

APPLICATION OF LAKES SYSTEM IN PREPARATION OF HAIR DYES POMADE CREAM OF FREEZE-DRIED RED DRAGON (*Hylocereus polyrhizus*) FRUIT PEEL JUICE AND ACUTE DERMAL IRRITATION

Malinda Prihantini¹✉, Muhammad Ikhsan², Hasan Abdul Karim³, Sindi Fadillah Rianis³, Sobari³, Faykar Rheza³

¹Department of Pharmaceutics and Pharmaceutical Technology, Faculty of Pharmacy, Universitas Wahid Hasyim, Semarang 50224, Indonesia

²Department of Pharmacology and Clinical Pharmacy, Faculty of Pharmacy, Universitas Wahid Hasyim, Semarang 50224, Indonesia

³ Undergraduate Program, Faculty of Pharmacy, Universitas Wahid Hasyim, Semarang 50224, Indonesia

✉ malindap@unwahas.ac.id

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ABSTRACT

Dyes used in market products are mostly synthetic, which can irritate the skin and in the long term increase the risk of skin cancer. The peel of dragon fruit contains the red-violet pigment Betacyanin, which is potential as hair dyes but extremely sensitive to light and oxygen. The lakes system is the option to overcome the problem and increase the stability of pigments. The objective of this study was to learn about the use of the lakes system in the preparation of hair dyes pomade cream of freeze-dried red dragon fruit peel juice and its irritation aspect on skin. The lakes system was made with a 50% red dragon fruit peel juice, mixed with 5% alucol, then dried with 45% aerosil. Pomade cream was prepared by mixing oil phase and water phase at a temperature of ± 60 °C with continuous mixing in 600 rpm for 10 minutes. Formula III is the bases without freeze-dried red dragon fruit peel juice. The non-lakes system of freeze-dried red dragon fruit peel juice was added to the bases for the FII, while the lakes system was added for the FI. Hair dyes pomade cream of red dragon fruit peel juice prepared in the lakes system has superior properties to the formula prepared without the lakes system. It demonstrated better homogeneity and softer texture, in addition acceptable skin pH range. The preparation do not cause skin irritation. The lakes system is suitable for the preparation of natural hair dye cream and does not irritate the skin.

Keywords: Complex; Dyes; Lakes; Pomade; Red-dragon

1. INTRODUCTION

Hair dyes preparations are cosmetics to color hair, either to restore the original hair color or to change into other colors. Hair dyeing preparation Pomade cream is a new type of temporary hair dye that can be applied directly to the hair (Auliasari et al., 2018). The dyes used in market products are mostly synthetic, such as rhodamine B, which can irritate the skin and, in the long run, increase the risk of cancer and liver damage (Syakri, 2017). Natural dyes, which are safer and have fewer side effects than synthetic dyes, may be the solution to this problem (Pardede et al., 2008). Dragon fruit peel contains pigment and can therefore be used as a natural dye. It is also commonly discarded as waste, despite accounting for nearly 30-35% of total fruit weight (Saati, 2011). Dragon fruit peel contains pigments anthocyanin and betacyanin, which can be used to create dyes (Sandy et al., 2021; Harjanti, 2016).

However, the pigment in dragon fruit peel is a type of phenolic compound that is extremely sensitive to light and oxygen. As a result, the dye's quality is deteriorating day by day (Citramukti, 2008). The lakes system can improve color pigment stability by forming a complex between reactive functional groups in pigments with hydrophobic substrates. It was created by absorbing pigments onto water-insoluble substrates like hydrated aluminum (alucol). The complex formed by the pigment and Al^{3+} from hydrated aluminum protects the pigment from light, chemicals, and heat (Cahyadi, 2006). Eventually, research will be conducted on the use of the lakes system in the preparation of hair dyes pomade cream of red dragon (*Hylocereus polyrhizus*) fruit peel juice and its irritation aspect on skin.

2. METHODS

2.1. Plant Determination

Plants are determined by matching the morphology of entire plant parts such as roots, stems, and fruits to a key of determination that refers to the literature (Backer, 1965). Plant determination was performed at the Ecology and Biosystematics Laboratory, Department of Biology, Faculty of Science and Mathematics, Diponegoro University, Semarang, Indonesia.

