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Formulation and evaluation test of lip balm from ethanol extract of fruit (*Salacca zalacca (Gaertener)* Voss.)

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Abstract

Lip balm is a product to temporarily prevents dryness of the lips and helps to treat chapped lips. The content of salak fruit, namely flavonoids and phenols, has benefits as an antioxidant. The difference in concentration is found in the ethanol extract of salak fruit, namely 1%, 3% and 5%. This type of research is quantitative with a pre-experimental design. The phytochemical screening test was carried out using reagents of Mg and HCl (for flavonoids) and FeCl3 (for phenol). Physical stability tests were carried out by organoleptic, homogeneity, and pH tests and characteristic tests, namely the melting point of 15 days of storage. Data analysis was carried out using a descriptive approach**.** The results of the phytochemical screening were positive for the presence of flavonoids and phenols as antioxidant compounds. The results of the melting point test at 1% F1, 3% F2, and 5% F3 showed a melting point of 51°C. Organoleptic test F1 1%, F2 3%, and F3 5% showed white results, had no aroma, and had a semi-solid form, pH test F1 1%, F2 3%, and F3 5% was 6, where all formulations were stated homogeneous. It can be concluded that lip balm preparations with concentrations of 1% F1, 3% F2, and 5% F3, which were stored for 15 days, were declared stable.

Keywords: Ethanol extract of salak fruit; Lip Balm; Physical Stability Test

Introduction

Currently, cosmetics play an important role in lifestyle. Makeup products used on the lips, especially by women, are used to add attractiveness and femininity, but men can use some products; usually, they are colorless products and are used to moisturize and protect from the sun (Baki *et al.*, 2019). One cause of lip problems is the use of K-10 and Sudan IV dyes. Lip balms are known to trigger allergic reactions or irritation of the lips in sensitive individuals (Prakash *et al.*, 2020). Chapped, dry, or peeling lips are a very common beauty dilemma due to direct contact with the chemicals contained in lip balms or lipsticks (caused by dyes and fragrances).

Problems with the lips can be prevented by using products that contain organic ingredients such as honey and vitamin E, which can help keep the lips hydrated and healthy (Vinodkumar *et al*., 2019). Lip problems can also be prevented by using lip balm products that contain extracts of natural ingredients, one of which is salak fruit. Natural ingredients can be formulated in the form of lip balm.

Salacca zalacca fruit contains active compounds such as flavonoids and polyphenols and has high antioxidant activity (Tilaar *et al*., 2017; Bunghez *et al*., 2016). In a study by Tilaar *et al*. (2017), 5% ethanol extract of salak fruit had an antioxidant potential of 99.5%. The study by Bunghez *et al*. (2016) informed that salak fruit has an antioxidant activity of 82.67%. Susiloningsih *et al.* (2016) reported that carrots have an antioxidant activity of 8.35%. Tambunan *et al.* (2018) reported that tomatoes have an antioxidant activity of 44.72%.

Based on these results, it is known that salak fruit has the highest antioxidant content among the others. Because of the problems, consequences, and previous research, the authors are interested in researching the physical stability of lip balm preparations from the ethanol extract of salak fruit as the active ingredient in the formulation because it has a higher antioxidant activity than tomatoes and carrots. The novelty of this research is the use of an ethanol extract of salak fruit at concentrations of 1%, 3%, and 5%. This study is expected to provide information regarding the physical stability of lip balm preparations with the formulation of ethanol extract of salak fruit, glycerin, cera alba, propyl paraben, butyl hydroxytoluene, and Vaseline album for 15 days.

Methods

This study used experimental design with descriptive analysis. This research was conducted at the Pharmaceutical Technology Laboratory, and the phytochemical screening test for flavonoids and phenols was carried out at the Phytochemical Laboratory of STIKes Mitra Keluarga Bekasi from February to March 2023. The population and

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sample in this study were fruit *Salacca zalacca* (*gaertener*) Voss.), which were obtained from a supplier of extracts from Palapa Muda Perkasa, Depok. The independent variable in this study was independent variableThe independent variables in this study were organoleptic, pH, homogeneity, melting point, and phytochemical screening.

