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Formulation and evaluation of Lip Scrub preparation from lemon peel extract Citrus Limon (L.) Osbeck

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ABSTRACT

Dry and chapped lips are a common disorder of the lips. Common causes of dry and chapped lips are damage to keratin cells due to sunlight and dehydration. This research aims to determine the physical properties of lip scrub preparations from lemon peel (Citrus limon L.) Osbeck and which concentration is the best. This research method was carried out experimentally, namely making extracts using the maceration method, then formulating lip scrub preparations with varying extract concentrations and physical stability testing which included organoleptic, homogeneity, pH and spreadability tests. The results obtained in this research, namely the organoleptic test evaluation, were light brown in color, had the smell of oleum ricini, and had a bitter sweet taste and semi-solid form. Homogeneity test to determine the homogeneity of the lip scrub preparation with the absence of lumpy materials that are not dissolved. The pH test obtained was a semi-solid lip scrub preparation that was stable, namely at an average pH of 5.5. The spreading power test obtained a diameter of 4-5 cm. Based on the evaluation results, lip scrub preparations that have good and stable physical quality are F3 with a lemon peel extract concentration of 6%.

Keywords: *Lip Scrub, Lemon peel, antioxidant, Citrus limon(L.) Osbeck*

INTRODUCTION

Cosmetic product is a preparation or combination of ingredients intended for use on the outside of the body, such as skin, teeth, skin, and others. The genitals, lips, nails, oral cavity, hair, not only to nourish the area and create attractiveness, but also to change its appearance, protect it,keep it in good condition, improve its smell, but its purpose is not to cure or treat any disease (Isnawati, 2020).

Dry and chapped lips are a common disorder of the lips. The cause of dry and chapped lips is keratin cell damage due to sunlight and dehydration. Keratin cells are cells that protect the outer layer of the lips. Exposure to the suncauses rupture of the surface layer of keratinized cells. Broken keratinized cells will be damaged. Damaged cells will occur continuously until the cells are peeled off and

new cells grow. In addition, the cause of dry and chapped lips is dehydration. Dehydration occurs due to insufficient fluid intake or excessive fluid loss caused by environmental influenceone of the cosmetic products that can remove dead skin cells on the lips is a lip scrub. Lip scrub is a scrub granule that works by exfoliating dead skin cells on the lips, so that the lips become moist (Nurmala, 2020). Lip scrub is basically a cosmetic used for skin care, especially in young women and women who use lipstick for a long time every day, causing the skin condition of the lips to become dry and cracked. Using a lip scrub can eliminate dry and dull skin and moisturize the skin without causing any side effects (Setyaningsih et al., 2018). This Lip scrub contains Soft Lip granules, providing exfoliation in the form of a cream or gel as a solution to lip problems. The cream has a rough texture and if applied to the lips, the outer layer will peel off (Isnawati, Nafisah and Stike dr.Soebandi, 2020).

Lemon peel contains antioxidants that are very effective in maintaining the immune system. This lemon peel waste tastes sour if eaten without the sugar mixture. The liquid from the lemon fruit contains 5% citric acid, which gives it a characteristic lemon flavor, and has a pH of about 2 to 3. Lemon peel contains many different compounds that are beneficial to the health of the body. Nine phytochemicals, including saponins, alkaloids, flavonoids, anthraquinones, resins, tannins, terpenes, steroids,and phenols, are known to be abundant in lemon peel extracts (Verdiana et al., 2018).

