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Formulation And Physical Stability Of Hair Tonic Ethanol Extract Of Katuk Leaves (Sauropus Androgynous (L.) Merr)

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ABSTRACT

Katuk leaves (Sauropus androgynus (L.) Merr) contain provitamin A and vitamin E, flavonoid, and ascorbic acid, which help hair growth. This study aims to determine the stability level of hair tonic preparations of ethanol extract from katuk leaves. The method used in this study is the cycling test method. This study made a hair tonic formulation with 2.5% ethanol extract of katuk leaves with a variation of 96% ethanol concentration of 35 ml and 10 ml. The stability parameters tested include organoleptic, pH, viscosity, specific gravity, and homogeneity. The pH test results range from 4.4 to 5.3; this pH value follows the Indonesian National Standard (SNI). The viscosity test ranged from 0.59-0.69; this pH value complies with the Indonesian National Standard (SNI). The specific gravity test results ranged from 1.02-1.04, and this particular gravity value was not the theory of specific gravity of hair tonic less than 1. The homogeneity test results showed that the hair tonic experienced precipitation and was not homogeneous. Furthermore, the hedonic test showed that the panellists preferred the negative control preparation, then the hair tonic preparation of katuk leaf ethanol extract with 96% ethanol variation of 60 ml and finally, the hair tonic preparation of katuk leaf ethanol extract with 96% ethanol variation of 35 ml.

Keywords: *Hair tonic, katuk leaves, hair tonic evaluation, hair growth*

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INTRODUCTION

Hair is one of the important anatomy of the human body; hair is the most effective scalp protector from sun exposure [1]. In addition, hair has an important role in the appearance of men and women, so having healthy hair can increase one's confidence [2]. Efforts made to avoid hair problems include using hair care. Cosmetic preparations that deal with hair problems, especially in overcoming hair loss, are hair tonics [2]. A cosmetic product known as hair *tonic* contains a combination of active substances and other additives that prevent hair loss, prevent dandruff and itch, facilitate blood flow, maintain a healthy scalp, and encourage hair growth [3]. Flavonoids have been recommended as hair follicle growth regulators in treating hair loss and *alopecia* [4]. Antioxidants contained in katuk leaves, such as flavonoids, can stimulate hair growth by causing muscle relaxation in the blood vessels around the hair follicles, thereby facilitating a constant blood supply with nutrients to the hair follicle cells. In addition, antioxidants can prevent unfavorable ultraviolet (UV) radiation on hair fibers [4].

METHODS

Extraction

The extraction process uses the maceration method, katuk leaf simplisia soaked using 70% ethanol solvent and allowed to stand for 3x24 hours while stirring periodically. The extraction results obtained are separated by filtration using filter paper. The remaining simplisia residue was macerated twice using 70% ethanol solvent with the same steps. The collected fiber is evaporated by distillation and concentrated using a rotary evaporator with a temperature of 50^0C until a thick extract is obtained.

Non-Specific Parameter Inspection of Extracts

1. Moisture content check

Weigh a 2 to 3 grams sample and then heat it in the oven at 105 ^{0}C for 5 hours. Repeat the process until you get a weight difference of less than 0.25% or a fixed weight.

2. Ash content check

Samples weighed as much as 2 to 3 grams and then heated in a furnace at 600^0C for 6 hours until the samples turned into ash, then weighed

Formulation of Hair Tonic

Hair tonic preparations are made in 3 formulations, namely, without adding extracts with variations in 96% ethanol concentration as much as 35 ml and 60 ml. Weighing the necessary ingredients, then dissolved methyl paraben and propyl paraben using propylene glycol until homogenous (solution 1); in different containers, dissolved sodium metabisulfite with aquadest until homogenous, then in the same container, mixed sodium metabisulfite solution with a thick extract solution that had previously been dissolved with 96% ethanol (solution 2). Solution 1 and 2 are mixed until homogenous, then add aquadest until 100 ml.