Plant determination was performed at the Bogorinse Herbarium, BRIN Cibinong. The salak fruits were extracted using the maceration method. Salak fruit simplicia powder (3 kg) was macerated by soaking in 5 L 96% ethanol. The powder was soaked for three days at room temperature (25-30°C). The extract was then filtered. The filtrate obtained from the maceration process was then concentrated using a rotary evaporator at 40°C to remove unwanted solvents to obtain a 137 g thick extract of Salak fruit. The flavonoid test was carried out by putting 0.5g of the extract into a test tube, adding 5 mL of 96% ethanol, then heating it in the test tube for 2 minutes, adding concentrated hydrochloric acid (HCl), then adding magnesium (Mg) powder. A positive result in the flavonoid test produces a red color. Quercetin was used as a positive control, and the extract with the addition of 96% ethanol was used as a negative control.

The phenol test was performed by placing the extract (0.5 g) into a test tube and adding 1% iron (III) chloride ($FeCl_3$). A positive result from the phenol test produces a dark green, blue, or black color. Quercetin was used as a positive control, and the extract with the addition of 96% ethanol was used as a negative control. The preparation was carried out by weighing the base vaseline album, and cera alba melted at a temperature of 62–65^{0}C (mixture A), propylparaben, BHT, and glycerin mixed (mixture B) were added to the melted base while stirring continuously, then added the ethanol extract of salak fruit. g while stirring. The preparation mixture was placed in a lip balm container and left at room temperature until it solidified (Handayani *et al.*, 2021). The lip balm ethanol extract of the salak fruit formula is shown in **Table 1.**

Table 1. Lip Balm Formula

Material Composition	Formula 1	Formula 2	Formula 3	Function
Ethanol extract of Salak fruit	1%	3%	5%	Active ingredient
Glicerin	8%	8%	8%	Emollient
Cera Alba	15%	15%	15%	Stiffening agent
Propyl Paraben	0,2%	0,2%	0,2%	Preservative
BHT	0,02%	0,02%	0,02%	Prevent rancidity in fats and oils
Vaseline Album	Ad 5g	Ad 5g	Ad 5g	Base *lip balm*

The Melting Point Test is carried out by melting the lip balm in the oven with an initial temperature of 50^{0}C for 15 minutes and then observing it. If it does not melt, the oven temperature can be increased every 15 min by 1^{0}C and the temperature at which the lip balm melts. Evaluation of Lip Balm Ethanol Extract of Salak Fruit: Organoleptic tests were carried out by observing lip balm preparations using senses. Organoleptic testing includes observation of the color, smell, and dosage form of the lip balm. Organoleptic tests were performed by examining the color, smell, and form of F1, F2, and F3 of the lip balm preparation (Ambari *et al.*, 2020). The final pH of the lip balm was measured using a universal pH indicator. A pH test was performed to avoid the risk of irritation to the skin of the lips when the lip balm was used. This test was carried out by applying lip balm preparations F1 1%, F2 3%, and F3 5% to a universal pH indicator. The pH of lip balm preparations is acidic or alkaline (Ambari *et al.*, 2020). A homogeneity test was performed by observing the presence or absence of coarse grains during the preparation. This test is carried out by taking an adequate amount of lip balm, smearing it on the watch glass, and then touching and rubbing the lip balm mass to show the absence of coarse particles (Amalia, 2021).

Results and Discussions

Determination of plants in this study was carried out at the Herbarium Bogoriense, Botany Field, Research Center for Biology BRIN, Cibinong. The results of the determination confirmed that the sample of the type of salak fruit used was *Salacca zalacca (Gaertener) Voss*, with the Arecaceae tribe. Based on the results of phytochemical screening, it can be seen that the flavonoid test, and phenol test showed positive results.

The melting point test is a characteristic of the physical properties of lip balm preparations F1 (1%), F2 (3%), and F3 (5%) carried out using an oven for 15 days of storage at a temperature range of 50°C to 70^{0}C. The melting point test

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was carried out to determine the temperature range where all the ingredients in the lip balm preparations at F1 (1%), F2 (3%), and F3 (5%) actually melted. The results of the lip balm melting point test in this study can be seen in table 2.

