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Physical Stability and Antioxidant Activity of Peel-Off Gel Mask Ethanol Extract of Buas-buas Leaf (Premna serratifolia L.)

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ABSTRACT

This study aims to determine the physical stability and antioxidant activity of peel-off gel mask ethanol extract of buas-buas leaf (*Premna serratifolia* L.). The ethanol extract of buas-buas leaves was obtained by maceration using 70% ethanol solvent, then concentrated using a rotary evaporator to get a thick extract. Formulation EEDB into a peel-off gel mask with a concentration of each EEDB (FI: 1%, FII: 2%, FIII : 3%). The EEDB peel-off gel mask formula was tested for physical stability by cycling test method using a climatic chamber at a temperature of 40°C ± 2°C and 4°C ± 2°C for six cycles. The tests included organoleptic, homogeneity, viscosity, pH, spreadability, and drying time. An antioxidant test was carried out using the DPPH method. The results of the physical stability test on the three EEDB peel-off gel mask formulas (FI: 1%, FII: 2%, FIII: 3%) showed good physical stability because there was no significant difference during six storage cycles (P>0.05) with the results of the preparation have a characteristic aroma of oleum citri, brownish-green in color, have a thick, homogeneous texture, meet the requirements of pH, viscosity, spreadability, and drying time. The stability test results of the antioxidant activity of the peel-off gel mask EEDB in the three formulas showed no significant change (P>0.05). The antioxidant activity during six storage cycles with the highest average percent inhibition before and after the cycling test, namely in Formula III, was 77.20 ± 0.28%.

Keywords: Peel-off gel mask; ethanol extract of *Premna serratifolia* L. leaves; physical stability; antioxidant activity

INTRODUCTION

Due to ultraviolet radiation, free radicals can be formed (Suhartono, 2016). Continuous exposure to ultraviolet (UV) rays can cause skin damage such as spots due to pigmentation, loss of skin elasticity, wrinkles, and rough skin (Barel et al., 2009). Antioxidants can neutralize free radical particles (Percival, 1998). Antioxidants can neutralize free radicals and prevent body damage caused by free radicals by complementing the lack of electrons in these free radicals (Febrianti & Wahyuningsih, 2016). One of the natural ingredients that can be an antioxidant is buas-buas leaf *(Premna serratifolia* L.).

Research states that *Premna serratifolia* L. leaves contain secondary metabolites of flavonoids, saponins, tannins, and triterpenoids/steroids, where flavonoids are known to have properties as free radical scavengers or antioxidants, inhibitors of hydrolysis and oxidative enzymes, and work as an anti-inflammatory (Liya, 2016). Puspita, W., et al. (2020) stated that the ethanol extract of *Premna serratifolia* L. leaves a very high antioxidant activity with an IC_{50} value of 20.66 µg/mL. Antioxidants for facial skincare would be better formulated in a topical form than oral because the active substances will interact longer with facial skin (Sutriningsih et al., 2017). One of them is the formulation of a peel-off gel mask preparation.

The peel-off gel face mask is a face mask in the form of a practical gel because, after the preparation, dries can be removed directly from the facial skin (Syarifah et al., 2015). In addition, the peel-off gel facial mask refreshes, cleanse, moisturizes, and relaxes facial muscles (Sulastri & Chaerunnisa, 2017). Peel-off masks can improve skin problems such as wrinkles and acne and can also be used to close enlarged pores (Grace et al., 2015). The peel-off gel facial mask is superior to other facial mask preparations and can be formulated into an elegant cosmetic product. (Rahmawanty et al., 2015).

The stability of a cosmetic preparation is something that must be considered. This is important considering that preparation is usually produced in large quantities and takes a long time to reach consumers. Stability is the ability of a drug product to withstand the specifications applied throughout storage and use to ensure the identity, strength, quality, and purity of the product (Kuncari et al., 2014). Evaluation of stability by cycling test is carried out on preparations with different storage temperatures in specific time intervals to accelerate changes that usually occur

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Table 1. Formula for Peel Off Gel Mask Ethanol Extract of Buas-buas Leaves

Composition	Concentration (%)		
	FI	**FII**	**FIII**
Premna serratifolia L. ethanolic extract	1	2	3
PVA	8,75	8,75	8,75
Propylene glycol	10	10	10
Carbomer 940	0,5	0,5	0,5
Triethanolamine	0,5	0,5	0,5
Methyl paraben	0,2	0,2	0,2
Propyl paraben	0,1	0,1	0,1
Oleum citri	0,5	0,5	0,5
Aquadest add	100 g	100 g	100 g

under normal conditions. A cycling test simulates temperature changes every year and even every day during product storage (Suryani et al., 2017). Stable peel-off gel mask preparations are preparations that are still within acceptable limits during the period of storage and use. Based on this background, this study aimed to test the peel-off gel mask's physical stability and antioxidant activity formulated from ethanol extract of buas-buas leaves (*Premna serratifolia* L.).

