

Stability Test Of Dyes From Beetroot Extract (Beta Vulgaris L.) In Hair Dye Cream

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ABSTRACT

Beet tuber (*Beta vulgaris* L.) is a plant of the Amaranaceae family which contains folic acid compounds, potassium, vitamin C, magnesium, iron, copper, phosphorus, fiber, tryptophan, coumarin and betacyanin. Betacyanin is a derivative of betalain which contains red or red-violet pigments that can be developed as natural hair dyes. One method for developing natural hair dyes can be applied in the form of oil cream preparations in water. Where the purpose of this research is to find out whether betacyanin compounds contained in beetroot (*Beta vulgaris* L.) can be formulated in cream preparations and how the stability of betacyanin dyes in their application. The results of the evaluation of cream preparations showed a homogeneous semi-solid dosage form, has a distinctive odor, the degree of acidity met the scalp cosmetics pH criteria of 5.0-8.0, with the spreadability of preparations below 5 cm and standard deviation (SD) 0.1387. The analysis of the stability of beetroot extract (*Beta vulgaris* L.) conducted included the stability of the effectiveness of hair dye, color stability against washing, and color stability to sunlight. From the analysis that has been done, it can be concluded that the betacyanin compound contained in beetroot extract (*Beta vulgaris* L.) has a bad color stability in the form of cream preparations.

Keywords: beetroot, Hair Dyes Cream, standard deviation, Amaranaceae.

I. INTRODUCTION

Hair coloring can be assumed as one of the trends that has become a necessity in one's life. In fact, not a few of these individuals deliberately color their hair as a means of actualizing their character or personality, besides that hair coloring in today's era is no longer owned by women for mere fashion reasons. Besides, it is human nature to appear different. This situation is due to the influence of globalization that has penetrated among young people this century, they are competing to follow trends, one of which is the

trend of hair coloring that maximizes appearance and can be a source of self-confidence (Nasution et al., 2012).

Hair dye preparations are cosmetics used in hair makeup to color hair, either to restore the original hair color or change the original hair color to a new color (Directorate General of POM, 1985). Hair color was determined by the melanin pigment in the hair present in the cortex layer. The origin of the pigment melanin is melanocytes that are in the hair bulb. Melanocytes are cells that produce pigment (dye) which causes natural hair to have various colors (Bariqina&Ideawati, 2001).

Most of the hair dyes currently circulating in the community use synthetic dyes. Synthetic dyes were allowed to be used but with a certain level limit, whereas if synthetic dyes were used continuously for a long period of time and with excessive levels it can cause various health problems (Directorate General of POM, 1985). Dyes that are often used in cosmetic preparations are generally chemical dyes.

The National Agency of Drug and Food Control (BPOM) in 2009 has withdrawn several brands of hair dyes from circulation because they contain chemicals that are harmful to health. Most of the hair dyes that were withdrawn were because they contained red dye K3, red dye K10 (Rhodamine B) and orange dye K1. K3 red dye, K10 red dye (rhodamine B) and K1 orange dye are synthetic dyes that are generally used as paper, textile or ink dyes. This dye is a carcinogenic substance (can cause cancer). Rhodamine B in high concentrations can cause liver damage.

The use of natural dyes in cosmetic preparations as a solution was needed because of the relatively small side effects as well as to better utilize the natural potential of Indonesia which is rich in plants containing natural dyes (Bariqina&Ideawati, 2001). One of the plants that give natural color is beetroot (*Beta vulgaris* L.). The pigment found in beetroot is betacyanin. Betacyanin is a natural colorant that was widely used in food systems. Until now, betacyanin pigments that have been produced on

a large scale only come from beetroot (*Beta vulgaris* L.). Betacyanin from beetroot (*Beta vulgaris*L) has been known to have anti-radical effects and high antioxidant activity (Mastuti, 2010). The red color of fresh beetroot is due to the nitrogen-containing pigment betacyanin. Betacyanin is a derivative of betalain, Betalain is a red-violet and yellow-orange pigment that was widely found in tubers and flowers. Betacyanins are easily soluble in water solvents, so betacyanins are very well developed as natural dyes (Strack et al., 2003).