According to research conducted by (Alfian et al., 2017), lemon fruit (Citrus lemon) is one of the fruits of antioxidant compounds that can ward off free radicals. Testing of antioxidant potential in local and imported lemons taken from skin extracts and fruit pulp juice. Extraction of fruit peel is done by maceration using ethanol solvent 96%. The test was carried out by qualitative and quantitative methods using the DPPH (1,1 - diphenyl-2-picrylhyrazil) method and the antioxidant potential seen from the EC50 value, which measured 50% reduction of DPPH free radical activity using visible spectrophotometric absorbance measurements. The results of qualitative testing of local and imported lemon peel and pulp extracts are known to have antioxidant activity by producing DPPH reduction which is characterized by reduced intensity of purple color. Quantitative testing produced EC50 value in the local lemon peel extract of 1002.57 bpj and 269.38 bpj lemonimport, while for the local lemon juice of 19205.96 bpj and lemon import 5388.58 bpj. The smaller the EC50 value, the greater the antioxidant potential. Imported lemons have a higher antioxidant potential than local lemons, while fruit peel extracts have a higher potency than lemon juice.

According to research conducted (Stevana, 2022), lemon fruit (Citrus limon L.) has antioxidant properties that can fight free radicals because it contains flavonoid compounds. This study revealed the antioxidant activity of lemon peel ethanol extract in vitro using DPPH (1,1-diphenyl-2-picrylhydrazyl) method based on IC50 value. The antioxidant activity test used DPPH (1,1-diphenyl-2-picrylhydridyl) and vitamin C as a comparison. Antioxidant activity was analyzed using the regression line equation y = 2.0742 x + 20.099, and determined

IC50 of 14.41 ° g/ml, and the comparison of vitamin C resulted in IC50 of 8.0 ° g/ml. The results of this study showed that the ethanol extract of lemon peel has a very strong antioxidant activity.

According to research conducted (Dyta et al., 2018), conducted a study aimed to determine the effect of maceration time on the antioxidant activity of lemon peel extract and to determine the best maceration time with the highest antioxidant activity of lemon peel extract. This study used a complete randomized design method with 6 levels of maceration time treatment: 18 hours, 24 hours, 30 hours, 36 hours, 42 hours and 48 hours. The experiment was repeated three times so that 18 experimental units were obtained. The Data were analyzed by ANOVA and then followed by DMRT. The results showed that maceration time treatment significantly affected the extraction results, vitamin C, total phenol, total flavonoids and antioxidant activity of lemon peel extract. The best treatment was 36 hours with medium extraction yield of 26.96%, vitamin C 5.22 mg AA/g, total flavonoids 69.64 mg QE/g, total phenols 16.73 mg GAE/g and antioxidant activity of 94.08%.

Based on the description above, the purpose of this study is to make the formulation and evaluation of lip scrub preparation from lemon peel extract (Citrus limon L.) Osbeck formulation containing high antioxidants that can remove dead skin cells on the lips.

METODOLOGI

Simplicia manufacturing and extraction process

Making Simplicia

The orange peel is cleaned and dried. Next, the size of the orange peel is reduced and drying using an oven at 50-55 ° C for 3-4 hours until it dries completely. The orange peel is then crushed, followed by the sieving process using a 60-mesh sieve. Furthermore simplisia kulitjeruk stored in a closed container and room temperature (Silalahi et al., 2015).

Extraction Making

700 grams of dry lemon peel Simplicia powder was extracted by maceration method using 70% ethanol solvent of 2 liters. then 70% ethanol solvent was added to the macerator until the simplicity was submerged and every 24 hours extraction was carried out for 3x24 hours. the 70% ethanol solvent must be replaced with a new solvent until a clear filtrate is obtained. The extraction results obtained were concentrated using a rotary vacuum evaporator at a temperature of 30-40^{o}c (Hindun et al., 2017).

Phytochemical screening

Phytochemical screening is a procedure performed to detect the presence of secondary metabolite compounds in natural materials. This detection is indicated by the color reaction between the reagent and the test compound. The existence of a color reaction will provide an initial prediction of a compound of natural materials (Hanani, 2016). In this study phytochemical screening showed positive results in tannins (blackish green), phenolic (black), flavonoids (dark red), saponins (foam formed), and terpenoids (brownish ring).