2.2. Freeze-Dried Red Dragon Fruit Peel Juice Preparation

Various sources cited the preparation of red dragon fruit peel juice, which is red dragon fruit peel juiced to separate the juice from the pulp. The juice was freeze-dried (Christ Alpha 1-2 LDplus), crushed, and sieved to achieve a small uniform particle size (Simanjuntak et al., 2014) (Sultan, 2017) (Husein & Lestari, 2019). The presence of secondary metabolite compounds of Alkaloids, Phenols, Flavonoids, Quinones, Saponins, and Tannins was determined using phytochemical screening on freeze-dried red dragon fruit peel juice (Kemenkes RI, 2011).

2.3. Lakes System of Freeze-Dried Red Dragon Fruit Peel Juice Preparation

The lake system is formed by pigment absorption on the hydrophobic substrate, which was created by combining 50% freeze-dried red dragon fruit peel juice with 5% alucol as the hydrophobic substrate. The mixture was then absorbed with 45% aerosil, resulting in the dried lakes. Sultan's research identified those compositions as optimal, with moisture content below 10% (Sultan, 2017).

2.4. Hair Dyes Pomade Cream of Freeze-Dried Red Dragon Fruit Peel Juice Formulation

Cream formula refers to (Husein & Lestari, 2019) with modification. Cream pomade is created by gently combining an oil phase mixture of 7.5 g of cera alba; 128.75 g Vaseline album; and 10 g of Span to an aqueous phase mixture containing 15 g of Tween 80; 75 g propylene glycol; 1.25 g methyl paraben; and aquadest at a temperature of $\pm 60^{\circ}C$. For 10 minutes, a Maspion-MT1140 mixer at 600 rpm was used to mix until a homogeneous cream base was formed. Formula III was the cream bases that did not contain freeze-dried red dragon fruit peel juice as a hair coloring agent. While in formula II (FII), an amount of 7.5 g non-lakes of freeze-dried red dragon fruit peel juice was geometrically added to the cream bases. On behalf, Formula I created by adding an amount of 7.5 g lakes system of freeze-dried red dragon fruit peel juice into the cream bases. Each formula was triple replicated and tested for its characteristics, which included:

2.4.1. Organoleptic

Conducted by visually observing the texture, color, and smell of cream preparations using the five human senses (Azkiya et al., 2017)

2.4.2. Homogeneity

The cream is shed transparently to the object-glass and its homogeneity is visually observed, with the parameters being free of coarse aggregate (Azkiya et al., 2017).

2.4.3. pH

A pH meter (HANNA HI8314-HI1612D) was employed to test the pH of 0.5 g of sample dissolved in 50 mL of aquadest. The pH meter was calibrated before measuring the sample with a standard buffer solution of pH 4, 7, and 10 (Kemenkes RI, 2020).

2.4.4. Dispersibility

A total of 0.5 g of the sample was placed on a glass surface above millimeter paper, then covered with another transparent glass for 1 minute and left. A weight of 50, 100, and 200 g was continuously added to the glass, and the diameter of the formed spread was measured (Saryanti et al., 2019).

2.4.5. Adhesion

A 0.5 g sample was placed on a glass object and then covered with another glass object for the test. For 5 minutes, a 500 g weight was placed above. A weight of 80 g is released to pull the bottom glass object, and the time it takes for the two glass objects to come off is recorded (Saryanti et al., 2019).

2.4.6. Viscosity

The viscosity of the cream was measured using a viscometer (RION VT-06 rotor no 2), which was placed in the center of the cream container. Keep an eye on the visibility needle. Once stable, the number displayed with the unit decipascal-seconds (dpas) was read (Mardikasari et al., 2020)

2.5. Hair Dyes Coloring Evaluation

Some shampoo-washed hair is dyed with a hair dye formula, then allowed to stand for 40 minutes and the color formed is observed. The coloring stability against washing and sunlight is then evaluated (Zaky et al., 2020).