Based on the description above, the researchers were interested in formulating beetroot extract (*Beta vulgaris* L.) in the form of hair dye cream, and testing the stability of the cream preparation.

II. EXPERIMENTAL

Instrumentation and Materials

The instrumentation used in this study include: rotary evaporator (IKA Hb 10), mortar (Grodwn), stamper (Godwon), spatula, stirring rod (Iwaki), petri dish (Duroplan), beaker glass (Iwaki), evaporation cup (Iwaki), measuring cup (Iwaki), dropper (Iwaki), funnel (Iwaki), horn spoon (Pt. Alkin global), weight (Iwaki), analytical balance (PrecisaXB 220A), slide (Iwaki), glass bottle (Merch), pH meter (Macherey Nagel), graph paper, Brookfiel viscometer, flannel, gauze, and plastic pot.

The materials used in this research include beetroot (*Beta vulgaris* L.), carbomer (Aqupack), Na. Laurel sulfate (Bratachem), cocamide DEA (Bratachem), resorcinol (Bratachem), Na.sulphite (Bratachem), sodium EDTA (Bratachem), Cetyl Alcohol (Pharma Lab), Stearate-25 (Bratachem), Sodium Pyrophosphate (Bratachem), Hydrogen

Peroxide (Bratachem), Phosphoric Acid (Bratachem), Ammonia (Bratachem), Distilled Water (Bratachem).

Procedure of analysis

Sampling

The sample used in this study was beetroot (*Beta vulgaris* L.) obtained from Lembang Village, West Bandung Regency, West Java Province, Indonesia.

Plant Identification

Plant identification was carried out at the Andalas University Herbarium (ANDA) Department of Biology, Faculty of Mathematics and Natural Sciences, Andalas University, Padang, Indonesia.

Production of Beetroot Extract(*Beta vulgaris*L.)

The beetroot (*Beta vulgaris* L.) was peeled first and then washed thoroughly. The beetroot (*Beta vulgaris* L.) was weighed as much as 1 kg, then chopped and cut into pieces, then the beetroot (*Beta vulgaris* l.) was put intoextract. the extractwas squeezed and filtered using a flannel cloth to separate the beetroot extract from the pulp. The liquid obtained was then centrifuged at 3000 rpm for 10 min. The filtrate obtained was concentrated using a vacuum rotary evaporator with a temperature of 55°C, until concentrated tuber extract was obtained. The beetroot extract obtained was then added with vitamin C to pH 5 as an antioxidant (Sanchez et al. 2006).

Extract Characterization

Specific characterization carried out included identity and organoleptic examination. While the non-specific characterization includes total ash content, acid insoluble ash content and drying shrinkage.

Hair Color Cream Formulation

Hair Dye Cream formulations can be seen in Table 1.

Table1. Hair Color Cream Formulation

Material Composition	Formula (g)				Function
	F1	F2	F3	F4	
Beetroot extract (<i>Beta vulgaris</i> L.)	-	30	40	50	Active substance
Carbomer	1.5	1.5	1.5	1.5	Emulsifier
Cocamided DEA	2.5	2.5	2.5	2.5	Emollient
Na.Lauryl sulfate	14.0	14.0	14.0	14.0	Emulsifier
Resorcinol	1.3	1.3	1.3	1.3	color binder
Na. Sulfite	0.2	0.2	0.2	0.2	Stabilizer
Na. EDTA	0.2	0.2	0.2	0.2	Stabilizer
Cetyl Alcohol	2.0	2.0	2.0	2.0	Emulsifier