Table 1. Katuk leaf hair tonic formulation

Bahan	Konsentrasi (%)		
	Formula I	Formula II	Formula III
Ekstrak daun katuk	2,50 gram	2,50 gram	-
Etanol 96%	35 ml	60 ml	35ml
Metil paraben	0,1 gram	0,1 gram	0,1 gram
Propil paraben	0,01 gram	0,01 gram	0,01 gram
Propilen glikol	10 ml	10 ml	10 ml
Sodium metabisulfat	0,1 gram	0,1 gram	0,1 gram
Aquadest	Ad 100 ml	Ad 100 ml	Ad 100 ml

Hedonic Test

This test is carried out by panellists giving an assessment based on the level of liking by observing the organoleptic preparation, including the parameters of the shape, color and aroma of *katuk leaf hair tonic* practices on the organoleptic test form that has been provided [5].

RESULT AND DISCUSSION

The maceration extraction method was chosen because the working mechanism and tools used are simple. Besides that the maceration process is also not

heated so that the active substance content in natural materials will not be damaged and allows more compounds to be extracted during the process [6]. The filter solvent used in the extraction process is 70% ethanol solvent, 70% ethanol solvent selection because ethanol is polar, and the compounds to be extracted are also polar. Ethanol can attract more compounds compared to other organic solvents. Besides, ethanol has a low boiling point, which is 79^0C, so that in the concentration process, less heating will be used [2]. After the liquid extract is obtained, the next step is the concentration process. The concentration of the extract is carried out using the help of a rotary evaporator with a temperature of 50^0C; the concentration process aims to remove the remaining solvent to obtain a constant extract weight [7]**.**

Secondary metabolites contained in plants cannot be ascertained to always be in stable conditions; this can be influenced by several factors, one of which is the extraction process, which can affect the stability of secondary metabolite content in plants [8]. Therefore testing non-specific parameters needs to be carried out to determine the safety and physical stability of the extract [9].

Organoleptic testing is a test carried out using the help of the five senses, including the sense of sight and sense of smell, to determine the aroma, shape, and colour [9]. Based on the results of organoleptic observations (as shown in Table 2), it is known that the extract has a liquid form with a thick consistency, dense green colour, and distinctive katuk leaf aroma.

Table 2. Organoleptic test result

Formula	Evaluation
Hair tonic katuk leaf 96% ethanol with a concentration of 35 ml	The preparation is in liquid form, dark green in color and has a distinctive aroma of katuk leaves and is not homogeneous
Hair tonic katuk leaf 96% ethanol with a concentration of 60 ml	The preparation is in liquid form, dark green in color and has a distinctive aroma of katuk leaves and is not homogeneous
Negative control	The preparation is in liquid form, clear in color and has an aromatic or non-aromatic aroma

Water content testing is a test carried out using heating at a temperature of 105^0C for 30 minutes; the purpose of testing water content is to determine the percentage of water content in the extract after the drying process [9]. Based on the test results, the moisture content of katuk leaf extract was 9% w/w. This shows that the water content obtained is by the conditions determined by Indonesian herbal pharmacopoeia [10], which is less than 10%.

Ash content testing is a test carried out using heating at a temperature of 600^0C for 6 hours; the purpose of ash content testing is to determine the percentage of internal and external mineral content derived from the process of simplisia formation to the formation of extracts [9]. Based on the test results, the ash content value of katuk leaf extract was 7% w/w. This shows that the ash content obtained is by the conditions determined by Indonesian herbal pharmacopoeia [10], which is less than 12%.

Hair tonic preparations begin by dissolving methyl paraben and propyl paraben into propylene glycol until homogeneous. Methyl paraben and propyl paraben in this preparation serve as preservatives. The use of preservatives in hair tonic preparations is because *hair tonic* preparations have a large enough water content, so they can be a medium for microbial growth [10]. The combination of methyl paraben and propyl paraben as a preservative because the combination of the two produces a synergistic effect as antibacterial and antifungal so that the results provided will be more effective [11].