2.6. Acute Dermal Irritation Test

On healthy male albino rabbits weighing 2.5-3.5 kg, a skin irritation test was performed. The experiment began with a preliminary test on one rabbit, with observations made at the third, sixty-first, and 240th minutes. If no irritation was observed, the procedure was repeated with the addition of two rabbits. Observations were administered after 24, 48, and 72 hours. The irritation score was calculated by observing the irritation parameters, which included the presence of erythema and edema (BPOM, 2020 & OECD, 2015).

3. RESULTS AND DISCUSSION

3.1. Plant Determination Result

The results of plant determination demonstrated that the plants employed were classified as Kingdom Plantae, Subkingdom Tracheobionta, Super Division Spermatophyta, Division Magnoliophyta (seed plants), Class Magnoliopsida (Dicotyledonae), Order Cactales, Family Cactaceae. Genus *Hylocereus*, Species *Hylocereus polyrhizus* (FAC Weber) Britton & Rose, with the local name Red Dragon Fruit.

3.2. Freeze-Dried Red Dragon Fruit Peel Juice

The freeze-dried red dragon fruit peel juice was described as red fines flakes as shown in **Figure 1**. The freeze-dried red dragon fruit peel juice yield was 2.28% of the fresh red dragon fruit peel weight. It is possible that this is due to the high-water content in the peel of fresh red dragon fruit. Red dragon fruit peel can be extracted using water or in conjunction with organic solvents such as methanol or ethanol (Sultan, 2017). The dragon fruit's red-purple hue is generated by betacyanin pigment, while the yellow tinge is caused by betaxanthin pigment. Both belong to the betalain pigment family. Several studies have shown that betacyanin pigments are more stable in aqueous solvents than in water-ethanol solvent combinations (Altamirano, 1993; Castellar et al.,

2006). This is thought to be due to the degradation of betacyanin pigments due to single or multiple decarboxylation mechanisms (Wybraniec et al., 2001).

The phytochemical screening revealed that alkaloids and flavonoids were present. It yielded the same results as phytochemical screening on super red dragon fruit peel extracted with water and dried using the freeze-drying method (Sultan, 2017). The morphology of the fruit distinguishes red dragon fruit used in this study from super red dragon fruit used in previous studies. The red dragon fruit is more oval and has dark red flesh, whereas the super red dragon fruit is round and has purple flesh (Le Bellec et al., 2006). Because betalains are a class of phenolic compounds with a hydroxyl group in their structure, which can be in the form of alkaloids or flavonoids, the results of this phytochemical screening indicate the possibility of betalain group pigments being present in the juice (Azeredo, 2009).



Figure 1. Freeze-dried red dragon fruit peel juice

3.3. Lakes System of Freeze-Dried Red Dragon Fruit Peel Juice

The lakes system has a morphology of dark red dry granules with uniform, homogeneous, and spherical particles. **Figure 2** depicts the interaction mechanism between the color pigment and the substrate at the C=O and -OH groups. Complexes between the C=O and C=O groups will form in mineral salts such as Al from alucol. A color pigment's -OH. Because the functional groups that have the potential to cause instability are used in complex formation interactions, the complex formed between the color pigment and the substrate in the lakes system can increase the color pigment's stability against light, chemistry, and heat (Kirby, 2011; Wongwad et al., 2012).

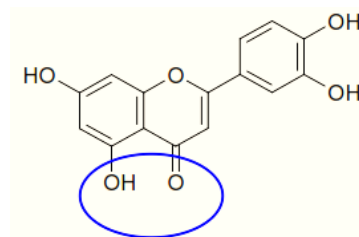


Figure 2. The position of the coordination bond between the substrate and pigment (Kirby, 2011)

3.4. Hair Dyes Pomade Cream of Freeze-Dried Red Dragon Fruit Peel Juice

The result of physical characteristics evaluation of hair dyes pomade cream of freeze-dried red dragon fruit peel juice was described below.

