

Formulation and Evaluation of Physical Quality of Peel-Off Masks of Essential Oils of Galam Kalimantan Plants

Noverda Ayuchecaria¹, Karelius¹, Lilis Rosmainar¹, Saftia Aryzki²

¹Chemistry Study Program, Faculty of Mathematics and Natural Sciences, University of Palangkaraya, Palangkaraya, Indonesia

²Pharmacy Study Program, Faculty of Health, Sari Mulia University, Banjarmasin, Indonesia.

*Correspondence Author : noverdaayuchecaria@mipa.upr.ac.id

Abstract:

The Kalimantan galam plant (*Melaleuca cajuputi* subsp *cajuputi*) has been used for generations but its use is very limited, only as wooden piles which form the basis of buildings in swamps (cerucuk), pillars for building construction, stakes (supports for newly planted seedlings) and siring. Street. The use of galam essential oil as an active ingredient in peel-off mask preparations can be efficacious in reducing inflammation and infection of acne or other skin problems. Facial masks in the form of peel-off itself have many advantages, namely their practical use and are easy to remove or lift like elastic membranes compared to conventional masks which leave residue. This study aims to design a peel-off mask formulation by utilizing the active ingredient in the essential oil of the galam plant. The preparations were then tested for physical quality requirements including organoleptic tests, homogeneity tests, pH tests, drying time tests, spreadability and adhesion tests. The results of the tests that have been carried out include the extraction of essential oils as the active substance. The yield of essential oil obtained was 0.12%. Organoleptic quality tests showed changes in color, smell and shape in the three formulations. The homogeneity test for formulas I and II remained homogeneous, while for formula III the consistency changed to 2 layers. The pH test showed that all formulations met the pH requirements for topical preparations, namely 4-8. The drying time test for formula I was more difficult to remove, formula II was not too stiff while formula III was stiffer. Spreadability test on formulas I, II and III 5-7 cm. The gel viscosity test decreased and the adhesion test on formulation I was easily released, formulas II and III were not easily released while the irritation test of 30 people did not experience redness in 3 formulas.

Keywords: Formulations, Essential Oils, Galams, Peel-off masks

Introduction

Essential oils have an antibacterial effect so they can inhibit the growth of pathogenic bacteria on the face, one of which is *S. Aureus*. *S. aureus* is a gram-positive bacterium that is normally found on human skin. Aside from being an antibacterial, essential oils also have other properties such as anti-inflammation and antioxidants, so they are good for the skin, considering that one of the consequences of a pathogenic bacterial infection on the face is causing inflammation and damaging the skin barrier.¹

Therefore, the use of essential oils in facial cosmetic preparations has multifunctional complementary effects. The great benefits of attrition oil in the cosmetic field, provides a great opportunity to create a preparation that is effective and acceptable when used by consumers.

Essential oils contain different compounds depending on the plant they are produced from. The chemical components of essential oils can generally be grouped into 4 groups that determine the properties of essential oils, namely: terpenes, compounds, benzene derivatives and other compounds.

Essential oils contain hydrocarbon compounds which have the empirical formula $C_{10}H_{16}$ and oxygen-containing compounds with the empirical formulas $C_{10}H_{16}O$ and $C_{10}H_{18}O$ which are referred to as terpenes (Ketaren, 1987). Terpene compounds are grouped into monoterpenes ($C_{10}H_{16}$), sesquiterpenes ($C_{15}H_{24}$), diterpenes ($C_{20}H_{32}$) and triterpenes ($C_{30}H_{48}$).

The essential oil of the galam plant can be extracted by distillation, especially from the twigs and leaves. The word "white" is taken from the white stem of the plant. Eucalyptus oil has volatile properties. On a hot day, people who are close to this tree

will be able to smell the oil produced by the galam plant. The eucalyptus oil of the galam plant has not been used optimally in Kalimantan.

Even though this type has been widely developed in Indonesia, especially on the islands of Java and Maluku by utilizing the leaves and branches to be distilled traditionally by the community and commercially into essential oils with high economic value. This plant has a long biological cycle, grows fast, can grow well on well-drained soil or not with high salt and acid levels and tolerant in the open.

Eucalyptus oil is a type of essential oil typical of Indonesia. This oil is known to have many properties, both for external treatment and internal medicine. Cineol is the main component of eucalyptus oil.