Phytochemical screening of Simplicia and extracts

Identification of flavonoids

The sample was weighed as much as 0.5 g added 10ml of hot water, boiled for 5 minutes and filtered in a hot state,into 5 ml of filtrate added 0.1 g of magnesium powder and 1 ml of concentrated hydrochloric acid and 2 ml of amyl alcohol, shaken and allowed to separate. Flavonoids are positive if there is a red or yellow or orange color on the amyl alcohol layer (Mayasari, 2018) (Ambarsari, 2022).

Identification of alkaloids

The sample was weighed 0.5 g and then added1 ml of hydrochloric acid 2 N and 9 ml of distilled water, heated on a water bath for 2 minutes, cooled and filtered. Filtrate obtained used for alkaloid test, input into a test tube 0.5 ml filtrate. Added 2 drops of Mayer's reagent. Alkaloids are positive if there is a precipitate or turbidity in at least two of the three experiments above (Mayasari, 2018) (Ambarsari, 2022).

Identification of tannins

The sample is weighed as much as 1 g, boil for 3 minutes in 100 ml of distilled water and then cooled and filtered. Solutiontaken 2 ml is added 1-2 drops of Iron (III) chloride reagent1%. If a blue-black or green-black color occurs, it indicates the presence of tannins (Mayasari, 2018) (Ambarsari, 2022)..

Identification of saponins

The sample was weighed as much as 0.5 g and put into a test tube, then added 10 ml of hot water, cooled and then shaken vigorously for 10 seconds. If a foam is formed as high as 1-10 cm which is stable and not less than 10 minutes and does not disappear with the addition of 1 drop of hydrochloric acid 2N (HCl) indicates the presence of saponins (Mayasari,2018) (Ambarsari, 2022).

Lip scrub preparation formulations

Table 1 Formula *lip scrub*

No	Nama bahan	F1 (%)	F2 (%)	F3 (%)
1	Skin extract	2	4	6
2	Lemon fruit	0.1	0.1	0.1
3	BHT	0.18	0.18	0.18
4	Methyl paraben	0.01	0.01	0.01
5	Oleum citri	12	12	12
6	Oleum ricini		5	5
7	Cera alba		Qs	Qs
8	Palm sugar		Ad 10	Ad 10
	Description	F = formula	Qs= enough	

Source : (Awalia Sariningsih et al, 2021).

The preparation of lip scrub consists of vaseline, BHT, methylparaben, Oleum ricni, cera alba. Making the base begins with the melting of Vaseline at a temperature of 60°C and cera alba at a temperature of 80°C. After melting, it is

transferred to a preheated mortar while slowly grinding. Dissolved BHT, nipagin, and Oleum ricini until homogeneous. Put the material into the mortar little by little while grinding continuously until homogeneous, then added Oleum citri. Left the preparation until the temperature decreases to 32°C (room temperature) then add palm sugar as a type of scrub. Grind all ingredients until homogeneous (Awalia Sariningsih et al, 2021).

Physical evaluation of lip scrub preparations

Organoleptic test

This is done by observing the color, smell and taste. This test uses the five senses including the eyes, nose, and skin to determine color, smell, and taste (Jessica, 2018).

Homogeneity test

This is done using a glass object that has been applied as much as 0.1 g of the preparation and then observed whether the ingredients and scrub have been mixed or there are still clumping ingredients (Yusuf, 2019).

PH test

This is done by dissolving 1 g of lip scrub preparation with 100 mL of aquadest and dipping a pH meter that has been calibrated first using aquadest and then left to stand for a while until a fixed pH is obtained. (The Beginning, 2021).

Scatter power test

This is done by taking a sample of the preparation sebanyak1 g and then placed on the watch glass, then close the watch glass using another watch glass. After the sample is in the middle of two watch glasses, it is then given a load and then the diameter of the lip scrub preparation that has spread is recorded (Jessica, 2018).

RESULT AND DISCUSSION

Simplicia test used is lemon peel (citrus limon L.) taken from biopharmaceutical cultivation Conservation Unit (UKBB) Tropical biopharmaceutical Center LRI-PGK IPB (Bogor Agricultural University). The determination of Simplicia was carried out at the biopharmaceutical Laboratory of the Bogor Agricultural Institute (IPB). The purpose of determination is to establish the correctness of the sample used in the study. The results showed that Simplicia determination used in this study is the peel of lemon (citrus limon L.), the results of the determination can be seen in the appendix.

