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Formulation of Peel-Off Masks Containing Duwet Leaf Extract (Syzygium Cumini)

Elva Asmiati, Dyah Rahmasari*, Dita Ayu Winata, Ilmi Nisak Prinastiti, Melinia Kartika Sari, Rizka Ayu Indah, Raditya Weka Nugraheni

Department of Pharmacy, Faculty of Health Science, University of Muhammadiyah Malang

ORCID
Dyah Rahmasari: https://orcid.org/0000-0001-9853-4024

Abstract.
The main cause of premature aging of the skin is the presence of free radicals which are created by UV rays. The use of antioxidants is a promising option for overcoming these problems. Duwet (*Syzygium cumini*) is a natural source of antioxidants. A peel-off mask is a cosmetic product which forms an easy to peel-off layer on the skin's surface. This study aimed to determine the physicochemical characteristics, antioxidant activity, irritation effect, and stability of duwet leaf extract peel-off mask preparations. Duwet leaf extract was macerated by a methanol solvent and formulated into the peel-off masks with a concentration of 1%, 3%, and 5%. The results showed that the preparation produced good characteristics with a drying time of fewer than 30 minutes and showed significant antioxidant activity with the highest inhibition in the preparation with 3% duwet leaf extract. This preparation also had no irritating effect on CAM and was stable during storage using freeze-thaw and real-time methods. It can be concluded that duwet leaf extract can be used as an active agent for anti-aging peel-off masks.

Keywords: duwet leaf extract, peel-off mask, physicochemical characteristics, stability test, antioxidant activity, irritation effect

Corresponding Author: Dyah Rahmasari; email: dyahrahmasari@umm.ac.id

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1. Introduction

In this modern era, air quality is decreasing due to increased pollution such as cigarette smoke, industry and motor vehicles. This can be a dangerous problem for the skin. Exposure to the sun's ultraviolet (UV) rays can also cause damage to the structure and function of the skin [1]. Premature aging is one of the skin problems caused by free radicals. Premature aging is the process of skin aging faster than it should be. This is usually caused by various internal and external factors. The aging process on the skin is characterized by the appearance of wrinkles, scales, dryness, cracks, looks dull and wrinkled. The skin ages faster and more dark spots appear [2]. Skin aging is intrinsically influenced by an imbalance between the production of free radicals, especially reactive oxygen species (ROS), the level of effectiveness of free radical scavengers, and body repair. The most abundant source of intracellular ROS comes from the mitochondria.

Increased ROS can cause damage to lipids, proteins and deoxyribonucleic acid (DNA) cells which will trigger the skin aging process. [3].

Efforts to prevent the harmful effects on the skin due to free radicals are with antioxidants. The use of antioxidants topically on the skin is useful to reduce or prevent damage to these cell structures [4]. Antioxidants such as vitamin C, vitamin E are several types of natural antioxidants, both are found in plants or food. In addition, synthetic antioxidants are also used. Synthetic antioxidants that are popularly used are antioxidants of the phenolic type, namely butylated hydroxyanisol (BHA), butylated hydroxy-toluene (BHT), tertiary butylhydroquinone (TBHQ), and esters of gallic acid, such as propyl gallate (PG). Synthetic antioxidants also have a usage limit, which is about 0.02% of the fat or oil content. Although it has been tested for toxicology and safety of use, the choice of using natural antioxidants is still preferred [5].

Choice of natural antioxidants taken from plant or food sources. In this study, duwet (Syzygium cumini) leaves were used which are widely grown in several areas in Indonesia. Antioxidant levels in plant extracts can be measured and seen from the IC50 results obtained after the study. A compound is categorized as a very strong antioxidant if the IC50 is <50 ppm, a strong antioxidant with an IC50 in the range of 50-100 ppm, a moderate antioxidant if the IC50 is in the range of 100-150 and a weak antioxidant if it is >150 ppm [6]. Duwet leaf extract (Syzygium cumini) is known to have an IC50 of about 8.85 ppm which indicates that the antioxidant content of Duwet (Syzygium cumini) leaves is high. The smaller the IC50 number obtained, the higher the antioxidant activity it has. So that it is categorized as Duwet leaf extract (Syzygium cumini) including antioxidants with very active activity levels [7].

In general, women like cosmetic products that are simple in use. One of the cosmetics that is easy to apply and practical is a peel-off gel mask that has a gelling agent base such as PVA. PVA (polyvinyl alcohol) has good film-forming properties, is emulsifying and adhesive so that it can form a good peel-off gel mask, besides that PVA can also be used as film-forming [8]. Based on this, it is necessary to research the formulation of peel-off gel mask preparations of duwet leaf methanol extract as an active ingredient by using various concentrations to know the levels of active ingredients that are good for Peel-off gel mask preparations. This study aimed to determine the physicochemical characteristics, antioxidant activity, irritation effect, and stability of duwet leaf extract peel-off mask preparation.

2. Method

This research was an experimental research methodology and was conducted from December 2020 to September 2021. The tests carried out were to determine the physicochemical characteristics, antioxidant activity, irritation effect, and stability of duwet leaf extract peel-off mask preparation. Duwet leaf extract was macerated by methanol solvent and formulated into the peel-off mask with the concentration of 1%, 3%, and 5%. This research is begin with the preparation of the peel-off gel mask. In this preparation, we need PVA (Polyvinyl Alcohol), Carbomer, TEA (Triethanolamine), nipagin, propylene glycol, sodium metabisulfite and the methanol extract of Duwet (Syzygium cumini) leaves. Aquadest and fragrance are added and stirred until homogeneous.

The test of physicochemical characteristics of the methanolic extract of Duwet (Syzygium cumini) leaf included organoleptic, homogeneity, viscosity, dispersion, drying time and pH of the peel-off gel mask. Antioxidant activity using the DPPH method, irritation test by HET-CAM method and Stability tests were carried out using the real-time & Freeze-Thaw cycling method. Every test group was replicated each 3.

3. Result

The results of organoleptic observations of the peel-off gel mask preparation of duwet leaf extract had almost the same color, texture and aroma in each formula. The resulting green color comes from the duwet leaf extract itself. In the 1% formula, the color is yellowish-green and as the extract content increases, the green color becomes darker, so in the 3% and 5% formulas the color is brownish-green. The texture of the three formulas is the same, namely soft texture. The three formulas have distinctive jasmine (jasmine) scent. The results of the homogeneity test observations of the three formulas and their replications showed homogeneous results. The peel-off gel mask shows a homogeneous composition if there are no visible coarse grains, the texture looks even and does not clump [8].

