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Formulation of Hairspray from Cherry Leaves (*Muntingia calabura* L.) with Antimicrobial Properties

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Abstract

Introduction: Indonesia is a tropical country with high temperatures and humidity. Poor personal hygiene can trigger bacteria to grow and multiply. According to research, bacteria that are usually found in skin and hair including *Staphylococcus aureus* (37%), followed by *Escherichia coli* (17%). Use of antibacterial hairspray is expected to inhibit the growth of pathogenic bacteria on the surface of the scalp and hair. Using natural ingredients, i.e., cherry (*Muntingia calabura* L.) leaves, in the form of antibacterial hairspray preparations, is expected not to irritate the scalp, be more eco-friendly, and the raw materials are easily obtained.

Methods: The preparation of cherry (*Muntingia calabura* L.) leaves extract was carried out by maceration method with 70% ethanol (simplisia : solvent = 1:10). Furthermore, the extract was then evaluated for phytochemical content, formulation optimization, and antibacterial activity. Afterward, formulation and preparation, and lastly, its evaluation in the form of an organoleptic test, pH test, viscosity test, homogeneity test, spreadability test, adhesion test, and product stability test.

Results: The results of phytochemical screening of flavonoid compounds and tannins from cherry (*Muntingia calabura* L.) leaves extract resulted in a positive reaction. The minimum inhibitory level of cherry (*Muntingia calabura* L.) leaves extract against *Staphylococcus aureus* and *Escherichia coli* bacteria was 40 mg/ml. Cherry (*Muntingia calabura* L.) leaves extract was then formulated into hairspray dosage forms with different concentrations of extract in Formula A (0.1%), Formula B (0.3%), and Formula C (0.5%).

Conclusions: The hairspray formula that had the best stability after passing the freeze-thaw cycling test and physical evaluation of the preparation during storage for 28 days was Formula C. The parameters of the observed hairspray were organoleptic, pH, homogeneity, viscosity, spreadability, and adhesion.

Keywords: Hairspray - Cherry Leaves – Stability - Antibacterial

INTRODUCTION

Indonesia is a tropical country with high temperature and humidity. Personal hygiene must be improved in these environments. A lack of proper sanitation often causes problems with one's hair, especially in children.[1] Good personal hygiene is expected to improve health status, increase self-confidence, and create beauty. Poor personal hygiene can trigger bacteria to grow and multiply.[2] The use of antibacterial hairspray is

expected to inhibit the growth of pathogenic bacteria on the surface of the scalp and hair.[3]

One of the plants that can be used in the preparation of antibacterial hairspray is the leaves of the cherry (*Muntingia calabura* L.) plant. This plant grows in the tropics, is easily found and obtained in Indonesia. Cherry (*Muntingia calabura* L.) plants with chemical content such as tannins and flavonoids are known to have antibacterial activity.[4,5] The types of flavonoids in cherry (*Muntingia calabura* L.) leaves include flavonones, flavans, flavonols, flavones, and biflavans which are bound to sugar. The flavonoid content will interact with bacterial DNA, so that the permeability of bacterial cell will be destroyed. The tannin content at small concentrations can inhibit the development of bacteria and tannins at large concentrations can coagulate proplasm, shrink cells and damage the permeability of bacteria.[6]

Natural materials have recently been widely used since they are considered to have the same properties as synthetic materials with less risk of side effects, more eco-friendly, easily obtained raw materials, and more economical prices. The antibacterial hairspray formulation will be made from natural ingredients that do not irritate the scalp. The antioxidant content of cherry (*Muntingia calabura* L.) leaves is also known to nourish the hair.[7] Previous research has also examined the antibacterial activity of cherry (*Muntingia calabura* L.) leaves water extract with minimum inhibitory and killing concentrations against *Staphyloccous aureus* and *Escherichia coli* bacteria are >0.5 mg/mL.[4] However, there has been no research and development of antibacterial hairspray solution dosage forms from cherry (*Muntingia calabura* L.) leaves extract. The designed dosage form is a solution dosage form since the active flavonoid and tannin compounds are soluble in water. Solution is a mixture with a homogeneous system, consisting of solvent and solute. This dosage form will also be combined with other additives or excipients such as solvents, surfactants, and preservatives that may increase the stability of the preparation.[8] The purpose of this research is expected to inhibit the growth of pathogenic bacteria on the surface of scalp and hair. Hairspray dosage form is expected convenience to used and natural ingredients is tended not to irritate the scalp, be more eco-friendly, and easily obtained.