3.4.1. Organoleptic

Hair dyes pomade cream with red dragon fruit peel juice at FI and FII had a dark red color, was semi-solid, and smelled like dragon fruit, while FIII as bases had a milky white color. It was depicted in **Figure 3**. FII has a rougher texture than FI and FIII. It was most likely caused by the

aggregation of red dragon fruit peel juice particles that had not yet been distributed uniformly in the bases. In contrast to FII, the particles in FI which the red dragon fruit peel juice was in the form of the lakes system were coated with alucol, which makes the surface more hydrophobic and thus helps to reduce aggregation.



Figure 3. Pomade cream of: (a) FI (lakes of freeze-dried red dragon fruit peel juice); (b) FII (non-lakes of freeze-dried red dragon fruit peel juice) and (c) FIII (cream bases without freeze-dried red dragon fruit peel juice)

3.4.2. Homogeneity

The homogeneity of FII was lower than that of FI and FIII, most likely because the red dragon fruit peel juice could not be dispersed homogeneously in the cream bases as well as the lakes system in FI. It was related to the organoleptic results. Because it is associated with drug dose at each use, homogeneity has an impact on therapy effectiveness. If the preparation is homogeneous, the active substance content is assumed to be constant at each intake (Roosevelt et al., 2019). In the context of cosmetic uses, it influences the acceptability of the consumer.

3.4.3. pH

Figure 4 depicts the pH values for the three formulas. The ANOVA testing revealed that it was statistically different in each formula with a significance of 0.05. Due to the alkaline alucol used in the lakes system, the pH values in FI are higher than in FII and FIII, potentially increasing the pH of the product. The pH of alucol, also recognized as colloidal aluminum hydroxide, is 8-9. The pH range that the skin can tolerate is 4.5-6.5. A preparation that is too acidic and far from the pH of the skin will most likely irritate it, whereas an alkaline preparation will build the skin's scalp (Siva & Afriadi, 2019). The statistical analysis revealed that changing the lakes system affects the pH of the hair dyes pomade cream.

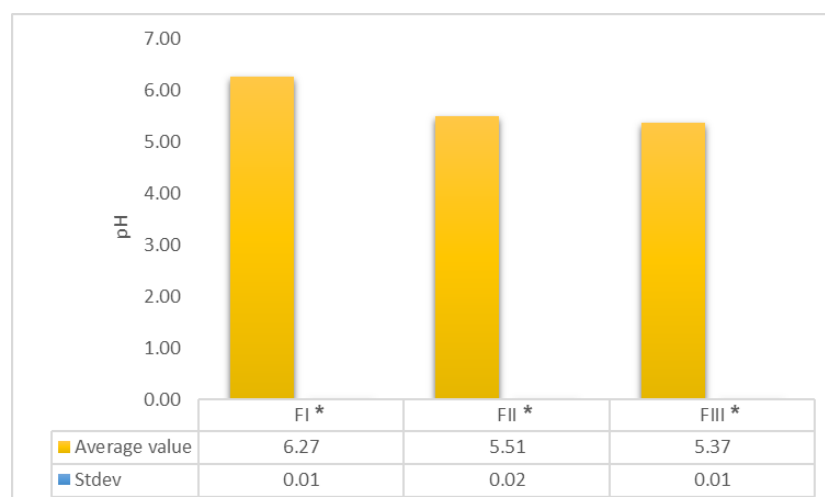


Figure 4. pH of pomade cream

3.4.4. Dispersibility

Dispersibility is inversely proportional to the value of viscosity (Zulfa & Mufrod, 2018). **Figure 5** depicts the dispersion results from the three formulas. It was discovered that the dispersion value is inversely proportional to the viscosity, with FI having the highest dispersion and FIII having the lowest. A statistical analysis revealed that each formula differed significantly from the others. It demonstrated that the laves system was given different physical properties to the hair dyes pomade cream of red dragon fruit peel juice. According to (Rasydy et al., 2021), the 5-7 cm spread demonstrates a semi-solid consistency that is very comfortable to use. The dispersibility of the three formulas is less than 5, but they are still acceptable because the characteristics of cosmetic preparations are not strict.