Formula 1 is 1% concentration, formula 2 is 3 and formula 3 is 5%. Based on the picture above, it can be seen that formulas 1, 2 and 3 have different viscosity. The viscosity of formula 1 is higher than that of formula 2 and 3. The viscosity of formula 2 is higher than that of formula 3. This is because the higher the concentration of duwet leaf extract, the lower the viscosity of the preparation. When viewed from the rheogram graph, the viscosity test of the peel-off gel mask preparation, duwet leaf extract, showed pseudoplastic non-Newtonian flow properties. To determine the effect of increasing the

Table 1: The result of physicochemical characteristics test.

Test type	Concentration		
	F1	**F2**	**F3**
organolepticity			
Color	yellowish green	brownish green	brownish green
Texture	soft	soft	Soft
scent	jasmine	jasmine	jasmine
homogeneity	homogeneous	homogeneous	homogeneous
Viscosity	-	-	+
F1	-	-	+
F2	+	+	-
F3	-	-	+
dispersion			
F1	-	-	-
F2	-	-	-
F3	-	-	-
drying time			
F1	-	+	+
F2	+	-	+
F3	+	+	-
pH			
F1	-	+	+
F2	+	-	-
F3	+	-	-

note : homogeneous: no coarse grain is seen, the texture looks even and does not clot. (-) not significantly different, (+) significantly different

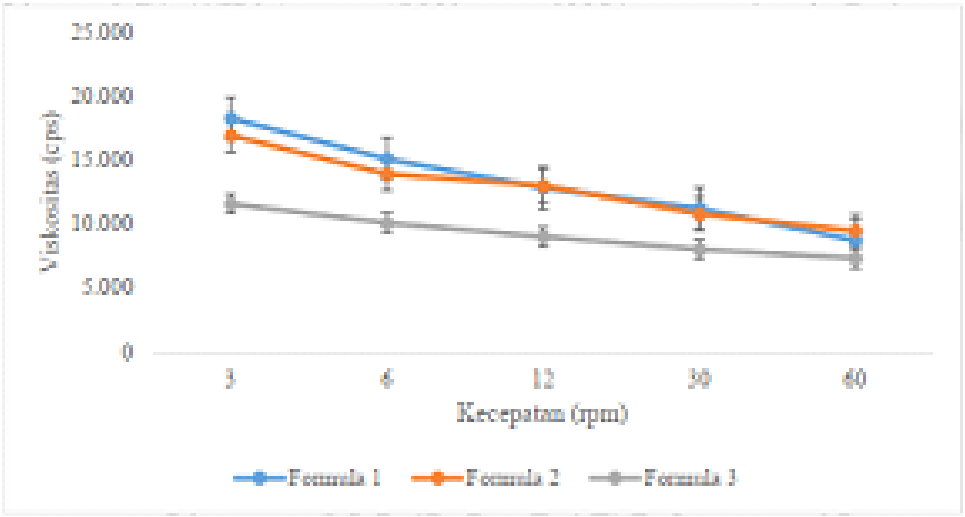

Figure 1: Result of viscosity test for Gel Peel-off mask preparations methanol extract of Duwet (*Syzygium cumini*) leaves.

active ingredient of duwet leaf methanol extract with the results of the viscosity test,

data analysis was carried out using One-way ANOVA with (degree of confidence) = 0.05. Viscosity results at a speed of 30 rpm obtained significant results (Sig.) 0.000 < α . So it can be concluded that there is a significant difference between the increase in the concentration of the methanol extract of duwet leaves in the peel-off gel mask preparation and the viscosity value. Data analysis can be continued using LSD (Least Significant Difference) and the results are shown in table 1.

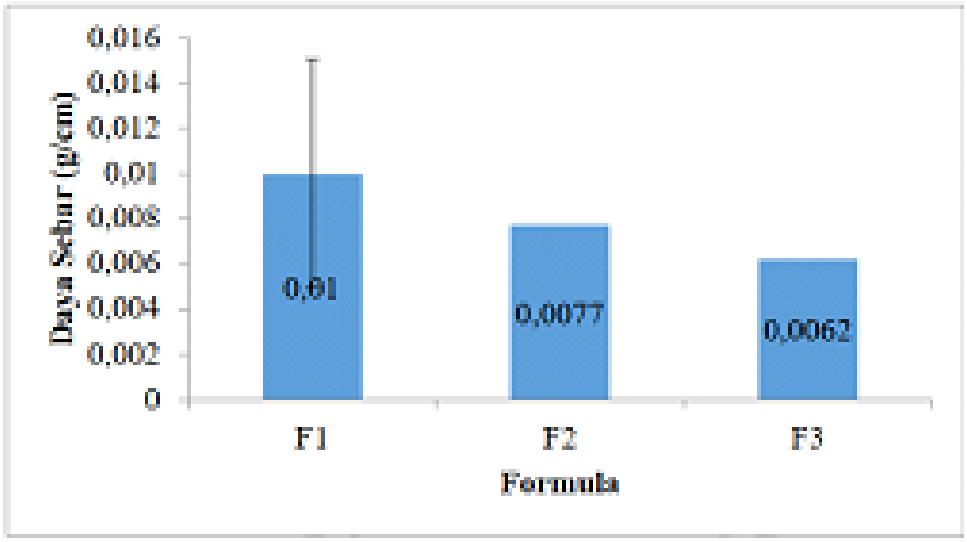

Figure 2: Histogram of the Spreading Power of the Peeloff Gel Mask Preparation of Duwet (*Syzygium cumini*) Leaf Methanol Extract.

The picture above shows that as the concentration of the methanol extract of duwet leaves increases in the peel-off gel mask preparation, the spreadability of the preparation decreases. The average diameter of each formula without the addition of load resulted in formula 1 (4.6 cm) which did not enter the range of good dispersion power, while formula 2 (5.2 cm) and formula 3 (5.4 cm) entered the power range. good spread of 5-7 cm [9]. To determine the effect of increasing the active ingredient of duwet leaf methanol extract with the results of the spreadability test, data analysis was carried out using One-way ANOVA with (degree of confidence) = 0.05. The results of the dispersion test showed significant results (Sig.) 0.352 > α . So it can be concluded that there is no significant difference between the increase in the concentration of the methanol extract of duwet leaves in the peel-off gel mask preparation and the dispersion value. The analysis of the results was continued using LSD (Least Significant Difference) and the results showed that there were no significant differences between all formulas or series of levels.