MATERIAL AND METHODS

Study Design

This study was an experimental research design with the formula determined randomly based on the acceptance range of the pharmacopeia. The research was conducted in the pharmacy department's Lab at Atma Jaya Catholic University of Indonesia from February until July 2022.

Material

Cherry (*Muntingia calabura* L.) leaves were self-collected from a residential area in Karang Tengah, Tangerang, Indonesia. Then the collected sample was sent to the Badan Riset dan Inovasi Nasional (BRIN) for the determination process to ensure that the sample was *Muntingia calabura* L. species. *Staphylococcus aureus* dan *Escherichia coli* ATCC was obtained from the pharmacy department collection. Etanol technical grade, propilen glycol technical grade, PEG-60 hydrogenated castor oil technical grade, steartrimonium chloride technical grade, natrium metabisulfite technical grade, blanc disc OXOID T0998B, disc antibiotic chloramphenicol 30 mcg. The instruments used in this study are Oven Memmert, Rotary Evaporator HEIDOLPH, Thermo Scientific HAAKE Viscotester D, and adhesion tester.

Method

Following the collection, Cherry leaves were washed, air aerated for 3 hours, dried using a dehydrator at 50°C for 4 hours, then blended until powder. The powder was macerated using 70% ethanol for 48 hours, then concentrated using a rotary evaporator at 55°C, 70 rpm for 4 hours. The extract then underwent phytochemical screening to optimize formulations and evaluate antibacterial activity. The macrodilution method was used to test for antibacterial activity.

Formulation

The extract was formulated in three formulations with different concentrations (Table 1). Afterward, the product was evaluated for several parameters: organoleptically, pH, viscosity, homogeneity, spreadability, adhesion, and stability.

Table 1. Hairspray Formulation

No	Ingredients	Formula concentration (%)			Function
		A	B	C	
1	Extract	0.1	0.3	0.5	Active ingredient
2	Propylene glycol	20	20	20	Co-solvent
3	Hydrogenated PEG-40 castor oil	15	15	15	Co-solvent
4	Sodium metabisulfite	0.1	0.1	0.1	Antioxidant
5	Steartrimonium chloride	0.5	0.5	0.5	Preservative, anti-static agent
6	Aquades	Ad 100 mL			Solvent

Formula Evaluation

Organoleptic evaluation

The organoleptic evaluation includes a visual assessment of color, scent, form, and clarity every seven days for 28 days.[9]

Acidity measurement

The pH was measured every week for 28 days, with an expected range of 4.5-6.5.

Viscosity test

The viscosity is measured using a viscometer with spindle R2 and a 200 rpm point measurement. The expected viscosity value for a slightly viscous solution is 100 cps.

Homogeneity test

The sample was placed on the object glass for the homogeneity test. There should not be non-dispersed particles present in a homogenous preparation.

Spreadability test

The formulation's spreadability was assessed by applying it to transparent mica plastic. After one minute, the diameter of the preparation's distribution was measured.

Adhesion test

The adhesion test was performed by putting 0.5 gram of the preparation on the slide and covering it with the slide other. The glass is loaded with 1 kilogram for 3 minutes. The adhesion tester is then used to record the slide separation time. Forecast value exceeds 1 second.

Stability evaluation

A three-cycle spinning test was used to evaluate the product's stability. For one test session, the mixture is kept for 24 hours at a frigid temperature of approximately -10°C, then for 24 hours at room temperature, 24 hours at 40°C, and then for another 24 hours at room temperature.