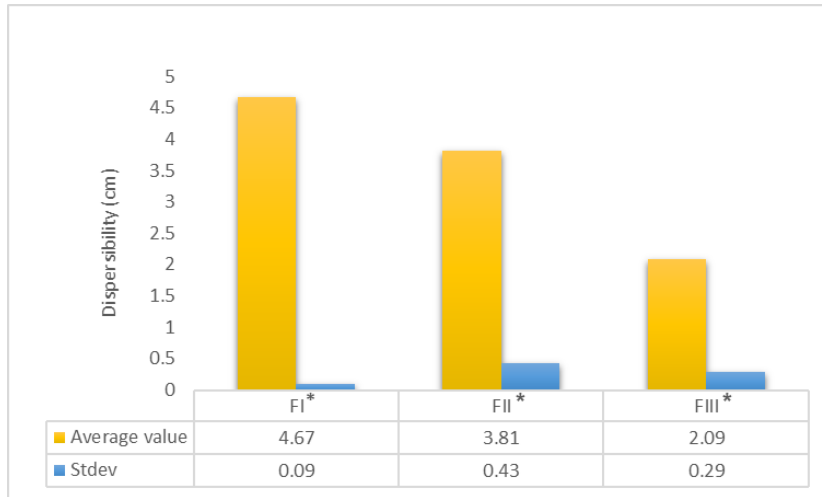


Figure 5. Dispersibility of pomade cream

3.4.5. Adhesion

The adhesion test is related to the time needed for the preparation to contact with the surface of the skin and cause physiological effects as well as related to the comfort of the preparation at this time applied (Marviani & Rochman, 2021). **Figure 6** depicts the results of the adhesion. FIII has the highest value, while FII has the lowest. It contradicts the dispersibility value, where the dispersion of FIII was lowest. It could be related to the FII homogeneity problem caused by aggregation of red dragon fruit peel juice that was not dispersed homogeneously. While in FI, the laves system aids in particle aggregation reduction by encapsulating the red dragon fruit peel juice with alucol, which is hydrophobic in nature, causing the particle's surface to become more hydrophobic.

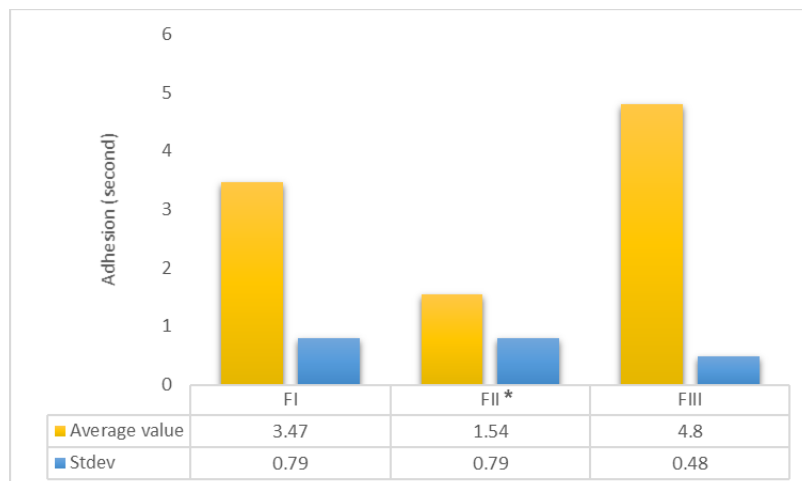


Figure 6. Adhesion of pomade cream

Statistical analysis revealed that the research treatment provided different adhesion characteristics to the hair dyes pomade cream of red dragon fruit peel juice, as demonstrated by the sig <0.05. Further statistical analysis was performed to determine whether each formula differed significantly. The results illustrated that FI (lakes system) differed significantly from FII (non-lakes system) but not from FIII (cream mass) (sig. 0.128). Furthermore, FIII demonstrated statistically significant differences to FII (sig. 0.003) but not to FI. The FII, on the other hand, demonstrated both significant differences with F1 and FII, with significant values in a row of 0.035 and 0.03.