Simplicia processing process begins with the extraction of raw materials from the biopharmaceutical cultivation Conservation Unit (UKBB) Tropical biopharmaceutical Center LRI-PGK IPB (Bogor Agricultural University). Then cleaned with running water, then separated the skin and pulp of kemon, then the skin is dried by means of in the oven at a temperature of 50±5^{o}C until a dry lemon peel is obtained. The skin that has dried, then smoothed and weighed. Lemon fruit collected 30 kg and obtained lemon peel powder of 1100 grams.

Organoleptic examination is performed on fresh and powdered Simplicia. The examination includes the shape, smell, taste and color of fresh and powdered

Simplicia. The macroscopic results can be seen in the table below.

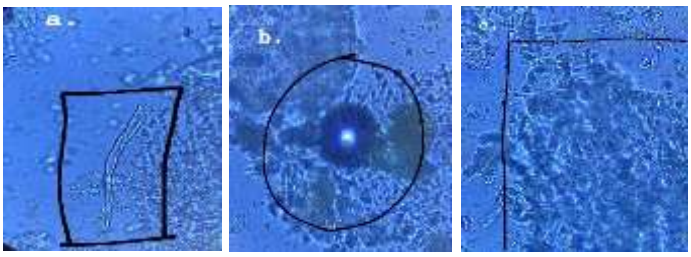
Table 2 Results Of Macroscopic Examination

Inspection	Results	
	Fresh Simplicia	Simplicia powder
Form	Round	Fine powder
Bau	Orange lemon	Orange lemon
Color	Yellow	Faded yellow
Taste	Acid	Sour, bitter

Source : research data processed in 2024

Microscopic examination is carried out to determine the identifying fragments contained in Simplicia. The addition of chloralhydrate aims to remove the content of cells such as amylum and protein so that other cells can be clearly seen under a microscope. Fixation is done so that the chloralhydrate evaporates slightly due to heating so that the Simplicia can adhere perfectly to the glass object. In addition to chloralhydrate can also use the addition with water (Supomo, et al, 2016).

Microscopic examination using a monocular microscope at 100X magnification obtained hair cover, oxalate crystals and fruit skin.



Microscopic : (a.) hair Cover, (b.) oxalate crystals, (c.) fruit peel.

Fig. 1 microscopic examination of lemon peel

700 grams of dry lemon peel Simplicia powder was extracted by maceration method using 70% ethanol solvent of 2 liters. then 70% ethanol solvent is inserted into the macerator until the Simplicia is submerged and every 24 hours the extraction is carried out for 3x24 hours. the 70% ethanol solvent must be replaced with a new solvent until a clear filtrate is obtained. The extraction results obtained are concentrated by using a rotary vacuum evaporator at a temperature of 30-40^{o}c (Hindun et al., 2017). The purpose of the rotary evaporator is to concentrate the extract from the previous maceration process that uses a lot of solvent by evaporating at a certain temperature so that a thick extract will be obtained (Ningsih, 2017). Yield obtained as much as 38.57%.

Table 3 results of phytochemical screening test Simplicia

Metabolit Sekunder	Simplisia	Ekstrak
Flavonoid	+	+
Alkaloid	-	-
Tannin	-	-
Saponin	+	+

Specification: (-) : does not contain secondary metabolite compounds
(+) : contains secondary metabolite compounds

Polyphenolic compounds with two or more hydroxyl groups, are mildly acidic, soluble in bases, and polar. Generally flavonoids are found to be linked with sugars to form glycosides so that in this study, 96% ethanol reagent is added to dissolve flavonoids. The polarity of flavonoids increases with the presence of glycoside-shaped sugar bonds (C - glycoside or O-glycoside) so that they are easily soluble in water. To detectthe presence of flavonoids it is necessary to reduce using Mg reagents. Flavonoid compounds reduced by Mg will produce red, yellow, and orange colors (Wahid and Safwan, 2020).