RESULTS

According to BRIN, the self-collected leaves were identified as *Muntingia calabura* L. (appendix 1). A dehydrator-dried powder has a drying loss of 1.5% with a return yield of 42.43%. The phytochemical screening of cherry (*Muntingia calabura* L.) leaf extract for flavonoid compounds and tannins yielded a positive result. After evaluating for antibacterial activity, we found that the minimal inhibitory concentration of cherry leaves extract was about 40 mg/ml. This concentration was then used to determine the quantity of extract to put in the formula. Three formulas with different levels of extract concentration were made to see which one has the best stability. The formula resulted a liquid hairspray dosage form. The color of these formulas is slightly different, as shown in Figure 1. These products also maintain color and scent after 28 days of storage at room temperature.

Preparation of hairspray dosage form with three different concentrations of cherry (*Muntingia calabura* L.) leaves extract, i.e., Formula A (0.1%), Formula B (0.3%), and Formula C (0.5%). The color of the dosage

forms produced were different due to the different extract concentrations, i.e. Formula A was brownish yellow, Formula B was light brown and Formula C was dark brown. Hairspray dosage forms did not change color or smell during 28 days of storage at room temperature 28°C±2°C with pH = 5.

Figure 1. The hairspray dosage form

Preparation of hairspray dosage form with three different concentrations of cherry (*Muntingia calabura* L.) leaves extract, i.e. Formula A (0.1%), Formula B (0.3%), and Formula C (0.5%). The color of the dosage forms produced was different due to the different extract concentrations, i.e. Formula A was brownish yellow, Formula B was light brown and Formula C was dark brown. Hairspray dosage forms did not change color or smell during 28 days of storage at room temperature with pH = 5.

Viscosity test results for 28 days with three replications were obtained for Formula A (37.04-38.26 cps), Formula B (37.10-38.28 cps), and Formula C (37.43-38.02 cps). Furthermore, in the homogeneity test, the dosage forms were stated as homogeneous solutions, and there were no insoluble particles, even after being left for 28 days at room temperature.

The results of the spreadability test on mica plastic with a spraying distance of 5 cm for 28 days for Formula A are 5.98-6.03 cm, Formula B are 6.76-6.90 cm and Formula C are 7.81-7.94 cm. The results of the adhesive test for 28 days with a load of 1 kg for 5 minutes resulted in the range of adhesion for Formula A are 1.28-1.31 seconds, Formula B are 1.42-1.45 seconds, and Formula C are 1.39-1.42 seconds.

Table 2. Parameter of the hairspray product

Parameter	**Formula A**	**Formula B**	**Formula C**
pH	5	5	5
Viscosity (cps)	37.04 – 38.26	37.10 – 38.28	37.43 - 38.02
Homogeneity (Presence of non-dispersed particle)	None	None	None
Spreadability (cm)	5.98 - 6.03	6.76 - 6.90	7.81 - 7.94
Adhesion (second)	1.28 – 1.31	1.42 – 1.45	1.39 – 1.42

Stability Test with Freeze-thaw Cycling test

The organoleptic test for 3 cycles produced the same color and odor as before the freeze-thaw cycling test. The color of Formula A is brownish yellow, Formula B is light brown, and Formula C is dark brown. The odor of the dosage form after the freeze-thaw cycling produces a distinctive smell of cherry (*Muntingia calabura* L.) leaves with a pH of 5. The results of viscosity test with a range of values for Formula A are 46.90-46.97 cps, Formula B are 48.42-48.52 cps, and Formula C are 59.96-50.04 cps. The homogeneity test on the three preparations after the freeze-thaw cycling test resulted in a homogeneous and soluble preparation. The spreadability test resulted in the spreadability of Formula A are 4.5-4.6 cm, Formula B are 5-5.1 cm, and Formula C are 5.7-6 cm. Meanwhile, the adhesive test produced a range of stickiness of Formula A are 1.3-1.35 seconds, Formula B are 1.45-1.48 seconds, and Formula C are 1.95-2.11 seconds. For stability evaluation, the results are shown in Table 3.

