadow cream that spreads (Jessica et al., 2018).

Cream type test: 1 drop of methylene blue solution is given to 0.1 gram of cream, and then the spread of methylene blue color is observed in the preparation (Elmitra, 2017).

Viscosity test: The preparation is put into a container, then the spindle numbers L-4 and 50 rpm are installed. tool will rotate to observe the cPs value listed at the 25th second.

Room temperature stability test: carried out by determining the organoleptic, pH, and viscosity at time intervals of 0, 3, 7, 14, 21, to 28 days after storage (Muhsinin et al., 2018).

3. Results and Discussions

Plant determination

The results of the determination show that the plant that will be used in this research is the flower of the Kasumba turate.

Simplicia making

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Making simplicia starts from the harvesting stage. Kasumba turate flowers that have been harvested are carried out wet sorting by separating foreign objects such as gravel or soil that are still attached to the plant, then cleaned with running water, then dried in a drying cupboard at 50ºC. The purpose of drying is to reduce the moisture content. After that, dry sorting was carried out to separate foreign materials that were still attached to the simplicia. The dried samples of kasumba turate flowers were mashed using a blender and sieved with mesh no. 40. The aim was to refine and sieve, to homogenize the simplicia to facilitate the extraction process. Simplicia powder obtained as much as 288 grams.

Simplicia test parameters

Specific Parameters

Specific parameters are the qualitative and quantitative aspects of chemical compounds (Kementerian Kesehatan RI, 2017).

Organoleptic: Test organoleptic aims to provide a simple initial introduction as objectively as possible to simplicial using the five senses, the results of organoleptic testing of simplicial kasumba turate flowers with an elliptical shape, distinctive odor, yellow color, and chelate taste (Kementerian Kesehatan RI, 2017).

Table 3. Specific simplicia test parameter results

Organoleptic Simplicia	
1. Texture	Elips
2. Scent	Typical Kasumba Turate Flower
3. Color	Yellow
4. Flavor	Chelate

Non Specific Parameter Test

Non-specific parameter tests were carried out to determine the purity and the presence or absence of contaminants in the simplicia.

Table 4. Results of non-specific simplicia test parameters

Characteristics	Results (%)
Water Content	3,57
Ash Content	3,67 ± 0,58
Drying Shrinkage	7,7 ± 0,71

Water content testing: aims to determine the water content of a material (Depkes RI, 2000). Determination of water content was carried out at LABKESDA Jakarta using the Karl Fischer method.

Ash content test: Determination of ash content was carried out at the Natural Materials Laboratory of the Muhammadiyah College of Pharmacy, Tangerang. The results obtained were in accordance with the requirements of the ash content for the simplicial flower kasumba turate< 4.6% (Kementerian Kesehatan RI, 2017).

Drying shrinkage test: Determination of drying shrinkage was carried out at the Instrument Laboratory of the Muhammadiyah Tangerang College of Pharmacy. The results obtained are in accordance with the quality requirements of the simplicial < 10% (Kementerian Kesehatan RI, 2017).

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Making Kasumba Turate Flower Extract

Carry out the maceration process by soaking the simplicia powder in 70% ethanol solvent (1:10) where 288 grams of simplicia kasumba turate powder is soaked in 2880 ml of 70% ethanol for 1 x 24 hours and soaked first for 6 hours while stirring occasionally, then let it rest for more less than 18 hours, after the maceration process is complete, filter using filter paper and obtain the filtrate. The dregs of the results filtering is done by re-maceration or remaceration. Remaceration is done to attract more active substance compounds. Remaceration was carried out 3 times. The filtrate obtained was concentrated using a rotary evaporator at a temperature of 50°C at a speed of 5 rpm, and focused again with a water bath at a temperature of 50°C to become a thick extract. The purpose of re-concentration with a water bath is to reduce or eliminate the solvent contained in the extract. The yield results obtained can be seen in **Table 5.**

Table 5. Extraction Result of Kasumba Turate Flower (*Carthamus tinctorius* L.)