The positive results of alkaloid compounds in mayer's reagent are indicated by the formation of a white to yellowish precipitate. This is due to the fact that alkaloid compounds are able to react with the indicated tetraiodomerurate (II) ionwith the formation of a white precipitate. However,this reagent has a weakness, which is able to bind to non-alkaloid compounds such as protein,coumarin, ③ -pyrone, hydroxy flavone and tannin. This bond causes a result called a false positive reaction. In addition, alkaloid compounds have quaternary forms that cannot be replaced with alkaline alkaloids and remain in cells, so they are not capable of detection with mayer's reagent. This result is called a false negative (Marliana et al., 2005 in Reza Anindita et al, 2022).

Samples with the addition of FeCl3 (heavy metals) are shown with a blue-black precipitate, while the condensed tannins will give a brownish-green color (Sukma et al., 2018).

As for saponins, a positive result is shown by the formation of foam over a long period of time. Saponins are water soluble, insoluble in ether, and include glycoside compounds with sugar molecules that bind to triterpene/steroid aglycones, the aglycone found in lemon peel is limonin which has a bitter taste. Hydrolysis of saponins will produce glycosides that are polar and aglycones have steroids or triterpenoids that are nonpolar. The existence of polar and non-polar properties causes saponins to form micellar structures when shaken with water which causes the nonpolar series to lead inward and the polar series to the outside (Habibi et al.,2018). This is evidenced in the form of a foam that looks stable.

Table 4 organoleptic test results

Formula	Color	Smell	Taste	Shape
F1	Chocolate	Oil distance	Bitter, sweet	Semi solid

	young			
F2	Chocolate young	Oil distance	Bitter, sweet	Semi solid
F3	Chocolate young	Oil distance	Bitter, sweet	Semi solid

Source : research data processed in 2024

Organoleptic test results have the same color because it uses the same formula only distinguished by the addition of concentration (%) extract.

F1 F2 F3

Fig. 2 organoleptic test results of orange peel extract lip scrab preparation

Tabel 5 Homogeneity test results

Formula	Uji pH Result
F1	Homogen
F2	Homogen
F3	Homogen

Source : research data processed in 2024

Homogeneity test results in formulations 1, 2 and 3 obtained homogeneous results and no coarse grains.

F1 F2 F3

Fig. 3 homogeneity test results of orange peel extract lip scrub preparation

Tabel 6 Uji pH Result

Formula	Uji pH Result

F1	5,61
F2	5,49
F3	5,50

Source : research data processed in 2024

The pH test showed that the eligible result was 4.5-6.5 (Titaley et al., 2014). The results of the pH test at F1 pH (5.61) , F2 pH (5.49)and F3 pH (5.50) obtained stable results at Number 5 which is still the safe limit of skin acidity. Conditions that are too acidic preparations will result in skin irritation, while conditions that are too alkaline can make the skin becomes scaly (Titaley et al., 2014).

F1 F2 F3

Fig. 4 pH test results of lip scrub preparation lemon peel extract

Table 7 power spread test results

Formula	**Hasil uji Daya Sebar**
F1	4 cm
F2	4 cm
F3	5 cm

Source : research data processed in 2024

The test results of F1 (4) and F2 (4) are not qualified while F3 (5) is qualified. A good spread is 5-7 cm (Sayuti, 2018).

F1 F2 F3

Fig. 5 test results of the spread of Lip scrub preparation lemon peel extract

CONCLUSION

Based on the results of research conducted, it can be concluded that the best preparation of lemon peel extract lip scrub is F3, because from the results of the physical properties test, it meets the requirements with homogeneous homogeneity, pH 5.50 is eligible, and the spread of 5 cm is eligible. The concentration that most affects the results of the evaluation of the preparation is F3 with a concentration of 6%.

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