The results of the drying time test based on the figure show that as the concentration of the methanol extract of duwet leaves increases, the drying time for the peel-off gel mask preparation is longer or increases. Data analysis used One-way ANOVA with

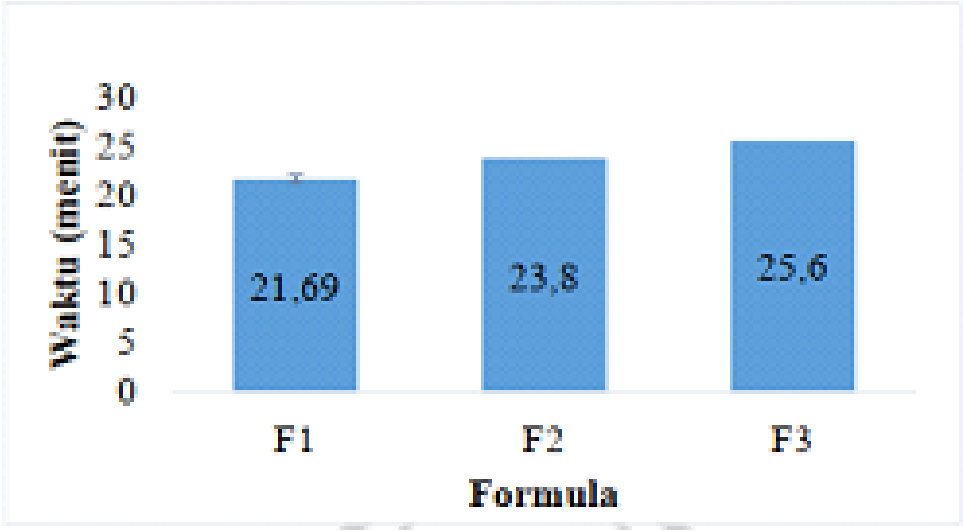

Figure 3: Histogram of Test Results Drying Time Preparation of Peel-off Gel Mask Methanol Extract of Duwet Leaves (*Syzygium cumini*).

(degree of confidence) = 0.05. The results of the drying time test showed significant results (Sig.) 0.000 < α . So it can be concluded that there is a significant difference between the increase in the concentration of the methanol extract of duwet leaves in the peel-off gel mask preparation of the methanolic extract of duwet leaves and the length of time it dries. The analysis of the results was continued using LSD (Least Significant Difference) and the results were obtained in table 1.

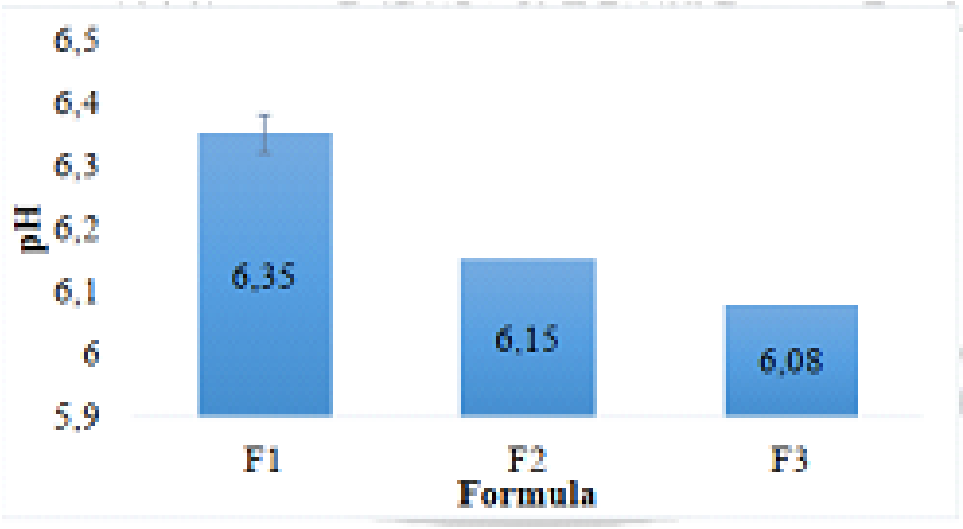

Figure 4: Histogram of pH Test Results Preparation of Peel-off Gel Mask Methanol Extract of Duwet Leaves (*Syzygium cumini*).

The pH test results based on Figure 4 above show that as the concentration of the methanol extract of Duwet leaves increases, the pH of the preparation decreases or becomes more acidic. To determine the effect of increasing the concentration of the active ingredient in the methanolic extract of duwet leaves, data analysis was

carried out using Oneway ANOVA with (degree of confidence) = 0.05. The results of pH measurements showed significant results (Sig.) 0.022 < α . So it can be concluded that there is a significant difference between the increase in the concentration of the methanol extract of duwet leaves in the peel-off gel mask preparation and the results of the pH test. The analysis of the results was continued using LSD (Least Significant Difference) and the results were obtained in table 1.

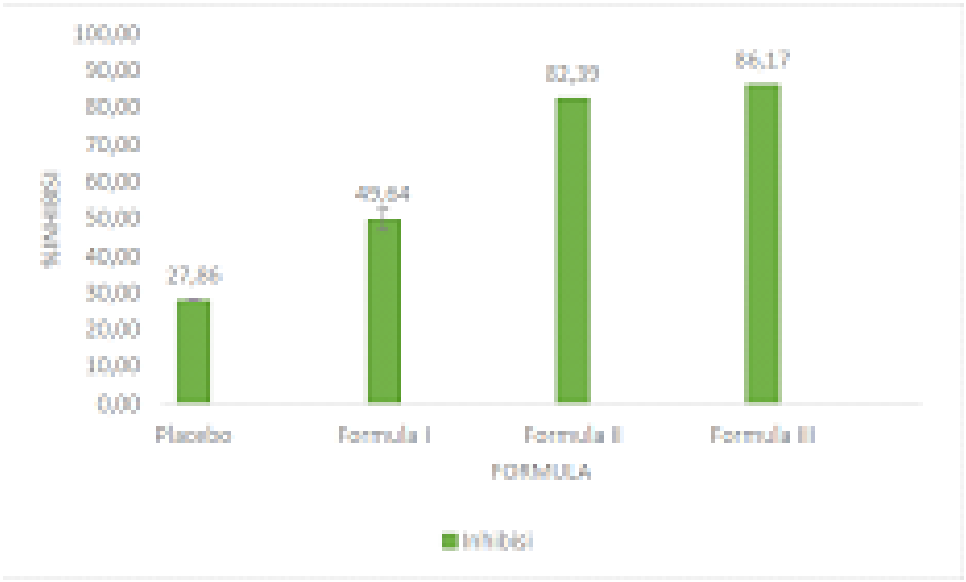

Figure 5: Histogram Percent Inhibition Preparation of Gel Mask Peel-off Methanol Extract of Duwet Leaf (*Syzygium cumini*)

The results of the measurement of the absorbance of the methanol extract of Duwet Leaf (*Syzygium cumini*) using the DPPH method with a maximum wavelength of 516.0 nm. The results of the one-way Anova analysis showed a significance value of <0.05, there was a significant difference in the percentage of inhibition between groups. Based on Tukey HSD, it can be seen in table 2 the difference between the inhibitory activities between the formulas.