Table 2. Melting Point Test

Day	F1 (1%)	F2 (3%)	F3 (5%)
1	51°C± 0,00	51°C± 0,00	51°C± 0,00
3	51°C± 0,00	51°C± 0,00	51°C± 0,00
6	51°C± 0,00	51°C± 0,00	51°C± 0,00
9	51°C± 0,00	51°C± 0,00	51°C± 0,00
15	51°C± 0,00	51°C± 0,00	51°C± 0,00

Examination of lip balm organoleptic tests on F1 (1%), F2 (3%), and F3 (5%). Organoleptic testing was carried out for 15 days and observed changes in color, odor and shape of the lip balm preparations. The results of the organoleptic test can be seen in **table 3.**

Table 3. Organoleptic Test

Formula	Hasil Uji Organoleptik	
	Hari ke-1	Hari ke-15
F1 (1%)	Color : White Odor : Odorless Form : Semi-Solid	Color : White Odor : Odorless Form : Semi-Solid
F2 (3%)	Color : White Odor : Odorless Form : Semi-Solid	Color : White Odor : Odorless Form : Semi-Solid
F3 (5%)	Color : White Odor : Odorless Form : Semi-Solid	Color : White Odor : Odorless Form : Semi-Solid

The pH test was carried out to determine the pH value of the lip balm at F1 (1%), F2 (3%), and F3 (5%) whether it is acidic, basic or neutral. pH stability test on F1 (1%), F2 (3%), and F3 (5%) was carried out for 15 days of storage. The results of the pH test in this study can be seen in **table 4.**

Table 4. pH Test

Day	F1 (1%)	F2 (3%)	F3 (5%)
1	6,00 ± 0,00	6,00 ± 0,00	6,00 ± 0,00
3	6,00 ± 0,00	6,00 ± 0,00	6,00 ± 0,00
6	6,00 ± 0,00	6,00 ± 0,00	6,00 ± 0,00
9	6,00 ± 0,00	6,00 ± 0,00	6,00 ± 0,00
15	6,00 ± 0,00	6,00 ± 0,00	6,00 ± 0,00

Research on the formulation and evaluation of lip balms from the ethanol extract of salak fruit (*Salaccazalacca* (Gaertener) Voss.) was carried out in several stages, namely the preparation of samples of salak fruit, extraction of salak fruit, phytochemical screening of the ethanol extract of salak fruit, and formulation and evaluation of lip balm from the ethanol extract of salak fruit. The evaluations included organoleptic, pH, homogeneity, and melting point tests.

Salak fruit extraction was performed using the maceration method with 96% ethanol as the solvent. This study used the maceration method because the compounds taken, namely phenols and flavonoids, which are polyphenolic compounds, are easily damaged when heated. The maceration method was selected to prevent damage to the compound due to heating. The solvent used was 96% ethanol, which was chosen based on safety considerations and the ease of evaporation. Ethanol (96%) can dissolve polar, semi-polar, and non-polar substances optimally to extract flavonoids and phenolic compounds (Ramadhani *et al*., 2020).

The yield is the ratio between the final weight of the ethanol extract of the salak fruit and the weight of the simplicial powder before extraction. In 3000 g of simplicia powder of salak fruit, 137 g of brown viscous extract was obtained, with a distinctive aroma and an extract yield of 7.22%. An extract yield of less than 10% w/w was considered a low yield. According to the Indonesian Ministry of Health (2008), extracts with an optimal yield above 10% w/w. The less-than-optimal extract is probably caused by factors such as the type of solvent, extraction time, ratio of materials and solvents, and particle size, which affect the extraction process (Chairunnisa *et al*., 2019). The higher the yield of the extract, the higher the content of the substance of interest in the sample (Senduk *et al*., 2020).

This phytochemical screening test was performed on viscous extracts to identify the presence of secondary metabolites using color reagents. This examination was performed to determine the contents of the extract preparations (Ramadhani *et al*., 2020). The flavonoid test was carried out using quercetin as a positive control and ethanol extract-

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salak fruit as a negative control. Testing for flavonoids on ethanol extract-salak fruit as a negative control was carried out by putting 0.5 g of the extract into a test tube, adding 96% ethanol, and then heating it for 5 minutes. Subsequently, ten drops of concentrated HCl were added, after which 0.2 g of Mg powder was added (Pangow *et al*., 2018). The addition of Mg powder and concentrated HCl causes a reduction process in flavonoid compounds to produce Mg-flavonoid complexes that are red or orange (Pratiwi *et al*., 2021). The phenol test was carried out using quercetin as a positive control and ethanol extract-salak fruit as a negative control. The phenol test for ethanol extract-salak fruit as a negative control was carried out by putting 0.5 g of the extract into a test tube and adding $FeCl_3$ (Putri *et al.*, 2019). The addition of FeCl31% produces a polyphenol complex compound with a central atom, Feber, which is blackish-green (Iskandar, 2020).