Table 3. Product parameter after stability test

Parameter	Formula A	Formula B	Formula C
pH	5	5	5
Viscosity (cps)	46.90 – 46.97	48.42 – 48.51	49.96 – 50.04
Homogenity (Presence of non-dispersed particle)	None	None	None
Spreadability (cm)	4.5 – 4.6	5 – 5.1	5.7 – 6
Adhesion (second)	1.3 – 1,35	1.45 – 1.48	1.95 – 2.11

DISCUSSION

A desired hairspray product is a leave-on product that does not require water rinsing after use the product is also a hair mist liquid that spreads easily on the surface when sprayed. The excipients used to increase the extract's solubility are propylene glycol and co-solvent PEG-60 hydrogenated castor oil.[10] PEG-40 hydrogenated castor oil is a surfactant nonionic that can mix oil with water to clean the scalp and hair dirt. PEG-40 hydrogenated castor oil is also included in the gentle class cleansing agent.[10,11] The amount of PEG-40 hydrogenated castor oil in hairspray preparations is 15%. At this concentration, hairspray preparations that were cloudy before can become apparent because PEG-40 hydrogenated castor oil is also used in the preparation as an emulsifier.[11] The preparation's consistency was increased by using 30% propylene glycol. Sodium metabisulfite functions as an antioxidant and inhibits the oxidation process from occurring in the preparation, thereby increasing its stability.[12] Steartrimonium chloride is a type of cationic surfactant. It has a positive charge

and can alter negative charges on the hair's surface. This cationic surfactant will bind directly to the negative charge on the surface of the hair and reduce frizz. Reduced frizz gives hair a smooth, light texture and makes it less sticky, so it's simpler to style with. Steartrimonium chloride does more than just make hair soft. It also has the capacity to function as a gentle cleaner, this cleanses hair without rinsing.[10]

Organoleptic evaluations of hairspray formulations over 28 days are intended to detect any potential for instability during storage at room temperature. The colour of the product depends on the extract concentration; the more extract present, the darker the liquid. During a 28-day monitoring period, there was no difference between formulations A, B, and C when it came to the liquid dose form (Fig. 1). Additionally, the product's smell did not produce an unwanted odor. Therefore, formulations A, B, and C of hairspray are stable for 28 days when stored at room temperature.

The product's acidity is important because it affects the absorption of the product into the epidermis. In addition, the acidity should be maintained within an acceptable range to prevent skin irritation. The range of skin pH balance values or acidity degrees is 4.0 to 6.5. If the pH value of the preparation is too high (alkaline), the skin may become callous; conversely, skin irritation may result if the pH value is too low (acidic).[13] All formulations result in products that are pH 5.0 acidic, stable for 28 days, and also after stability tests after freeze-thaw cycles.

The prepared hairspray dosage form is a leave-on dosage form that does not need to be cleaned with water after use. The solubility of the extract was increasing in solution using propylene glycol and PEG-60 hydrogenated castor oil co-solvents.[14] The concentration of this co-solvent in leave-on dosage forms is 0.0007-22%.[10,11] In this study, the concentration of hydrogenated castor oil used was 15%. Propylene glycol as a solvent or co-solvent in the range of 5-80% w/v.[12] In this study, a 30% concentration of propylene glycol was used and able to produce the desired viscosity of the dosage form. Sodium metabisulfite as an antioxidant that prevents oxidation processes in dosage forms, such as propylene glycol so that it is not easily oxidated at certain temperatures.[10] Steartrimonium chloride is a cationic surfactant that can clean residues on hair without the need for rinsing.[10] At low concentrations, this material can be used as a preservative in the dosage forms and maintain stability from external contamination.