TABLE 2: Results of Tukey HSD Test Antioxidant Activity of Duwet (*Syzygium cumini*) Leaf Peel-Off Gel Mask Preparation.

	placebo	Formula 1	Formula 2	Formula 3
Placebo	-	+	+	+
Formula 1	+	-	+	+
Formula 2	+	+	-	-
Formula 3	+	+	-	-

Keterangan : (-) not significantly different, (+) significantly different

The results of organoleptic observations at a temperature of 4^{o}C ± 2^{o}C and 40^{o}C ± 2^{o}C in 12 days (6 cycles). Based on table 4, it can be seen that all formulas underwent

Table 3: Results of Irritation Test Duwet (*Syzygium cumini*) Leaf Peel-Off Gel Mask Preparations with the HET-CAM Method.

	hemorrhage	coagulation	Mean Irritation Score	Irritation level
Postif Control	+	+	15,98	Strong Irritation
Negatif Control	-	-	0	Not Irritating
Formula 1	-	-	0	Not Irritating
Formula 2	-	-	0	Not Irritating
Formula 3	-	-	0	Not Irritating

Note : positive control : Sodium Lauryl Sulfate; Negative control : aquadest

Table 4: Results of Organoleptic Observation Freeze Thaw cycling Stability Test Preparation of Duwet Leaf Peel-Off Gel Mask (*Syzygium cumini*).

Test	Cycle											
	1		2		3		4		5		6	
	4°C	40°C	4°C	40°C	4°C	40°C	4°C	40°C	4°C	40°C	4°C	40°C
Organoleptic												
F 1	S	S	S	S	US	US	US	US	US	US	US	US
F 2	S	S	S	S	US	US	US	US	US	US	US	US
F 3	S	S	S	S	US	US	US	US	US	US	US	US
Homogeneity												
F 1											Homogeneous	
F 2											Homogeneous	
F 3											Homogeneous	

Note : S : Stable; US : Unstable

organoleptic changes in the 3rd cycle. This is indicated by the change in the color of the preparation from yellowish green to brownish-green. However, the jasmine aroma and soft texture of the peel-off gel mask preparation remained stable until the 12th cycle. The observation of the homogeneity of the peel-off gel mask preparation was carried out at the end of the 6th cycle or the 12th day. Based on the results of the homogeneity observations in Figure 5.1 of the three formulas, the stability of the homogeneity of all formulas was good and there was no phase separation the same as the homogeneity of the 12th starting preparation.

Based on table 5, it can be seen that all formulas at temperatures of 30°C±2°C and 40°C±2°C were organoleptically unstable. This is indicated by the change in the color of the preparation from yellowish green to brownish green. However, the jasmine aroma and soft texture of the peel-off gel mask preparation remained stable. While the formulas were stored at 4°C±2°C, the organoleptics remained stable for up to 1 month of storage. The preparation remains yellowish green, jasmine-scented, and the

Table 5: Results of Observation Real Time Stability Test Preparation of Duwet Leaf Peel-Off Gel Mask (*Syzygium cumini*).

		Temperature	
	30°C	40°C	4°C
Organoleptic			
F 1	US	US	S
F 2	US	US	S
F 3	US	US	S
Homogeneity			
F 1	H	H	H
F 2	H	H	H
F 3	H	H	H

Keterangan : S : stable; US : Unstable; H : Homogeneous

texture remains soft. The homogeneity observation also shows that from the three temperatures, all formulas have good homogeneity stability and no phase separation occurs.

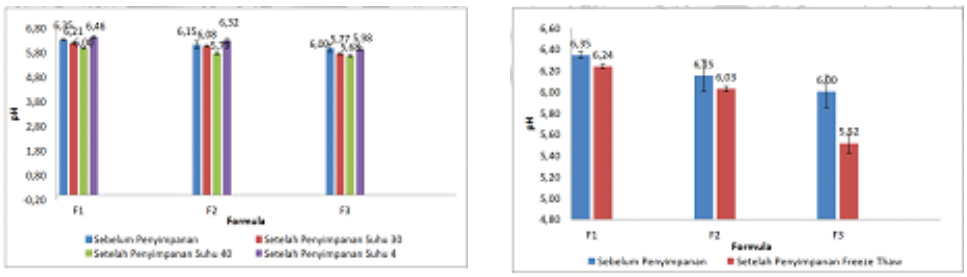

Figure 6: Comparison of pH Before and After Freeze Thaw (left) and Real Time (right) Preparation of Peel-Off Gel Mask Duwet Leaf Extract (Szyzygium cumini).

Based on Figure 6, it can be seen that there was a decrease in the pH value before and after the stability test was carried out using the freeze-thaw method. The results of data analysis using the paired T-test method with (degree of confidence) = 0.05. The results obtained for formula 1 p-value = 0.026 < αwhich means there is a significant change before and after storage. Meanwhile, for formulas 2 and 3, the p > values were 0.307 and 0.054, it can be concluded that there was no significant change in the pH values before and after storage. The test results using the Real Time method showed a decrease in the pH value. The results of the paired T-test on the Real-Time method concluded that at 4°C storage temperature there was no significant change in the three formulas. The results for the storage temperature of 30°C, formula 1 has a p value: 0.021 < , while for formulas 2 and 3 it has a p value > α, namely F2: 0.509; F3 : 0.088. These results prove that at 30°C storage temperature, formula 1 has a significant difference, while formulas 2 and 3 have no significant change in the pH value. The results for the

storage temperature of 40^{o}C, formula 1 has a p value: 0.03 < α, while for formulas 2 and 3 it has a p value > α, namely F2: 0.082; F3 : 0.088. These results prove at 40^{o}C storage temperature, formula 1 there is a significant difference, while formulas 2 and 3 there is no significant change in the pH value.

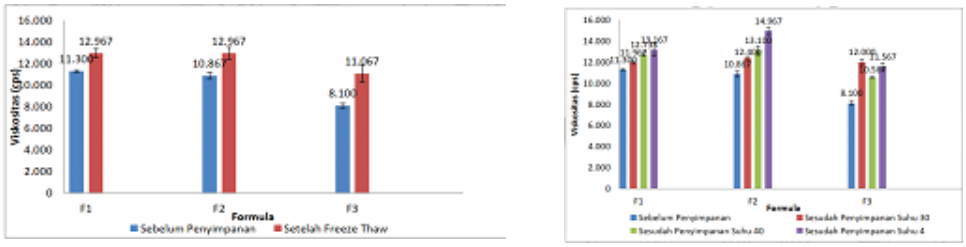

Figure 7: Comparison of Viscosity Before and After Freeze Thaw (left) and Real Time (right) Preparation of Peel-Off Gel Mask Duwet Leaf Extract (Szyzygium cumini).