No.	Type	Results
1.	Powder	288 gram
2.	Thick Extract	45,57 gram
3.	Result	15,82 %

The extract yield results obtained in this study were greater than the results of previous research conducted by Hamsidi et al. (2018) who used 3 kg of kasumba turate flowers which were extracted with 10 L of 80% ethanol by maceration with a yield of 5.96%. In the latest research conducted by Juwita et.al. (2019) obtained a yield of 19.54% using 649.58 g of sample with 6 L of 70% ethanol solvent and the amount of crude extract obtained was 126.91 g and this yield was higher than in this study.

The occurrence of differences in yield values is largely determined by the characteristics of the solvent and the sample used, such as the length of heating in the oven which can cause evaporation of the volatile substances contained in the sample (Juwita et al., 2021)**.**

Extract test parameters

Specific test parameters

Table 6. Specific extract test results

Organoleptic Exract	
1. Texture	Thick Extract
2. Scent	Typical Kasumba Turate Flower
3. Color	Browniseh Yellow

Non-specific test parameters

The non-specific parameters were water content, ash content and residual solvent.

Table 7. Non-specific extract test results

Characteristics	Results (%)
Water Content	10,12
Ash Content	4,22 ± 0,17
Remaining Solvent	0,01

Water content testing: The purpose of the extract water content testto provide a minimum limit or range of the amount of water content in the material (Depkes RI, 2000). The results of testing the water content of the extract obtained are 10,12%, where it exceeds the specified requirements but is still in the range of thick extract moisture content between 5-30% (Voigt, 1994). Water content that is too high (> 10%) can cause the growth of microbes to reduce the stability of the extract (Purwoko et al., 2020).

Ash content test: The purpose of the ash content test is to provide an overview of internal and external mineral content originating from the initial process to form natural plant-derived materials and contaminants during the process (Depkes RI, 2000). The results obtained have met the requirements of 4,22 ± 0,17% and the total ash content according to the Indonesian Herbal Pharmacopoeia is not more than <10%. Another study produced an ash content of 12.27% (Nurfaidillah et al., 2024).

Solvent Residual Testing: The residual solvent test aims to provide assurance that during the process it does not leave residual solvent which should not be present (Aprilyanie et al., 2023). The results of the remaining solvent content of kasumba turate flower extract obtained 0.01%. The results of these inspections meet the requirements for the maximum limit of residual solvent in ethanol, which is not greater than 1%.

Phytochemical Screening

Phytochemical screening aims to determine the secondary metabolites contained in the 70% ethanol extract of kasumba turate flowers.

Table 8. Phytochemical Screening Results

Phytochemical Screening	Results	Description
Alkaloid	White precipitate is formed	+
Flavonoid	An orange coloris formed on theamylalcohol layer	+
Kuinon	A red precipitate is formed	+
Tanin	No white precipitate is formed	-
a. Katekat	No red precipitate is formed	-
b. Galat	Ink blue color formed	-
Saponin	Foam is formed	+
Steroid/triterpenoid	Purplish red formed	+

Physical Evaluation of Eyeshadow Cream Preparations

Organoleptic Test

Table 9. Results of organoleptic completion

Formula	Texture	Scent	Colorcode
F1 (1%)	Semi Solid	Orange essence	RAL 1018 (Zinc Yellow)
F2 (2%)	Semi Solid	Orange essence	RAL 1012 (Lemon Yellow)
F3 (3%)	Semi Solid	Orange essence	RAL 1032 (Broom Yellow)
K(-)	Semi Solid	Orange essence	RAL 9010 (Pure White)
K(+)	Semi Solid	Distinctive scent	RAL 1018 (Zinc Yellow)

Note : K(-) : Preparation without 0% ektract
K(+) : Eyeshadow Cream preparations on the market

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RAL 1018 Zinc Yellow

RAL 1012 Lemon Yellow

RAL 1032 Broom Yellow

RAL 9010 Pure White

RAL 1018 Zinc Yellow

The results obtained from the five formulas show that the greater the concentration of extract contained in the eyeshadow cream, the darker the color produced by the eyeshadow cream (Ulfa & Hardianti, 2017).