Stearic Acid	5.0	5.0	5.0	5.0	Emulsifier
Sodium pyrophosphate	0.1	0.1	0.1	0.1	Emollient
Hydrogen Peroxide	20.0	20.0	20.0	20.0	Oxidizing
Ammonia	9.0	9.0	9.0	9.0	Oxidizing
Phosphoric Acid (pH-5)	few drops	few drop s	few drops	few drops	Alkalizing agent
Distilled water	up to 100	up to 100	up to 100	up to 100	Solvent

Making hair dye cream without beetroot

Making cream formula 1 without the addition of beetroot extract (*Beta vulgaris L.*). Carbomer added enough water, then stirred until homogeneous (mass 1). Na. lauryl sulfite, cocamide DEA, resorcinol, Na. Sulfites, Na. EDTA, heated until dissolved, then stirred until homogeneous (mass 2). Solutions 1 and 2 were stirred until homogeneous. The base preparation was carried out at the same time, namely Cetyl alcohol and cetareth-25 were heated until dissolved (M1), Sodium Pyrophosphate was added to water and heated until dissolved (M2). M1 and M2 were mixed in a warm state, cooled, added hydrogen peroxide, and phosphoric acid and then stirred until homogeneous until a cream mass was formed. hair dye cream and base were mixed then add ammonia, grind until homogeneous. The cream is put in a container.

Making hair dye cream with the addition of beetroot

Making cream formulas 2,3 and 4 with the addition of beetroot extract (*Beta vulgaris L.*) Carbomer added enough water, then stirred until homogeneous (mass 1). Na. Laurel sulfite, cocamide DEA, resorcinol, Na. Sulfites, Na. EDTA, heated until dissolved, then stirred until homogeneous (mass 2). Solutions 1 and 2 were stirred until homogeneous. The base preparation was carried out at the same time, namely Cetyl alcohol and cetareth-25 were heated until dissolved (M1), Sodium Pyrophosphate was added to water and heated until dissolved (M2). M1 and M2 were mixed in a warm state, cooled, added hydrogen peroxide, and phosphoric acid and then stirred until homogeneous until a cream mass was formed. hair dye cream and base were mixed then add beetroot extract (*Beta vulgaris L.*) and ammonia, grind until homogeneous. The cream was put in a container.

Evaluation Preparation

a. Organoleptic Test

Organoleptic test was carried out to see the physical appearance by observing the shape, color and smell of the preparations that have been made (Anief, 2008).

b. Homogeneity Test

Cream homogeneity test was carried out to determine whether the mixing of each component in the manufacture of cream was evenly mixed. This is to ensure that the active substances contained in it have been distributed evenly. If applied to a piece of glass or other suitable transparent material, it must show a homogeneous composition (Ministry of Health of the Republic of Indonesia, 1979).

c. pH test

The pH test were carried out to see the level of acidity. The test was carried out using a pH meter, then the pH test was measured by taking a small sample of hair dye cream preparations from beetroot (*Beta vulgaris L.*). beetroot extract (*Beta vulgaris L.*) and then dissolved in a little distilled water, dip the pH meter indicator then let stand and see the test results. Preparations must meet the skin pH criteria, namely in the interval 4.5-6.5.

d. Spreadability test

The spreadability test was carried out to ensure an even distribution of the cream when applied to the skin which was carried out immediately after the gel was made. 0.5 g of cream was weighed and then placed on a petri dish. On top of the cream was placed another petri dish and weights with various weights of 5, 10, 20 and 100 grams, allowed to stand for 1 minute, then the diameter of the distribution was recorded. Good cream spread between 5-7 cm (Garg et al., 2002).

e. Freeze thaw test

Freeze thaw test was carried out to see the physical stability of the cream after being stored for one week at different temperatures, namely 40°C and 25°C. The temperature range for freezing conditions is >15°C, for cool conditions it is 15-25°C, for cold temperatures it is 2-8°C and for cool box temperatures 8-15°C. Storage was carried out in six cycles and one cycle lasted for one day at each temperature. This needs to be done to see the stability of the cream temperature during storage. Emulsion systems at high temperatures can cause an increase in the kinetic energy of the droplets of the dispersion phase so that it is easier to combine and an increase in the size of the globule diameter. While at cold temperatures the solubility of the emulsifier in the oil phase and in the water phase will decrease so that the effectiveness of the emulsifier to coat the globules was reduced (Lachman et al., 1994).