Based on Figure 7, it can be seen that there was an increase in viscosity in the three formulas before and after the stability test was carried out using the freeze thaw method. analysis of paired T test data with (degree of confidence) = 0.05. The results obtained after the paired T-test were, formula 1: 0.012 < α; formula 2: 0.040 < α ; formula 3: 0.013 < α . It was concluded that the three formulas had significant changes after the freeze thaw stability test was performed. In the Real Time Test, it can be seen that there is an increase in viscosity in the three temperature treatments and the three formulas in it. Paired T-test results, obtained p-value results for each storage temperature. Storage temperature of 4^{o}C the three formulas have p < α values, namely F1: 0.023; F2 : 0.007 ; F3 : 0.001. This concluded that storage at 4^{o}C had significant changes in the three formulas. The results for the storage temperature of 30^{o}C, the three formulas had p < values, namely F1: 0.002; F2 : 0.012 ; F3 : 0.002. This concluded that storage at a temperature of 30^{o}C there were significant changes in the three formulas. The results for the storage temperature of 40^{o}C, the three formulas have a p value < , namely F1: 0.002; F2 : 0.023 ; F3 : 0.001. This concluded that storage at a temperature of 40^{o}C there were significant changes in the three formulas.

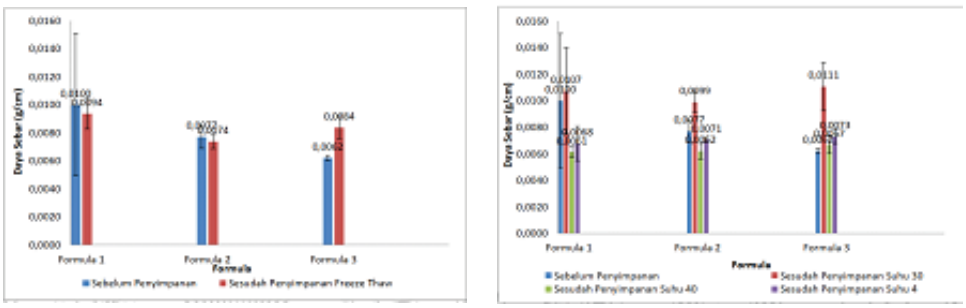

Figure 8: Comparison of Spreading Power Before and After Freeze Thaw (Left) and Real Time (right) Preparation of Peel-Off Gel Mask Duwet Leaf Extract (Szyzygium cumini).

The dispersion test on the peel-off gel mask preparation was carried out on the 12th day or on the 6th cycle. Based on Figure 8, it can be seen that formulas 1 and 2 have decreased dispersion. While in formula 3 there is an increase. Analysis of paired T-test data obtained a significance value of formula 1: 0.808 > α; formula 2: 0.267 > α ; formula 3: 0.065 > α. It can be concluded that there is no significant change in dispersion after the freeze thaw stability test. The results of the test using the Real Time method can be seen at storage temperatures of 40˚C and 4˚C that the dispersion power decreases. While at 30˚C storage temperature increased. Data analysis using paired T-test with (degree of confidence) = 0.05, the results obtained significant values at 4^{o}C storage temperature of the three formulas p>α , namely F1: 0.331; F2 : 0.281 ; F3 : 0.086. This concluded that there was no significant change in storage at 4^{o}C in the three formulas. The results for the storage temperature of 30^{o}C, formulas 1 and 2 have p > values, namely F1: 0,562; F2 : 0.077. This concluded that there was no significant change in storage at 30oC in formulas 1 and 2. Meanwhile, the p value of formula 3 < α, which was 0.039. This concludes that there is a significant change in storage at 30^{o}C in formula 3. The results for storage at 40^{o}C, the three formulas have p > α values, namely F1: 0.337; F2 : 0.093 ; F3 : 0.401. This concluded that there was no significant change in storage at 40^{o}C in the three formulas.

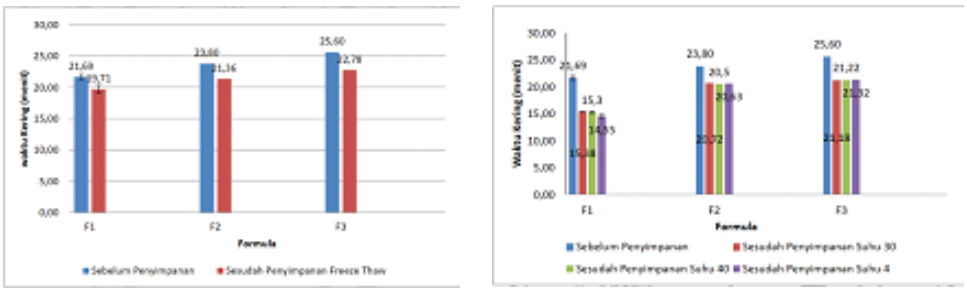

Figure 9: Comparison of Drying Time Before and After Freeze Thaw (left) and Real Time (right) Preparation of Duwet Leaf Extract Peel-Off Gel Mask (Szyzygium cumini).

Examination of the drying time on the peel-off gel mask preparation was carried out on the 12th day or on the 6th cycle. Based on Figure 9 above, it can be seen that there is a decrease in drying time in the three formulas. This shows that the peel-off gel mask time is getting faster after the freeze thaw stability test. Data analysis using paired t test obtained p value, formula 1: 0,292 > α; formula 2: 0.025 < α; formula 3: 0.022 < α. It can be concluded that there is no significant difference in formula 1, while formulas 2 and 3 have significant differences after the freeze thaw stability test was performed. Tests using the Real Time method showed that the three storage temperatures decreased for drying time. This decrease means that the time for the peel-off gel mask to dry is faster. Besides being processed in graphical form, data analysis

using paired T-test with (degree of confidence) = 0.05 was also carried out. This was done to determine the effect of variations in the concentration of the active ingredient, namely Duwet (*Syzygium cumini*) leaf extract. After the paired T-test was carried out, the p-values for each storage temperature were obtained. Storage temperature of 4^oC the three formulas have p < αvalues, namely F1: 0.003; F2 : 0.008 ; F3 : 0.008. This concluded that storage at 4^oC had significant changes in the three formulas. The results for the storage temperature of 30^oC, the three formulas have p < α values, namely F1: 0.001; F2 : 0.018 ; F3 : 0.007. This concludes storage at a temperature of 30^oC there is a significant change in the three formulas. The results for the storage temperature of 40^oC, the three formulas have p < α values, namely F1: 0.001; F2 : 0.009 ; F3 : 0.008. This concluded that storage at a temperature of 40^oC there were significant changes in the three formulas.