According to Khabibi, J. (2011), states that the main components of eucalyptus oil are cineol ($C_{10}H_{18}O$), pinene ($C_{10}H_{18}$), benzaldehyde ($C_{10}H_{5}H_0$), limonene ($C_{10}H_{16}$) and sesquiterpenes ($C_{15}H_{24}$).

Research Method

One kilogram of fresh galam leaves is washed and drained and then put into the destilator which has previously been filled with aquadest. The distillation process is stopped if the volume of essential oil that comes out does not increase. The distillation results are separated from water by adding anhydrous sodium sulfate. The essential oil obtained was then stored in a tightly closed bottle and protected from light.²

Antibacterial activity was determined using the diffusion method. Bacterial culture was rejuvenated in Nutrient Broth (NB) medium (incubation temperature $37^{\circ}C$ 24 hours). The refreshed bacterial culture was then spread onto solid NA medium and wells were made. A total of 50 μl the sample was put into the well, then incubated at $37^{\circ}C$ for 24 hours. The clear

zone formed was then measured using a vernier caliper. In this study, Dimethyl sulfoxide (DMSO) was used as a negative control and chloramphenicol as a positive control.³

Determination of Minimum Inhibitory Concentration (MIC) Value. The MIC value was determined using the Bloomfield method. The working procedure used to determine the MIC value is the same as for determining antibacterial activity, namely the agar diffusion method with a total of 5 concentration series. The clear zone formed was then measured using a vernier caliper.

Mask preparations were made in 50 grams with an initial design using a modified standard base formula as follows: 10% polyvinyl alcohol, 1% HPMC, 12% Glycerin, 2% TEA, 0.05% Propyl Paraben and 0.2% Methyl Paraben.

The mixture is then added with distilled water until it reaches 50 grams. Variations on masks were made in 4 formulas by differentiating the concentration of essential oils added. The process of making a peel-off mask is preceded by dissolving PVA use aquadest (temperature 80°C) until it expands and is crushed homogeneously (mass I).

HPMC was developed first with cold distilled water in a mortar for 15 minutes and added glycerin, methyl paraben, propyl paraben which were previously dissolved in hot aquadest (mass II). Mass I and mass II are mixed homogeneously while adding TEA little by little. Leave the mixture at room temperature and add galam essential oil according to varying concentrations. The mixture is then added to the remaining distilled water little by little and stirred until homogeneous.

The finished preparation is then put into an appropriate container and labeled according to the concentration of the mask.⁴

Peel-Off Mask Physical Quality Test

Observations are seen directly in the form of color, smell, and texture of the preparation during the storage time of 2 weeks.⁴

Homogeneity test was carried out using a glass slide. The preparation was checked for homogeneity by smearing it on the slide and then flattening it with another slide and then observing whether there are coarse particles.⁵

pH measurement was carried out by dipping the pH meter into a mixture of 1 gram of mask preparation which had previously been dissolved in water with a volume of 10 mL.⁶

As much as 1 gram of the preparation was rubbed on the back of the hand with a length range of 5 cm and 5 cm wide, then the speed of drying the mask was calculated to form a film layer using a stopwatch.⁷

In spreadability test, 1 gram of preparation was placed in the center between 2 glasses, where the upper glass was loaded by placing weights so that it reaches a weight of 150 grams, measurements were carried out until the diameter of the spread of the preparation is constant.⁷

In stickiness test, 0.25 gram of the preparation was placed between 2 glass objects on the stickiness test apparatus, then pressed with a 250 g load for 1 minute, the weight was lifted and given a load of 80 grams on the tool and the release time was recorded.⁷

The viscosity test was carried out using a Brookfield spindle viscometer no.4 with a speed of 20 rpm. The preparation was put in a glass container and then the spindle that has been installed is lowered so that the spindle border is immersed in the mask. Then the viscosity scale is recorded.⁷

Irritation test was carried out on volunteers. The mask preparation was

taken sufficiently then rubbed on the back of the ear for 30 minutes. Symptoms that arise were observed, generally positive irritation was characterized by redness, itching, and swelling of the treated skin.⁷

The criteria for volunteers were able-bodied women, aged between 20-30 years, no history of disease related to allergies, willing to volunteer and willing to fill out an informed concern.

Results

Researchers took fresh samples in the form of Galam plant leaves taken from residents' yards along Jalan G.Obos VII, Palangka Raya City, Central Kalimantan. Leaf samples were taken at 09.00 WIB. The sampling time was chosen at 9 o'clock to keep the leaves fresh. The leaves taken are young leaves ranging from 6-7 leaves from the shoots.