METHODOLOGY

Tools and ingredients

The tools in this research are mortar and stamper, analytical balance, stir bar, hot plate (Maspion), aluminum foil, beaker glass (Pyrex), watch glass, pH meter (Handylab pH 11/SET), viscometer (Rion VT-06), refrigerator (Sharp), rotary evaporator (Heidolph), UV-Vis spectrophotometer (Shimadzu), pH meter, measuring flask (Pyrex), measuring cup (Pyrex), micropipette, dropper, stopwatch. The ingredients in this study were *Premna serratifolia* L. leaf, 96% ethanol, methanol p.a, Carbomer 940, Triethanolamine, Propylene glycol, PVA, Methyl Paraben, Propyl Paraben, Oleum citri, Aquadest, DPPH.

Methods

Formulation of Peel-off gel mask of Buas-buas Ethanol Extract

The manufacture of a peel-off gel mask dissolves the extract in propylene glycol little by little until the extract is completely dissolved. Then in a separate place, PVA was developed with warm aquadest (80°C) until it expanded ultimately, then homogenized (container A). Furthermore, carbopol was developed in hot water then added triethanolamine, ground until homogeneous and transparent (container B). In another container (container C), dissolve the methylparaben and propylparaben into the propylene glycol. Then mix container B and container C successively into container A, then stir until homogeneous. Add the dissolved extract and oleum citri little by little, stir until homogeneous, and add aquadest up to 100 grams.

Evaluation of Physical Stability of Peel-Off Gel Mask Ethanol Extract of Buas-buas Leaves

The stability test was done by a cycling test method. The cycling test was carried out for six cycles. The preparation was stored at a cold temperature 4°C ± 2°C for 24 hours and then removed and placed at a temperature of 40°C ± 2°C for 24 hours; this process was counted as one cycle. The treatment was repeated for 6 cycles and observations were made with organoleptic parameters, homogeneity, pH, viscosity, dispersion, and drying time of the preparation (Suryani et al., 2017).

Testing the antioxidant activity of peel-off gel mask preparations ethanol extract of Buas-buas Leaves

Antioxidant testing of the peel-off gel mask preparation of ethanol extract of buas-buas leaves was carried out by inserting as much as 2 mL of peel-off gel mask preparation of ethanolic extract of buas-buas leaves, 2 mL of 0.1 mM DPPH, and 4 mL of methanol. Vortexed until homogeneous and read at a wavelength of 516 nm. The peel-off gel mask preparation was tested before and after being stored in the cycling test for six cycles.

RESULT AND DISCUSSION

This research determined used buas-buas leaf samples (*Premna serratifolia* L.) at the Biology Laboratory, Department of Mathematics and Natural Sciences, Tanjungpura University, Pontianak. Determination aims to determine the authenticity of the plants used in a study. The extraction method used in this research is the maceration method. Maceration was chosen

Table II. Results of Organoleptic

Formula	Organoleptic					
	Before Cycling Test			After Cycling Test		
	Color	Aroma	Tekxture	Color	Aroma	Tekxture
F I	Brownish Green	Typical Oleum citri	Thick	Brownish Green	Typical Oleum citri	Thick
F II	Brownish Green	Typical Oleum citri	Thick	Brownish Green	Typical Oleum citri	Thick
F III	Brownish Green	Typical Oleum citri	Thick	Brownish Green	Typical Oleum citri	Thick

Note : Gel Mask Peel Off Formula I (1% buas-buas leaf ethanol extract), Formula II (2% buas-buas leaf ethanol extract), and Formula III (3% buas-buas leaf ethanol extract)