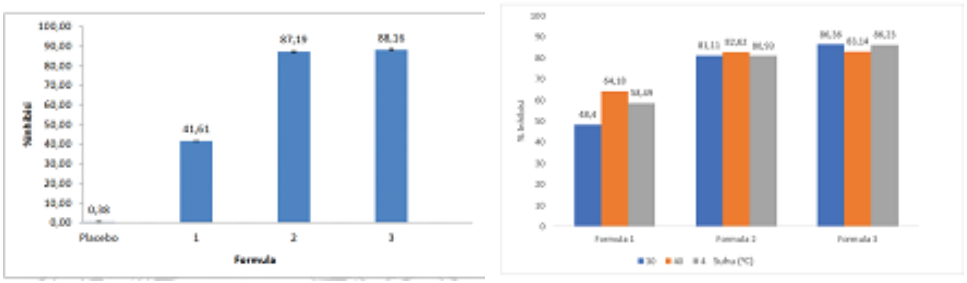

Figure 10: Percent Inhibition Before and After Freeze Thaw (left) and Real Time (right) Preparation of Peel-Off Gel Mask Duwet Leaf Extract (Szyzygium cumini).

Measurement of the absorbance of antioxidant activity of the peel-off gel mask with the active ingredient of duwet leaf extract after the freeze thaw test using methanol p.a spectrophotometrically at a maximum wavelength of 516.0 nm. The results of the T-test obtained were p-values (0.000) < 0.05 (-value) for placebo, and p-values > 0.05 for formulas 1, 2, and 3, namely F1: 0.230; F2 : 0,504 ; F3 : 0.356. It is concluded that there is a significant difference in placebo. Meanwhile, for formulas 1, 2, and 3 there was no significant difference after the freeze thaw test. measurement of the absorbance of antioxidant activity from peel-off gel masks with the active ingredient of duwet leaf extract after the real time test was carried out at a temperature of $40^oC \pm 2^oC$, at room temperature $30^oC \pm 2^oC$, and at a low temperature of $4^oC \pm 2^oC$, using methanol pa by spectrophotometry at a wavelength maximum 516.0 nm. The results of the T test at a temperature of 30oC obtained p values (0.000) < 0.05 (value) for placebo, and p values > 0.05 for formulas 1, 2, and 3, namely F1: 0.817; F2 : 0.849 ; F3 : 0.919. It is concluded that there is a significant difference in placebo. Meanwhile, for formulas 1, 2, and 3, there was no significant difference after the real time test at 30˚C. The results of the paired T test by comparing the antioxidant activity before and after the real time test at a

temperature of 40˚C in all formulas obtained p value (0.000) < 0.05 (value) for placebo, and p value > 0.05 for formula 1, 2, and 3, namely F1 : 0.091 ; F2 : 0.974 ; F3 : 0.202. It is concluded that there is a significant difference in placebo. Meanwhile, for formulas 1, 2, and 3 there was no significant difference after the real time test at 40˚C. The results of the paired T test by comparing the antioxidant activity before and after the real time test at a temperature of 4˚C in all formulas obtained p value (0.000) < 0.05 (value) for placebo, and p value > 0.05 for formula 1, 2, and 3, namely F1 : 0.200 ; F2 : 0.831 ; F3 : 0.977. It is concluded that there is a significant difference in placebo. Meanwhile, for formulas 1, 2, and 3 there was no significant difference after the real time test at 4˚C.

4. Discussion

In the manufacture of this peel-off mask, the active ingredient used is methanol extract of duwet leaves in a concentration of 1% as Formula 1, 2% as Formula 2 and 3% as Formula 3. polyvinyl alcohol (PVA) based film causes occlusion and mask tensor effect [10]. Polyvinyl alcohol is used as a film former which is widely used in topical preparations because it is biodegradable and biocompatible. Polyvinyl alcohol can produce a gel that dries quickly and forms a film that is transparent, strong, plastic and adheres well to the skin [11]. The gelling agent used in this formula is the carbomer. Carbomer is a gelling agent that absorbs liquid so that the liquid is retained and forms a gel mass. Carbomer is a class of synthetic polymers that produce a more transparent system and better viscosity. The carbomer was chosen because it is hydrophilic, which makes it easily dispersed in water at a low concentration of 0.5-2.0% and has a suitable viscosity as a gel base. Carbomer has a better consistency of active substance release than other gelling agents [12]. To form a gel mass from carbomer, an alkalizing agent is needed, namely TEA. TEA or triethanolamine is a neutralizing base that can adjust the pH so that the carbomer can enter the pH range which can form a gel mass at pH 6-7 [13]. Furthermore, the material used is propylene glycol as a plasticizer and humectant. Humectants function to maintain the stability of the preparation by absorbing moisture from the environment and reducing evaporation of water from the preparation [11]. Propylene glycol is used as a plasticizer to reduce the viscosity of the preparation in a formulation [14]. The preparation contains a lot of water so it is very easily contaminated by bacteria and fungi, so preservatives are added in the preparation. The preservative used is methyl paraben. Methyl paraben is effective over a wide pH range and has a broad spectrum of antimicrobial activity [14]. In the manufacture of peel-off gel mask preparations at the stirring stage with a homogenizer, PVA undergoes foaming because

PVA itself can be foam forming which can increase the mass of the peel-off gel mask [15]. Because of this, an additive is needed as an anti-foaming, namely dimethicone. To prevent oxidation from the duwet leaf extract itself, synthetic antioxidants are added, namely Na metabisulfite. Furthermore, to add aroma to the peel-off gel mask, corrigen odoris was added, namely fragrance jasmine or jasmine.

The first characteristic test was organoleptic observation on the peel-off gel mask preparation of methanol extract of duwet leaves which included color, texture and aroma. The results of organoleptic observations of the three formulas and their replication, in formula 1 it is yellowish green then for formulas 2 and 3 the same color is darker than formula 1 which is brownish green. The increase in the intensity of the darker color is caused by the methanol extract of duwet leaves which is dark green in color. The texture and aroma of the three formulas and their replication are the same, namely soft texture and jasmine-scented. The second characteristic test is the homogeneity test. This homogeneity test aims to see and know the mixing of the components of the preparation. This ensures that the active substances contained in it have been distributed evenly [16]. Judging from the observation of the homogeneity of all the formulas on the glass plate the result is homogeneous because there are no coarse grains or substances that do not blend. Homogeneity is indicated by the absence of coarse and separating particles in the preparation [17]. The third characteristic test is the viscosity test. Viscosity testing aims to determine the value of the viscosity of a substance. The higher the viscosity value, the higher the viscosity level of the substance [18]. The viscosity results obtained can be seen in Figure 1. Based on the viscosity requirements of the preparation stated that the viscosity is in the range of 4000-40,000 cps according to the specifications of the preparation and it can be concluded that all formulations of peel-off gel mask preparations are methanol extracts [19]. Duwet leaves meet the requirements of good viscosity. Based on the viscosity results obtained, formula 3 has the lowest viscosity compared to formulas 1 and 2 because it contains the most duwet leaf methanol extract. Duwet leaf methanol extract is thick but still a little runny because it contains water, so formula 3 contains a lot of water too. The higher the level of viscous extract used, the more the amount of water so that the viscosity decreases. In the results of statistical analysis Oneway annova viscosity test obtained results as shown in table 1.