The collected leaves were then immediately sorted and washed. The purpose of sorting and washing is to remove dirt/dust attached to the leaves. In addition, sorting also aims to separate leaves that are not suitable, for example, with holes or withers.



Figure 1 Process of Sorting, Cleaning, and Slicing Galam Leaves

The clean leaves were then chopped into 1 cm pieces using a knife. This chopping aims to reduce the sample size to facilitate the extraction process.

Galam oil distillation was carried out using the steam distillation method.⁸ The 2 kg of chopped galam leaves and water is put into the distillation kettle. Then distilled for 4 hours with a temperature ranging from 100-120°C. The water vapor flows into the distillation kettle which contains galam leaves. The resulting distillate is accommodated in an Erlenmeyer. The distillate is added with anhydrous sodium sulfate to absorb the water contained in the distillate. The essential oil obtained was stored in a tightly closed bottle and protected from light.

**Figure 2** Distillation Process

The yield of essential oils is found by the formula:

$$\text{Rendemen} = \frac{\text{massa minyak (gr)}}{\text{massa sampel (gr)}} \times 100\%$$

Table 1 Weight of Oil from Distillation Process

No	Galam Leaf Weight	Oil Weight
1	1 kg	1,2 gr
2	1 kg	1,3 gr
3	1 kg	1,1 gr
Average		1,2 gr

Based on the distillation results of fresh galam leaves, 1.2 grams in 1 kg is obtained. This shows that the essential oil content contained in galam leaves is 0.12%.

Peel-Off Mask Preparation Formulation

In this study, a gel peel-off mask preparation was chosen because it is more effective and efficient and easier to apply on the face. The preparation is then allowed to dry and form a thin, transparent, and elastic film so that it can be easily removed without washing.

These preparations can provide the appropriate effect contained. The gel mask formula is made of 3 different gelling agent variations. The use of this concentration is based on testing the activity of the extract at a minimum concentration that can inhibit initially, namely 10%. The excipient in the gel mask formula used is PVA as a gelling agent which has adhesive properties or can form a gel film which

can be peeled off after drying. HPMC functions as viscosity increaser of gel mask base. Propylene glycol functions as a humectant which can bind water (hydration) so that the preparation remains moist and does not dry out when used. Nipagin and nipasol are used as preservatives, while 70% ethanol functions as a solvent for the active substance. In addition, it can be used to speed up the drying time of the gel so that the formation of a peel-off gel mask film is formed.

Table 2 Functions in Formulation

Material	Use	Total Material (%)		
		Formulas I	Formulas II	Formulas II
Galam Essential Oil	Active Ingredients	3 mL	3 mL	3 mL
Carboxymethylcellulose (CMC)	<i>Gelling agent</i>	0,75	1,5	2,25
Polyvinil Alcohol (PVA)	Gelling Agent	11	9	8
Glycerol	Humektan	10	10	10
Nipagin	Preservative	0,2	0,2	0,2
Triethanolamin (TEA)	Alkalizing agent	1	1	1
Aquadest ad	Solvent	100	100	100

Physical Quality Test of Peel-Off Mask Preparation

The results of organoleptic observations including shape, color, and smell are as seen in Table 3.

Table 3 Observation Results on Organoleptic

Organoleptis	Day 1			Day 3			Day 14		
	FI	FII	FII	FI	FII	FII	FI	FII	FII
Color	+	+	+	+	+	+	+	+	+
Smell	+	+	+	+	+	+	+	+	+
Form	+	+	+	+	+	+	+	+	-

The results of organoleptic observations for 3 days did not show any changes in color, smell, and shape in the three formulations. White in color with a slight yellowish tint. The distinctive aroma of eucalyptus oil is obtained from the addition of the essential oil of the galam plant. Formulas I, II and III have the same consistency, namely viscous semisolid. On the 14th day of observation, formula III changed its color to become more yellow than before. In addition, formula III also changed its shape to 2 layers.

This is due to the process of syneresis. The 4% CMC concentration should bind the gel more strongly than formulas A, B, and C. However, the minimal amount of water in formula D was unable to disperse.

CMC perfectly. As a result, CMC is unable to bind to the gel and water comes out of the gel structure.