Propylene glycol used to dissolve methyl paraben and propyl paraben also increases the preparation's viscosity so that the contact time between the preparation and the skin is longer and the extract penetrates the scalp more [12]. The concentration of propylene glycol used in this preparation is 10%. Based on research by (Desriani *et al.*, 2018) states that the use of propylene glycol at concentrations of 10%, 15%, and 20% can produce good preparations and good viscosity [12].

In another container, sodium metabisulfate is dissolved using aquadest; sodium metabisulfite functions as an antioxidant in this preparation. The use of antioxidants in *hair tonic* preparations aims to prevent the oxidation process in

the preparation. In addition, antioxidants also can repair damaged hair cells, improve blood circulation and produce healthy hair tissue for hair growth [13]. After that, in the same container, mixed sodium metabisulfate solution with an extract that has been dissolved by 96% ethanol.

96% ethanol is a solvent used to dissolve thick katuk leaf extract,. 96% ethanol is a common basic ingredient commonly used in making *hair tonic preparations*, besides that 96% ethanol can also function as a penetration enhancer [12]. After all the solution is mixed until homogeneous, the next aquadest up to 100 ml is added.

A stability test is a test that aims to observe changes in shape in preparations during the storage period, including organoleptic parameters, homogeneity, pH, density, and viscosity [14]. One method used to speed up the evaluation of the stability of a preparation is to store the preparation at a temperature higher than normal temperature for several periods. This method is used to determine the preparation's shelf life with a cycle between two temperatures [15].

One of the stability testing methods is the *cycling test* method; this method is a simulation of temperature changes during the storage process. This method is carried out as many as 6 cycles by storing the preparation at a temperature of 4 0 C for 24 hours, then storing again at 40 0 C for 24 hours (counting one cycle). This method is a simulation of storage of preparations at room temperature for 2 years [14].

The results of the examination of organoleptic found that each formulation did not experience significant differences after the storage period was accelerated. *hair tonic* with the addition of extracts had a color like the color of the extract, which is dark green with a characteristic aroma of katuk leaves, but instability in homogen parameters was seen after the storage period was accelerated; it is characterized by the deposition of and clumped extract particles. At the same time, the dosage formula in the absence of the addition of extract has a clear color and has an aromatic and homogeneous aroma.

The results of pH testing found that after the accelerated storage period, the hair tonic decreased pH. The decrease in pH in *katuk leaf hair tonic* preparations can be caused by CO_2 that enters during the testing process. The presence of CO_2 that reacts with water can cause pH to become acidic; besides that, a decrease in pH can also be caused by the interaction of ingredients in the preparation and the influence of extreme temperatures from cold to hot [12]. However, in negative control preparations, the pH after the storage period is accelerated increases. However, the pH values obtained after *cycling tests* still meets SNI requirements, which is between 3.0 - 7.0, which does not cause skin irritation or scaly skin. So, it can be concluded that the pH of hair tonic preparations can be said to be good (as shown in figure 1) .

(a) (b) (c)

Figure 1. Organoleptic test results of hair tonic preparations (a) Hair tonic preparations of katuk leaves, variations of 96% ethanol, 35 ml (b) Hair tonic preparations of katuk leaves, variations of 96% ethanol, 60 ml (c) Negative control preparations