f. Test the effectiveness of hair coloring

Test the effectiveness of hair coloring tested on black hair. This evaluation was conducted to determine the presence or absence of color produced by beetroot extract (*Beta vulgaris* L.).

g. Irritation test

Irritation test was carried out to see whether the preparation of hair dye cream from beetroot had a reaction between the components so that an irritant substance was formed or not.

h. Cream type testing

The type of cream test was carried out to see whether the preparations that had been made were of the type of cream M/A (oil in water) or A/W (water in oil).

i. Color stability test against washing

The color stability test for washing hair dye cream preparations from beetroot was carried out to see changes in hair color after washing.

j. Color stability test against sunlight

Color stability test against sunlight preparation of hair dye cream from beetroot was carried out to see changes in hair color after being exposed to the sun.

Data analysis

The results of the study were analyzed using tables and textual.

III. RESULT AND DISCUSSION

In this study, the sample used is beetroot obtained from Lembang Village, West Bandung Regency, West Java Province, Indonesia. 1 kg of fresh beetroot was collected. Then the sample was identified at the Andalas University Herbarium, Department of Biology, Faculty of Mathematics and Natural Sciences (FMIPA), Andalas University, Padang, Indonesia. This sample identification was carried out to find out and ensure the correctness of the identity of the sample and avoid errors in sampling. The beetroot (*Beta vulgaris* L.) was peeled first and then washed thoroughly, the beetroot (*Beta vulgaris* L.) was weighed as much as 1 kg, then chopped and cut into pieces, then the beetroot (*Beta vulgaris* L.) is put into extract. The extract was squeezed and filtered using a flannel cloth to separate the beetroot extract from the pulp. The liquid obtained was then centrifuged at 3000 rpm for 10 min. The filtrate obtained was concentrated using a vacuum rotary evaporator with a temperature of 55°C, until concentrated tuber extract was obtained. The beetroot extract obtained was then added with vitamin C to pH 5 as an antioxidant (Sanchez et al. 2006). The principle of this tool is to separate the extract from the liquid by means of heating accelerated by the rotation of the flask and can evaporate 5-10°C below the boiling point of the solvent due to a drop in pressure.

Furthermore, beetroot extract was formulated into a hair dye cream preparation. Cream is a semi-solid dosage form containing one or more drug ingredients dissolved or dispersed in an appropriate base material (FI Edition IV). Hair dye preparations are cosmetic preparations used in hair make-up to color hair, either to restore the original hair color or other colors (Ministry of Health of the Republic of Indonesia, 1985). Cream was made by mixing the oil phase and the water phase. The cream requirement is the presence of an emulsifier, base and active ingredient. The use of cetylalcohol, stearic acid and carbomer as emulsifiers to stabilize the cream and the use of emollients such as cocamide DEA and sodium pyrophosphate to moisturize the cream at the time of application. The use of Sodium EDTA was needed for stability in cream preparations.

The mechanism of changes in hair color using hair dye preparations is oxidation and coupling or condensation at an alkaline pH atmosphere using ammonia and an oxidizing agent using hydrogen peroxide. In hair dye, the coloring process goes through 2 stages. The first stage, hydrogen peroxide will dissolve or oxidize hair

pigments so that the hair will fade in color. The second stage, the hair cuticle (the outermost part of the hair) must be opened before the hair dye enters the hair using ammonia. Ammonia compounds in the hair will open the hair cuticle so that the hair dye penetrates into the hair cortex so that the non-pigmented hair was easily colored.

To determine the quality of the hair dye cream preparation is good or not, it is necessary to evaluate the preparation consisting of organoleptic test, homogeneity test, pH test, spreadability test, viscosity test, freeze thaw test, irritation test, cream type test, hair dye effectiveness test, staining test against sunlight and staining test on washing power. The viscosity test here is not carried out because the tool to be used is not in good condition.