Homogeneity Test

The gel preparation is said to be homogeneous if there is an even color equation and no particles or coarse material that can be touched.⁸

The requirement for homogeneity of the gel is intended so that the active ingredients in the gel are evenly distributed. In addition, so that the gel does not irritate when applied to the skin. Formulas I and II remained homogeneous until the 14th day. While formula III changed its consistency to 2 layers due to the syneresis process (Table 4).

Table 4 Homogeneity test results of Gel Peel-Off masks

Formulas	Day 1	Day 3	Day 14
I	homogeneous	homogeneous	homogeneous
II	homogeneous	homogeneous	homogeneous
III	homogeneous	homogeneous	inhomogeneous

Test of pH

Examination of pH is a physicochemical parameter that must be carried out for topical preparations because pH is related to the effectiveness of the active substance, the stability of the active substance and the preparation, as well as comfort on the skin when used.

A pH that is too acidic can cause irritation, while a pH that is too alkaline can cause scaly skin. From the results of pH measurements, all gel mask formulas are peel-off.

Galam plants meet the pH requirements for topical preparations, namely between 4-8.⁹

Tabel 6 Test Results pH

Formula	Test	pH	Average $\pm$ SD
I	1	6,14	6,16 $\pm$ 0,015275
	2	6,17	
	3	6,16	
II	1	6,51	6,51 $\pm$ 0,005774
	2	6,51	
	3	6,50	
III	1	7,44	7,46 $\pm$ 0,015275
	2	7,46	
	3	7,47	

Spreadability Test

The spreadability test of gel preparations is intended to determine the ability of the gel to spread when applied to the skin. Spreadability is an important characteristic in formulations because it affects the transfer of the active ingredient to the target area in the right dose, ease of use, pressure required to exit the package, and consumer acceptance.¹⁰

From the results of measuring the diameter of the spreadability, Formulas I, II and III meet the spreadability requirements of 5-7 cm.

Viscosity Test

Viscosity is a statement of the pressure of a liquid to flow, the lower the viscosity, the higher the resistance. Viscosity is a physical parameter that is

usually measured to assess the effect of pressure conditions on semisolid products.¹¹

The difference in the viscosity values of formulas I, II and III is caused by differences in the concentration of CMC as a gel matrix former. CMC includes cellulose derivatives.¹²

In polymer dispersions derived from cellulose, polymer molecules enter the cavity formed by water molecules causing hydrogen bonds to occur between the hydroxyl groups (-OH) of the polymer and water molecules. This hydrogen bonding plays a role in hydration in the swelling process of a polymer so that with an increase in CMC concentration it causes more hydroxyl groups and a higher viscosity. Gel viscosity decreased during storage. This can be possible due to several factors such as packaging that is less tight and humidity in the gel storage room that is not controlled can cause the gel to absorb moisture from the outside, so that the viscosity of the gel decreases.

Dry Time Test

The dry time test of the preparation was carried out by observing the time

required for the gel to dry, namely the time from when the peel-off gel mask was applied to the glass until a layer was formed that could be peeled off. All formulas meet the dry time test requirements of 15-30 minutes.³

Peel-off gel masks that dry faster provide comfort for use, while peel-off gel masks dry longer causing discomfort in use. PVA concentration effect on the

drying speed of the preparation, the greater the concentration of PVA, the faster the drying time of the preparation. PVA works through a development process by binding to the existing water so that the water molecules will be close together and there will be attraction between them water molecules that cause an increase in cohesiveness.¹³

Tabel 6 Formulas

Formula	Day 1	Day 14
I	25 minute 35 second	24 minute 15 second
II	19 minute 10 second	18 minute 55 second
III	17 minute 30 second	15 minute 55 second

After the preparation dries, the mask is then peeled off. In formula I, the mask is more difficult to remove because the layer formed is very thin. Whereas in formula III the preparation is stiffer so that when removing the mask layer, it hurts more. Formula II shows the ease of removing the mask where the layer formed is thick enough, so it doesn't tear when removed. The preparations are also not too stiff, so

they are still comfortable when used or removed.

Stickiness Test

As much as 0.25 gram of the preparation is placed between 2 glass objects on the stickiness test apparatus, then pressed with a 250 g load for 1 minute, the weight is lifted and given a load of 80 grams on the tool and the release time is recorded.⁷

Table 7 Stickiness Test Results

Formula	Day 1	Day 2	Day 3
I	8 minute 37 second	8 minute 25 second	8 minute 30 second
II	8 minute 55 second	8 minute 57 second	8 minute 50 second
III	9 minute 10 second	9 minute 14 second	9 minute 15 second

The results of the adhesion test showed that formula I was easily released, formulation II and formulation III took a very long time to release. These results show that the gel consistency, which is large, runny has less time than the gel preparation which has a denser consistency. This is the length of time the release of the preparation is proportional to the high value of adhesion. This meets the requirements for adhesion to the gel

preparation, which is not less than 4 seconds.