The fourth characteristic test is the dispersion test. In testing the spreadability of the peel-off gel mask, which is to see the ability to spread over the skin surface when used [20]. The results of the dispersion measurement can be seen in Figure 2. Based on the results of the average observation of each formula without additional load, the results

of formula 1 (4.6 cm) are not included in the good dispersion range, while formula 2 (5.2 cm) and formula 3 (5.4 cm) is included in a good dispersion range of 5-7 cm [9]. Spreadability is inversely proportional to viscosity, where the increasing viscosity of a preparation, the lower the dispersion power [21]. The results of the statistical test One-way annova dispersion obtained a significance value (Sig.) 0.352 > α or can be seen in table 1. So there is no significant difference between the increase in the concentration of methanol extract of duwet leaves in the peel-off gel mask preparation and the dispersion value.

The fifth characteristic test is the drying time test. The drying time test aims to find out how long the mask dries on the skin surface [17]. The results of the drying time in Figure 3 show that the drying time of the peel-off gel mask preparation falls into the range and specification of 15-30 minutes [22]. Based on the figure, it can be seen that formula 3 has a longer drying time than formulas 2 and 1, formula 2 which has a longer drying time than formula 1. The more peel-off gel masks, the longer the drying time. The results of the peeling of the film on the peel-off gel mask preparations both formulas 1, 2 and 3 that have been made are easy to peel off after drying and when removed from the skin it is rather easy to tear because the film layer of PVA formed is not optimal. The results of statistical data analysis Oneway annova drying time test obtained a significance value (Sig.) 0.000 < α. So the result is that there is a significant difference between the increase in the concentration of the methanol extract of duwet leaves in the peel-off gel mask preparation and the length of time it dries. To find out which formulas were significantly different, the LSD (Least Significant Difference) test was carried out as shown in table 1.

Testing the pH value of the peeloff gel mask preparation of methanol extract of Duwet (*Syzygium cumini*) leaves. Tests are carried out to determine whether the preparation is suitable for use and is safe and does not cause skin irritation [16]. It can be seen from the results of Figure 4 that all preparations are in the good pH range of topical skin preparations and are included in the dosage specifications, namely 4.5-8.0. pH range of the preparation should follow the skin pH, which ranges from 4.5 to 6.5 [22]. Furthermore, it can be seen in the histogram graph of Figure 4 that the increasing concentration of the methanol extract of duwet leaves, the lower the pH value or the more acidic. This is because the pH of the methanol extract of Duwet leaves itself has an acidic pH of 3.75. This acidic pH is due to the presence of betulinic acid and crategolic acid compounds in the duwet leaf itself [23]. The results of statistical data analysis One-way annova pH test obtained a significance value (Sig.) 0.022 < α. So the result is that there is a significant difference between the increase in the concentration

of the methanol extract of Duwet leaves in the peel-off gel mask preparation and the results of the pH test. The LSD (Least Significant Difference) test is found in table 1, meaning that formulas 1, 2 and 3 have significant differences in drying time with p value = 0.022 < α. However (F2=F3) did not have a significant difference in pH.

the results of the antioxidant activity test on the methanol extract of duwet leaves with the DPPH method at a maximum wavelength of 516.0 nm showed that the % inhibition value was increasing. The IC50 value of the methanol extract of duwet leaves obtained was 3.32 ppm. When compared with the table of antioxidant power intensity (Figure 5), the IC50 value which has a value range of 1-50 ppm has a very strong antioxidant activity, so that the methanol extract of duwet leaves has the potential to be used as an external source of antioxidants. Antioxidant activity in Duwet leaves (*Syzygium cumini*) contains phenolic compounds. These compounds are catechins and ferulic acid which act as antioxidant activity. The function of phenolic compounds is to prevent oxidative stress caused by free radicals that cause cell damage that causes aging and various [24]. Based on the data above, it is known that with increasing levels of the active ingredient of Duwet leaf methanol extract, it can increase the inhibition value in preparations that will increase the ability to inhibit free radicals. The results of this study indicate that the peel-off gel mask with the active ingredient of duwet leaf has the potential as a preparation that has an antioxidant effect with increasing inhibition at increasing concentrations in each formula due to the presence of phenolic compounds, namely ferulic acid and catechins [24].

The irritation test was carried out using the HET-CAM method. This test is carried out because because it is made from methanol extract of duwet leaves, methanol itself can cause dry, cracked skin, and dermatitis or irritation. The positive control used was SLS and as the negative control was distilled water. SLS was used as a positive control because sodium lauryl sulfate can damage proteins in the skin and cause damage to the integrity of the skin barrier so that it can cause irritant contact dermatitis [25]. The results of the irritation test on all peel-off gel mask formulas of duwet leaf extract showed that variations in the levels of the active ingredients did not show any change in CAM or did not cause irritation. This can happen because the methanol contained in the extract has disappeared during the evaporation process and has been proven by a flame test. So it can be said that the variation in the levels of methanol extract of duwet leaves (1%,3%,5%) does not irritate the skin and is safe to use.

The results of organoleptic observations on the stability test of the freeze thaw method, it was found that the peel-off gel mask preparation was unstable starting from the 3rd cycle. The color of the preparation changed to darker than before storage.

This can be caused by a browning reaction [26]. Although synthetic antioxidants have been added, namely sodium metabisulfite, it still does not prevent browning of the preparations. According to [14], sodium metabisulfite when dissolved in water and becomes a solution of sodium metabisulfite can decompose or oxidize in air, especially if there is heating. Because the freeze thaw method uses a high temperature of 40˚C, there is a possibility of oxidation. The results of organoleptic observations in real time tests at 30˚C±2˚C and 40˚C±2˚C storage became unstable. This explains that the preparation also undergoes a browning reaction because sodium metabisulfite will decompose at hot temperatures. Meanwhile, in storage at 4˚C±2˚C, it remained stable until the end. The homogeneity test of the three formulas is the same as the initial preparation, because there is no phase separation of all components of the material mixed homogeneously so that the preparation remains stable during the real time storage test.

The results of the observation of the homogeneity stability test with the freeze thaw and Real Time methods obtained that the results of the three formulas and their replication were homogeneous. Gel preparations can be said to be homogeneous if the overall color equation is evenly distributed. In addition, it is not seen that the particles are different, it is also said that the preparation has good homogeneity [27].