Organoleptic test was done by observing the shape, color and smell. Organoleptic test was done by observing the shape, color and smell. In F1, F2, F3 and F4 the resulting form is a semi-solid preparation and the resulting odor is the typical smell of beetroot. The results of the evaluation showed that F1 (without beetroot extract (*Beta vulgaris* L.)), F2 (with beetroot extract (*Beta vulgaris* L.) 30%), F3 (with beetroot extract (*Beta vulgaris* L.) 40%) and F4 (with beetroot extract (*Beta vulgaris* L.) 50%) had different colors for six days of storage. The color changed day by day, according to Han et.al., 2004, this was due to the unstable betacyanin pigment contained in beetroot (*Beta vulgaris* L.), so the storage time of hair dye cream from beetroot (*Beta vulgaris* L.) is relatively short.

The homogeneity test was carried out to see whether the hair dye cream preparations were homogeneous or not. Homogeneity is one of the factors that affect the quality of cream preparations. It was said to be a homogeneous cream if the preparation does not contain lumps of residual material that does not dissolve or is not well mixed. All cream preparations produced meet the requirements of a good gel, namely homogeneous.

pH test was carried out using a pH meter. The results of the pH evaluation showed that the beetroot cream preparations at F1, F2, F3 and F4 produced a pH above 6 and a standard deviation (SD) of 0.0972. The results of this test indicate that the preparations that have been made meet the

criteria for scalp cosmetic pH of 5.0-8.0. Because the more acidic the pH value, the more irritating the skin, and the more alkaline the pH value, the more scaly or rough the skin.

The spreadability test was carried out to ensure the ability and even distribution of the hair dye cream preparation when applied or applied to the skin. If a preparation has a high dispersion, it means that the area of distribution is greater so that the active substances contained will be spread evenly and more effectively in producing a therapeutic effect. Good gel spreadability is 5-7 cm. The results of the evaluation of the average dispersion of hair dye preparations from beetroot extract with variations in weights obtained different values, namely at F1, F2, F3 and F4 the dispersion value was below 5 and theSD was 0.1387. The requirements for the spreadability test for topical preparations are around 5-7, which means the value obtained from the results of the spreadability test for hair dye cream preparations does not meet the dispersibility requirements (Ulaen et al., 2012).

Freeze thaw test was carried out to see the physical stability of the cream after being stored for one week at different temperatures, namely 4°C and 25°C. The temperature range for freezing conditions is >15°C, for cool conditions it is 15-25°C, for cold temperatures it is 2-8°C and for cool box temperatures 8-15°C. Storage was carried out in six cycles and one cycle lasted for one day at each temperature. This needs to be done to see the stability of the cream temperature during storage. Emulsion systems at high temperatures can cause an increase in the kinetic energy of the droplets of the dispersion phase so that it is easier to combine and an increase in the size of the globule diameter. While at cold temperatures the solubility of the emulsifier in the oil phase and in the waterphase will decrease so that the effectiveness of the emulsifier to coat the globules is reduced (Lachman et al., 1994). The evaluation results in this test showed a color change in the six cycles of the four formulas that have been prepared. for. The color changed more and more during storage time, this indicates that the dye contained in the beetroot did not last during storage. Freeze thaw test results can be seen in Figure 1 and Figure 2.