The purpose of the melting point test is to identify the melting point, which is defined as the temperature range in which all materials melt. Based on the results of testing the melting point of Formula 1 (F1) 1%, Formula 2 (F2) 3%, and Formula 3 (F3) 5%, the results obtained have an average melting point of 51°C. According to Amalia (2021), due to the use of the same amount of additives between formulas 1, 2, and 3 so that the melting point of the lip balm preparations does not differ, the melting point test results obtained for each formula are the same. The preparations made have a good melting point, and each formula meets the standards by the requirements of SNI 16-4769-1998.

An *organoleptic test* is a procedure that utilizes the five human senses to assess texture, color, and smell (Tanone & Prasetya, 2019). In Formula 1, which uses 1% extract, lip balm preparations are obtained in a semisolid form, white in color, and odorless. Formula 2, which uses 3% extract, is obtained as a lip balm preparation in semisolid form, white in color and odorless. In Formula 3, which uses 5% extract, lip balm preparations are obtained in a semisolid form, white in color, and odorless. Based on the results obtained, the lip balm preparations did not change in color, smell, or shape, both before and after storage for 15 days. According to Amalia (2021), these results indicate that the formula used is stable because it does not cause interactions between the active substance and other ingredients.

A pH test was performed to determine the safety of the lip balm preparation. Based on the physiological pH of the skin the recommended pH for cosmetic products ranges from 4.5 to 6.5 according to the physiological pH of the skin (Permatananda, 2021). Based on the results of testing the pH values obtained in Formula 1 (F1) 1%, Formula 2 (F2) 3%, and Formula 3 (F3) 5%, the average pH value obtained was 6. According to Ambari *et al.* (2020), lip balm preparations are said to be good if they have a pH in the pH range of the lips, namely 4.5-6.5. Results from an average pH range of 6 are known to meet the requirements, namely, entering the range of 4.5-6.5. Cosmetic products with a pH range between 4 and 6.5 are considered a normal pH for the skin, including lips (Azmin *et al.*, 2020). These results indicated that the results of the pH test fulfilled the requirements, and the preparations made were safe and did not irritate the lips. Acidic or alkaline pH can irritate the lips, so the pH of the formulation is kept as close as possible to a neutral pH (Waykule *et al*., 2022).

The acquisition of the same pH value was six because the pH meter used was a universal pH indicator paper. This was done because of the limited ability of the pH meter to measure the pH of lip balm preparations, because the lip balm preparations had a semisolid form, making it difficult to measure the pH of lip balm preparations, and the limited availability of pH meter preparations. In a study conducted by Amalia (2021), the pH of lip balm preparations from strawberry extract was measured using a universal pH indicator paper obtained at pH 5 in Formulas 1, 2, and 3. In a study conducted by Shaikh (2022), pH measurements of lip preparations balm from beetroot extract using a pH meter (model HI-2211-01) obtained an average pH of 5.6.

The homogeneity test aims to determine whether the ingredients are mixed when creating a lip balm. This also determines whether the lip balm mixture is homogeneous or contains coarse grains or not. Based on the homogeneity test that was carried out to prove that all preparations of salak extract lip balm do not show any coarse particles when applied to the watch glass, this proves that the salak extract lip balm has a homogeneous composition of components. These results indicate that the homogeneity obtained has met the requirements of homogeneity, namely homogeneous or no coarse grains, according to the requirements of SNI 16-4769-1998.

Conclusions

The ethanol extract of salak fruit (*Salacca zalacca* (*Gaertener*) Voss.) can be formulated into lip balm preparations with concentrations of 1% F1, 3% F2, and 5% F3 by obtaining good physical stability results in all formulas. The results of physical stability showed that in the organoleptic test for 15 days, there was no change in color, smell and shape, the pH value was stable with an average pH value of 6, and all preparations had good homogeneity,

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namely, there were no coarse grains. Thus, the conclusion of this study was that lip balm preparations with concentrations of 1% F1, 3% F2, and 5% stored for 15 days were declared stable. Suggestions in this study are an antioxidant testing is also needed to determine the activity of antioxidant compounds in lip balm preparations with a concentration of 1% F1, 3% F2, and 5% F3, and future researchers are expected to test the physical stability of lip balm preparations for one to three months.

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