Irritation Test

Irritation test was performed on volunteers. The mask preparation is taken sufficiently then rubbed on the back of the ear for 30 minutes. Symptoms that arise are observed, generally positive irritation is characterized by redness, itching and swelling of the treated skin.⁷

The criteria for volunteers: able-bodied woman, aged between 20-30 years, no history

of disease related to allergies, willing to volunteer and willing to fill out an informed concern.

In the results of this irritation test conducted on 30 people, it was seen that there was no redness in formulas I, II and III. But in Formula III it looks stiff so it's uncomfortable when removed.

Conclusions

Having an essential oil yield of 0.12% by organoleptic test showed a change in color, smell and shape in the three formulations. The homogeneity test for formulas I and II remained homogeneous, while for formula III the consistency changed to 2 layers. The pH test was 4-8, the dry preparation time test on formula I was more difficult to remove, formula II was not too stiff while formula III was stiffer. Spreadability test is 5-7 cm. Gel viscosity test decreased. While the adhesion test on formula I was easily released, formulas II and III were released for a long time and none of them experienced redness in the irritation test.

References

1. Saputri & Zahara, 2016. *Uji Aktivitas Anti-Inflamasi Minyak Atsiri Daun Kemangi (Ocimum americanum L.) pada Tikus Putih Jantan yang Diinduksi Karagenan*. Pharmaceutecal Sciences and Research, 3(3) : 107-119.
2. Yuliani, R, Peni I., Septi SR, 2011. *Aktivitas Antibakteri Minyak Atsiri Daun Jeruk Purut (Citrus hystrix) Terhadap Staphylococcus aureus dan Esherichia coli*. Pharmacon. 12 (2): 50-54.
3. Slavtcheeff, C.S 2000. Cosmetics Mask. US Patent5747022a.<https://www.google.com/Patents/US5747022> (diakses 05 Agustus 2016).
4. HC A. 1989. *Pengantar Bentuk Sediaan Farmasi IV*. Penerjemahan Farida Ibrahim, editor. Jakarta.
5. Khabibi, J., 2011. *Pengaruh Penyimpanan Daun dan Volume Air Penyulingan terhadap Rendemen dan Mutu Minyak Kayu Putih*. Skripsi. Departemen Hasil Hutan, Fakultas Kehutanan, Institut Pertanian Bogor.
6. Liza Pristianty, 2011. *Buku Ajar Preskripsi Obat dan Resep JILID 1*. Surabaya: Airlangga University Press.
7. Bajaj S, Singla DSN, 2012. *Stability Testing of Pharmaceutical Products Japs*.
8. Setyaningrum, N.L. 2013. Pengaruh Variasi Kadar Basis HPMC dalam Sediaan Gel Ekstrak Etanolik Bunga Kembang Sepatu 9Hibiscus rosa-sinensis L.) Terhadap Sifat Fisik dan Daya Antibakteri Pada Staphylococcus aureus. Naskah Publikasi. Universitas Muhammadiyah Surakarta. Surakarta.
9. Aulton M. 1998. *Pharmaceutics: The Science of Dosage Form Design*. Curchill LivingStrone. New York.
10. Garg A., Anggarwal D., Garg S., Sigla A.K. 2002. Spreading of Semisolid Formulation: An Updtade. Pharmaceutical Technology, September 2022: 84-102.
11. Lieberman H.A., Martin M.R., Banker G.S 1996. *Pharmaceutic Dosage Forms Disperse Systems*. 3rd edition. Volume 2. New York: Marcel Dekker Inc. New York.
12. Rowe R.C., Paul J.S, Marian E.Q. 2009. *Handbook of Pharmaceutical Excipient*. Edition. Pharmaceutic Press. London.
13. Puspita A., Ari W., Sutiasningsih. 2014. Optimasi Penggunaan Polivinil Alkohol Sebagai Gelling Agent Pada Masker Gel PEEL-OFF Sari Daging Kulit Buah Semangka (*Citrullus vulgaris (Thunb) Maksum*.