Figure 1. Preparation of hair coloring cream on the first day



Figure 2. Preparation of hair coloring cream on the last day

Description:

1. Preparation of hair dye cream at cold temperature.
2. Preparation of hair dye cream at hot temperature.

Test the effectiveness of hair coloring tested on black hair. This evaluation was conducted to determine the presence or absence of color produced by beetroot extract (*Beta vulgaris L.*). After testing the effectiveness of coloring hair dye preparations for six days of storage with concentrations of 30%, 40% and 50% produced different colors, namely on the first day of preparations F2, F3 and F4 with concentrations of 30%, 40% and 50% respectively, there was a change in color, namely reddish black, while on the third and fifth day all hair dye preparations showed no color change. This is because the formula for hair coloring cream from beetroot contains betacyanin which is very sensitive to several factors, while the factors that affect the stability of betacyanin compounds are temperature, light and oxygen (Herbach, et.al., 2006). The addition of ascorbic acid (vitamin C) to cream preparations as an antioxidant that were able to slow down or prevent the oxidation process also does not affect

the resulting color change. The results of the hair dye effectiveness test can be seen in Figure 3.

The color stability test for washing hair dye cream preparations from beetroot was carried out to see changes in hair color after washing. The results of this evaluation indicate that at F1, F2, F3, and F4 there is no color change, which means that washing does not affect the color change formed. The results of the test of the effectiveness of hair dye on washing can be seen in Figure 4.

Color stability test against sunlight preparation of hair dye cream from beetroot was carried out to see changes in hair color after being exposed to the sun. The results of the evaluation on the first day showed that there was no change in F1, while F2, F3, and F4 there was a change from reddish black to blonde black color. While on the third and fifth day on F1, F2, F3 and F4 there was no color change. This is because the preparation of hair dye from beetroot is easily oxidized when exposed to sunlight so that the hair color will be lighter than the previous color. The results of the hair dye test against sunlight can be seen in Figure 5.

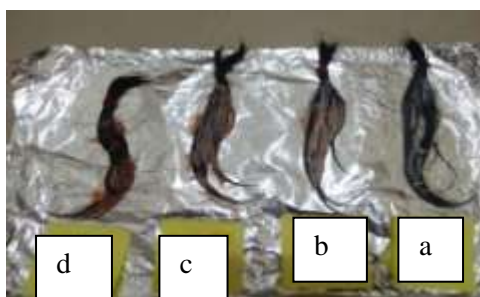


Figure 3. The effectiveness of hair dye cream

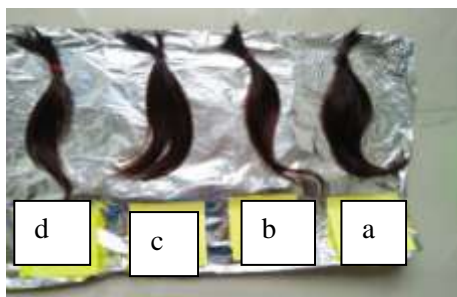


Figure 4. The effectiveness of hair dye cream on washing power

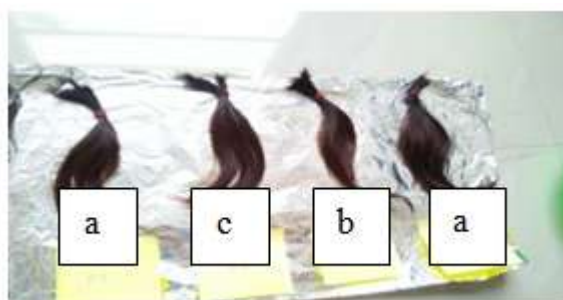


Figure 5. The effectiveness of hair dye cream against the sun

Description:

- (a) Cream preparation without beetroot
- (b) Formulation of cream preparations with the addition of 30% beetroot
- (c) Formulation of cream preparations with the addition of 40% beetroot
- (d) Formulation of cream preparations with the addition of 50% beetroot

IV. CONCLUSION

Based on the research that has been done on the stability test of the dye from beetroot (*Beta vulgaris L.*) in the preparation of hair dye cream, the following conclusions can be drawn, extract of beetroot (*Beta vulgaris L.*) can be formulated in the preparation of hair dye cream, dyes from beetroot extract cream (*Beta vulgaris L.*) with concentrations of 30%, 40% and 50% can be formulated as hair dye cream preparations but only in a short time, this is because the preparation of hair dye cream from beetroots does not has good stability to color due to color changes during the observation process and there is no color change in black hair.

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