3.4.6. Viscosity

Viscosity, spreadability, and adhesion are all characteristics of preparations that can be correlated. Viscosity is sometimes proportional to adhesion and inversely proportional to dispersion. According to the data in [Figure 7](#), statistically, each formula produced a significant difference. Preparations containing the active ingredients of red dragon fruit peel juice, whether in lakes or non-lakes systems, tend to decrease viscosity, with FIII showing the highest and FI showing the lowest. It is most likely due to an increase in FI hydrophobicity caused by the impact of alucol used in the lake system, which creates hydrophobic boundaries on the surface of particles. Hydrophobicity and hydrophilicity affect viscosity as a result of dissolved gas strongly adsorbed on the surface of the particles ([Sendner et al., 2009](#)).

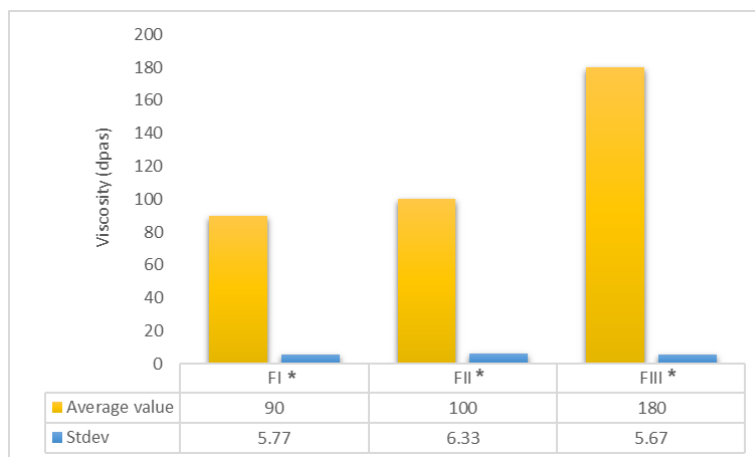


Figure 7. The viscosity of pomade cream

3.5. Hair Dyes Coloring Evaluation

[Figure 8](#) depicts the outcome of the hair dye coloring evaluation. The figure illustrated that when exposed to direct sunlight, the color intensity of the hair dye pomade cream of red dragon fruit peel juice decreased and turned brown. Light, air, and temperature all contribute to the degradation of betaxanthin pigments ([Sari, 2018](#)). The degradation rate of betalain increased by 14.6% in air and 15.6% in light, resulting in a decrease in color intensity ([Elbe et al., 1974](#)).



Figure 8. Hair bleached condition after application of F1 (lakes), F2 (non-lakes), and F3 (only base) after a) 40 minutes observation, b) exposure of direct sunlight, and c) washing.

Furthermore, testing was also administered on washing. Water can be used to cleanly remove all three formulas. It suited the study's end goal of being a temporary hair dye. Furthermore, this can meet the needs of consumers who are always dynamic and want to change hair colors without worrying about hair damage.

3.6. Acute Dermal Irritation Test

There was no irritation on the rabbit's skin (rabbit A) based on the preliminary test results at the 3rd, 60th, and 240th minutes, as indicated by an irritation score of 0. As a result, observations were continued with two more rabbits (rabbit B and C) for 24, 48, and 72 hours. **Figure 9** depicts the results, which show that there was no irritation of the rabbit skin in the form of erythema or edema.

The consumption of temporary hair dye cream is less than 24 hours, and the testing results on the rabbit's back skin did not irritate after 24, 48, and 72 hours. Furthermore, there was no irritation or other skin damage on the 14th day of observation. The objective of this observation is to identify the possibility of corrosive and irreversible skin damage (BPOM, 2020).



Figure 9. Rabbit's back skin after the application of hair dye pomade cream preparation in: (a) rabbit A; (b) rabbit B and (c) rabbit C (K = control without treatment; NL = nonlakes system; L= lakes system; B = only base without red dragon fruit peel juice)

4. CONCLUSION

Hair dyes pomade cream in the lakes system have better characteristics than the formula prepared without the lakes system. It showed better homogeneity, softer texture, acceptable skin pH range, viscosity, dispersibility, and adhesion are consumer-acceptable than the formula in non-lakes system. The preparation can color bleached hair and can be removed when washed with water. Preparations do not irritate the skin.

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6. CONFLICT OF INTEREST

All authors declare no conflict of interest.

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