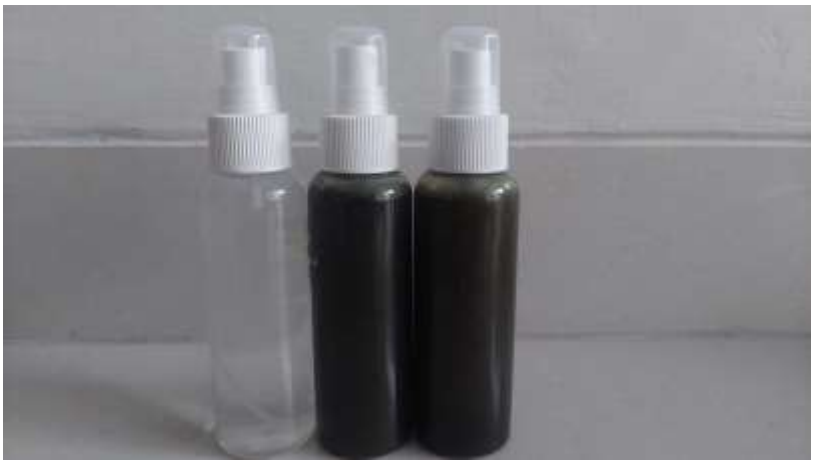

Figure 2. Katuk leaf hair tonic

The results of viscosity testing found that after the storage period is accelerated, the hair tonic decreases viscosity value, where the *hair tonic* preparation becomes diluted. This can be due to *katuk* leaf hair tonic preparations experiencing precipitation or not being entirely dissolved during the temperature change process during the storage period [15]. However, the range of viscosity values obtained before and after the *cycling test* still meets the SNI requirements of less than 5 cPs. If the viscosity value is high, it will trigger the growth of crusts on the scalp, which will cause dandruff. So, the viscosity of hair tonic preparations can be said to be good. Based on the results of the one-way-ANOVA analysis test on each preparation formula, it shows that each formula has experienced insignificant changes; this can be shown through a significant value of P = 0.759 (P>0.05).

Specific gravity testing is a test that aims to knowing the purity value of a substance [13]. Based on the results of observations, it showed that the katuk leaf hair tonic preparation in each formula after 12 days of accelerated storage experienced an increase in specific gravity as shown table 3. This can be caused by precipitation and evaporation [18]. However, the range of specific gravity values obtained after the cycling test does not comply with SNI requirements, namely less than 1 (specific gravity of water). Based on the results of *the one way-ANOVA* analysis test on each formula shows that each

formula has experienced insignificant changes, this can be shown through a significant value of P = 0.962 (*P*>0.05).

Table 3. Density test result

Formula	Evaluation	
	Before stability test	After stability test
Hair tonic katuk leaf 96% ethanol with a concentration of 35 ml	0.98	1.043
Hair tonic katuk leaf 96% ethanol with a concentration of 60 ml		1.039
Negative control		1.026

Based on the results of the hedonic test, it was found that the panelists preferred the negative control preparation formula, in terms of organoleptic, homogeneity, and also the aroma of the preparation. In the second place, panelists preferred 96% ethanol variation katuk leaf hair tonic as much as 60 ml, followed by 96% ethanol variation *katuk leaf hair* as much as 35 ml in terms of organoleptic, homogeneity and also dosage aroma.

Table 4. Viscosity test result

Formula	Evaluation of viscosity	
	Before stability test	After stability test
Hair tonic katuk leaf 96% ethanol with a concentration of 35 ml	0.73	0.59
Hair tonic katuk leaf 96% ethanol with a concentration of 60 ml	0.72	0.69
Negative control	0.73	0.67

Table 5. pH test result

Formula	Evaluation of viscosity	
	Before stability test	Before stability test
Hair tonic katuk leaf 96% ethanol with a concentration of 35 ml	6.0	5.1
Hair tonic katuk leaf 96% ethanol with a concentration of 60 ml	6.2	5.3
Negative control	3.7	4.4

CONCLUSION

The preparation of hair tonic ethanol extract of katuk leaves with variations of 96% ethanol concentration as much as 35 ml and 60 ml, showed instability in homogeneity, pH, and viscosity parameters. However, the values obtained were still within the range of SNI requirements. However, the specific weight value of hair tonic after the storage period is accelerated is not by the theory, which states that the specific gravity value is less than 1 and is not by SNI requirements.

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