The pH stability test using the freeze thaw method found significant differences in formula 1 but there was no significant difference in formulas 2 and 3. The three formulas when observed from the graph experienced a decrease in pH to become more acidic. pH stability test with Real Time method at 4˚C±2˚C, pH measurement results obtained after storage ± 30 days, formula 1 (6.46 ± 0.05), formula 2 (6.32 ± 0.05) and formula 3 (5.98 ± 0.06). The results of pH measurements on the 30^{o}C±2^{o}C temperature stability test were obtained after storage for ± 30 days, formula 1 (6.21 ± 0.02), formula 2 (6.08 ± 0.04) and formula 3 (5.77 ± 0, 03). The results of pH measurements on the temperature stability test of 40^{o}C±2^{o}C were obtained after storage for ± 30 days, formula 1 (6.00 ± 0.03), formula 2 (5.79 ± 0.05) and formula 3 (5.68 ± 0, 05). Based on these results, it is known that the three formulas that meet the criteria for a good pH for gel preparations are those that are close to the skin pH or range from 4.5 to 6.5 [27]. The results of the statistical test on the Real Time method data obtained a significance value of α in the group before and after the stability test at a temperature of 4˚C±2˚C, which means that there is no significant difference in pH. the temperature group 30˚C±2˚C and the temperature group 40˚C±2˚C it was found that there was a significant difference in formula 1 and no significant difference in formulas 2 and 3. The decrease in pH could

be due to hydrolysis of acidic compounds triggered by an increase in pH. temperature during storage [2].

Viscosity stability test after freeze thaw method and Real Time method showed an increase in viscosity. The results of the statistical test explained that there were significant differences but the three formulas had a viscosity that still entered the standard range of viscosity for topical preparations between 2,000-50,000 cPs [28]. This significant difference was indicated by the thicker each formula at the three storage temperatures. The longer the storage, the water content in the gel can evaporate and make the preparation thicker [29]. Another possibility can also be due to the retention of the solvent because it is absorbed by the gelling agent [21]. The thickening of the peel-off gel preparation is also influenced by PVA as a filming agent. This is because PVA has the ability to increase the viscosity of the gel and form an elastic film layer [30]The three formulas at each test temperature had a pseudoplastic flow type. This is indicated by a decrease in the viscosity value with an increase in the shear speed (rpm) [29].

The dispersion stability test after the freeze thaw method showed a decrease for formulas 1 and 2. While for formula 3 there was an increase. The real time test of the spreadability of the peel-off gel mask at storage temperatures of 40˚C and 4˚C decreased the spreadability. Meanwhile, at 30˚C storage temperature increased. The decrease in dispersion was influenced by the increase in viscosity. The thicker the viscosity, the lower the spreadability of the preparation [31]. For formula 3 which has increased, there is a possibility because the concentration of extract is higher, so the water content in formula 3 is still quite high. The results of the statistical test between the three formulas from the Freeze Thaw method obtained a significance value of more than . This means that there is no significant difference in dispersion before and after freeze thaw. The results of the statistical test of the Real Time method data have a p value > α , which means that there is no significant difference in the dispersion before and after real time. Except for formula 3, the temperature is 30˚C p < α , which means that there is a significant difference in the dispersion before and after real time. This is because the concentration in formula 3 is the most, so the water content in the preparation affects the spreadability.

The drying time stability test after the freeze thaw method showed the drying time of each formula, for formula 1 (19’56|| ± 0.63), formula 2 (21’22|| ± 0.28), and formula 3 (22’ 60|| ± 0.52). All three formulas meet the requirements of good drying time. A good drying time for a peel-off gel mask is around 15-30 minutes [22]. The results of statistical tests obtained formula 1 > α; formula 2 and formula 3 < α. It can be concluded that

there is no significant difference in formula 1, while formulas 2 and 3 have significant differences after the freeze thaw stability test was performed. The real time method found that the drying time of each formula at storage temperatures of 4˚C, 30˚C and 40˚C all formulas met the requirements of a good drying time. The results of data analysis with paired T-test showed the significance value of the three formulas < α, so there was a significant difference between before and after real time. This is because the real time storage period is 30 days, making the preparation thicker [29], which makes the drying time of the preparation faster because the water content decreases.

Antioxidant activity test after freeze thaw and real time storage. First, the antioxidant activity test studied were those treated with the freeze thaw test. This is to determine the effect of increasing and decreasing temperature during storage on the antioxidant activity of the peel-off gel mask preparation with duwet leaf extract. From the absorbance measurement of the peel-off gel mask after manufacture, it was found that the absorbance value was smaller than that of the DPPH blank, then the % inhibition of each formula was calculated. The results of the % inhibition of each formula obtained were placebo 0.38% ± 0.15, formula 1 41.6% ± 0.10, formula 2 87.19% ± 0.20, and formula 3 88.16% ± 0.53. The results of the T-test obtained for placebo are that there is a significant difference between before and after the freeze thaw test with a p value (0.000) <0.05 (value), and there is no significant difference for formulas 1, 2, and 3 before and after freeze thaw test with p value > 0.05. Next is the antioxidant test after being given real time test treatment at 4˚C ± 2˚C, 30˚C ± 2˚C, and 40˚C ± 2˚C. First at a temperature of 4˚C ± 2˚C. From the absorbance measurement of the peel-off gel mask after manufacture, the absorbance value was smaller than that of the DPPH blank, then the % inhibition of each formula was calculated. The results of % inhibition at a temperature of 4˚C ± 2˚C for each formula obtained were placebo (-13.96% ± 1.25), formula 1 (58.49% ± 0.24), formula 2 (80 .93% ± 0.11) and formula 3 (86.23% ± 0.08). Second, at 30˚C ± 2˚C, the results were placebo (-1.88% ± 0.12), formula 1 (48.40% ± 0.07), formula 2 (81.11% ± 0.06), and formula 3 (86.36% ± 0.03). Finally, at 40˚C ± 2˚C, the results were placebo (-4.88% ± 0.10), formula 1 (64.18% ± 0.10), formula 2 (82.62% ± 0.06), and formula 3 (83.14% ± 0.10). The results of the T test obtained for the placebo are that there is a significant difference between before and after the real time test on all temperature treatments with a p value (0.000) < 0.05 (value), and there is no significant difference for formulas 1, 2, and 3 on all temperature treatments before and after the real time test with p value > 0.05. There was a significant difference in the placebo between before and after the real time test was carried out because the placebo contained Na Metabisulfite, where Na Metabisulfite can decompose when

dissolved in water especially when there is heating which will cause it to lose its activity [14], so that causes a decrease in the value of % inhibition after the real time test. While for formulas 1, 2, and 3 there was no significant difference before and after real time, so it can be concluded that the peel-off gel mask duwet leaf extract did not experience a decrease in antioxidant activity on storage either by the Freeze Thaw or Real Time methods.

5. Conclusion

The results showed that the preparation produced a good characteristic with a drying time of fewer than 30 minutes and showed significant antioxidant activity with the highest percent inhibition in preparation with 3% duwet leaf extract. This preparation also had no irritating effect on CAM and was stable during storage using freeze-thaw and real-time method, chemically. Based on the results of this study, it can be concluded that duwet leaf extract has the potential to be used as an active for the anti-aging peel-off mask.

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