The color of the cherry (*Muntingia calabura* L.) leaves extract affects the color produced by the dosage forms. The higher concentration of the extract, the browner color of the dosage form will be. The pH value of the dosage forms

during 28 days of storage is pH 5, while the range of pH balance values or skin acidity is 4.0-6.5.

The viscosity value decreased and increased every week for 28 days. However, the viscosity values of all formulas are less than 100 cps and this value is suitable for non-viscous solution dosage forms. The three dosage forms meet the viscosity requirements, and it appears that there is no significant difference in viscosity between Formula A, Formula B, and Formula C. The homogeneity test is carried out to see the distribution and solubility of active substance particles in the dosage forms so that it gives the same effect in each application.[15] Hairspray dosage forms of Formulas A, Formula B, and Formula C produce homogeneous dosage form when observed under a glass object.

Spreadability test of hairspray dosage forms aims to determine the distribution of particles evenly when being sprayed onto the hair.[16] When being compared to the requirements of hairspray gel, which is 5-7 cm.[16] Hairspray solution has a higher spreadability because the preparation used is a water-based preparation.[17] Adhesion test of the dosage forms aims to see the inherent ability of the dosage form and the ability to coat the hair and scalp so that the dosage form is functioning optimally. The test results of all dosage forms have an adhesion time of more than 1 second. Too high adhesion of the dosage form will make the dosage form difficult to be rinsed with water during shampooing, leaving new residues on the scalp and causing factors for the appearance of dandruff.[18] However, this cherry (*Muntingia calabura* L.) leaves extract hairspray dosage form is water-based and has a not too high adhesion that is 1.3-2.11 seconds so that it gives the impression of a light texture on the hair, and does not need to be cleaned with water (leave-on dosage form).

Stability test for hairspray dosage form by freeze-thaw cycling test method. This test was carried out to determine the product's response to the treatment given. Thus, it can be understood the appropriate product storage conditions and the length of time the dosage forms are safe to use. The given treatment was the freeze-thaw method with three different temperature points in each cycle. The test results for all formulas showed a homogeneous dosage form and there was no phase separation, had organoleptic, homogeneity and a stable pH value.[19,20] The results of the spreadability test decreased after freeze-thaw cycling test treatment. According to Mao et al., the higher the viscosity, the lower the spread rate of the solution.[19,20] With regard to the adhesion test, it has increased because the adhesion is directly in line with the viscosity, the higher viscosity value of the dosage forms, the higher possibility of the dosage forms to stick (longer

adhesion time) so that such increase may be influenced by an increase in viscosity.[19]

CONCLUSION

The formulation of hairspray from cherry leaf extract (*Muntingia calabura* L.) produces the desired product, a hair mist liquid that spreads smoothly on the hair when applied. Based on stability and physical evaluation tests, formula C is the most stable. Formula C consists of 0.5% extract, 20% propylene glycol, 15% PEG-40 hydrogenated castor oil, 0.1% sodium metabisulfite, 0.5% steartrimonium chloride, and aquadest. The limitation of this study is that the dosage form's antimicrobial activity has not yet been determined, whereas the extract has.

CONFLICT OF INTEREST

No conflict of interest found in this study.

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Appendix

DIREKTORAT PENGELOLAAN KOLEKSI ILMIAH
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Perihal : Hasil Identifikasi/Determinasi Tumbuhan

Yth.
Bpk./Ibu/Sdr(i). **Karmill Tri Nanda**

Bersama ini kami sampaikan hasil identifikasi/determinasi tumbuhan yang Saudara kirimkan ke "Herbarium Bogoriense", Direktorat Pengelolaan Koleksi Ilmiah BRIN Cibinong, adalah sebagai berikut :

No.	No. Kol.	Jenis	Suku
1.	Daun Kersen	*Muntingia calabura* L.	Muntingiaceae

Demikian, semoga berguna bagi Saudara.

Plt. Direktorat Pengelolaan Koleksi Ilmiah,
Badan Riset dan Inovasi Nasional

TT ELEKTRONIK

Dr. Ir. Hendro Wicaksono, M.Sc., Eng