Homogeneity Test

Table 10. Homogeneity Test Results

Formula	Day					
	0	3	7	14	21	28
K(-)	+	+	+	+	+	+
K(+)	+	+	+	+	+	+
F1 (1%)	+	+	+	+	+	+
F2 (2%)	+	+	+	+	+	+
F3 (3%)	+	+	+	+	+	+

Note : K(-) : Preparation without 0% ektract K(+) : Eyeshadow Cream preparations on the market
+ = Homogenic

The results of the observation of the homogeneity of the eyeshadow cream have met the physical homogeneity requirements, namely that there are no visible coarse particles if applied to the slide, there are no particles, and the separation between the components that make up the emulsion.

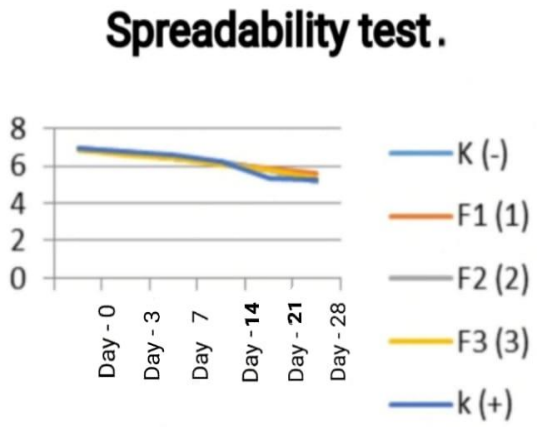

Figure 1. Spreadability test

Results dispersion test then F2 with a concentration of 2% is a preparation that has the best viscosity value among other formulas with a dispersive value range of 6.93-5.15 cm. Good dispersion for semi-solid preparations is 5-7 cm (Jessica et al., 2018).

Cream type test

Table 11. Cream Type Test Results

Formula	Test Result
F1 (1%)	+
F2 (2%)	+
F3 (3%)	+
Kontrol (-)	+
Kontrol (+)	+

Information :
+: A/M Type Cream
-: CreamType M/A

The results of the cream type test showed that the 3 formulas of *Eye shadow cream*. Kasumba turate flower extract (*Carthamus tinctorius* L.) as well as negative control and positive control obtained uneven color results and the color was only in the form of spots meaning the cream type was water in oil (W/M) (Elmitra, 2017).

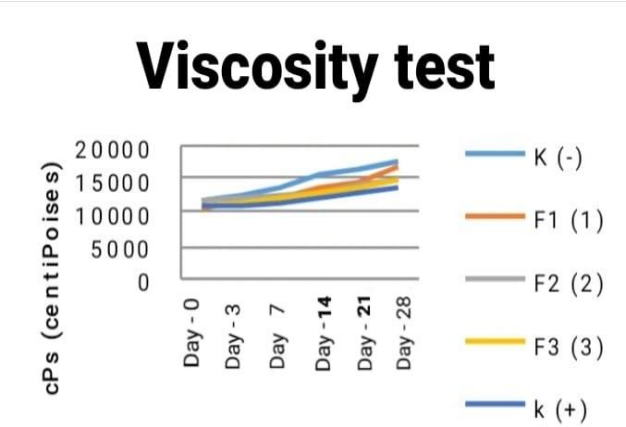

Figure 2. Viscosity test chart

Based on the results of testing the viscosity of the eye shadow cream, kasumba turate flower extract increased the viscosity value of all formulas every day. The results of the viscosity test on the control (-), control (+), F1, F2, and F3 with a concentration of 1%, 2%, 3% have met the requirements of good viscosity.e. 10,000-20,000 cPs (Jessica et al., 2018).

4. Conclusions

Based on the research that has been done, it can be concluded that:

1. Kasumba turate flower extract with extract concentrations of 1%, 2% and 3% produces a dye from Carthamidin (yellow) which can be formulated as an eyeshadow cream and has the potential to be developed as a natural dye in the cosmetics industry.
2. The results of the physical evaluation show that the resulting Eyeshadow cream has met the requirements for good quality Eyeshadow cream. The description test results have a yellow to

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brownish-yellow color with a semi-solid form and a distinctive smell of orange essence. The homogeneity test showed that the four formulas and the positive control had good homogeneity. The pH test met the skin pH requirements, namely 4.5-6.0. Cream type test all formulas have A/M cream type. The dispersion test met the requirements of 5-7 cm. The viscosity test met the requirements for a good eyeshadow cream viscosity, namely 10,000-20,000 cPs.

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