Table III. Results of Homogeneity

Formula	Results of Homogeneity	
	Before *Cycling Test*	After *Cycling Test*
F I	Homogeneous	Homogeneous
F II	Homogeneous	Homogeneous
F III	Homogeneous	Homogeneous

Note : Gel Mask Peel Off Formula I (1% buas-buas leaf ethanol extract), Formula II (2% buas-buas leaf ethanol extract), and Formula III (3% buas-buas leaf ethanol extract)

Table IV. Result of pH

Formula	pH of Peel Off Gel Mask	
	Before Cycling Test	After Cycling Test
FI	5,65 ± 0,02	5,77 ± 0,11
FII	5,60 ± 0,12	5,71 ± 0,03
FIII	5,54 ± 0,01	5,64 ± 0,16

Note : Gel Mask Peel Off Formula I (1% buas-buas leaf ethanol extract), Formula II (2% buas-buas leaf ethanol extract), and Formula III (3% buas-buas leaf ethanol extract)

because it can extract compounds well and can prevent the decomposition of compounds that are unstable to heat (Murbantan et al., 2010). The maceration process of buas-buas leaves uses 70% ethanol as a solvent due to its polar nature, so it is expected that all types of flavonoids are extracted. The final result of the extraction process with this maceration method is a liquid extract that is then concentrated with a rotary evaporator, from 2100 grams of simplicia, a thick section of approximately 450 grams with a yield of 21.42%. Physical stability test for peeling off gel mask ethanol extract of *Premna serratifolia* L. leaves included observation of organoleptic parameters, homogeneity, pH, viscosity, dispersion, and drying time of the preparation.

The organoleptic test is a physical parameter test to see the shape/texture, color, and aroma. An organoleptic test is done by looking at the color, smelling the aroma, and seeing the shape/texture of the natural gel made (Mumtihanah et al., 2015). The results of organoleptic examination in table II, show that the addition of ethanol extract of buas-buas leaves to the three formulas resulted in a peel-off gel mask preparation that was brownish green in color, had a characteristic aroma of oleum citri, and had a thick texture. The three preparations of this peel-off gel mask preparation resulted in the preparations being organoleptically stable both before and after the cycling test.

The homogeneity test smoothes the dispersed phase in the dispersing agent, the absence of secondary particle aggregation, the uniform and regular distribution of the dispersed phase, and the refining of large primary particles (Voight R., 1995). The results of the homogeneity test in table III show that the three peel-off gel mask formulas both before and after the cycling test did not show any coarse grains when the

preparation was applied to the transparent glass. The conclusion indicates that the peel-off gel mask preparation has a homogeneous arrangement.

The results of testing the pH of the peel-off gel mask preparation of ethanol extract of *Premna serratifolia* L. leaves can be seen in table IV, where temperature changes after the cycling test for six cycles affect the pH of the peel-off gel mask preparation. The pH value of the peel-off gel mask preparation of the three formulas after the cycling test increased compared to the pH value of the trial before the cycling test. This is influenced by environmental factors and the interaction of ingredients in the gel. The results of the statistical value showed the value of Sig. (2-tailed) 0.4226, this value was >0.05, so it could be concluded that there was no significant difference between the pH of the peel-off gel mask before and after the cycling test, and it was a stable peel-off gel mask. Based on the results of measuring the pH of the peel-off gel mask preparation before and after the cycling test, the pH of the practice is in a good acidity (pH) range for the peel-off gel face mask preparation, which is between 4.5-6.5 (Zhelsiana et al., 2016). The pH value should not be too acidic because it can irritate the skin, and it should not be too alkaline because it can make the skin scaly. (Dureja H. *et al.,* 2010).

The viscosity test aims to determine the resistance of a liquid to flow. Viscosity testing is an essential factor because it affects the parameters of the spreadability and the active substance of the gel mask preparation. In addition, a gel with an optimum viscosity will hold the active substance dispersed in the gel base and increase the consistency of the gel (Madan and Singh, 2010). The peel-off gel mask viscosity test results can be seen in table V, where there is a decrease in the peel-off gel mask viscosity in conditions after the cycling test. The reduction in viscosity can be caused by storage environmental conditions such as light and humidity. Less impermeable packaging can cause the gel to absorb moisture from the outside, thereby increasing the volume of water in the gel. The longer the storage period, the smaller the number of trapped air bubbles (Sihombing and Lestari, 2015).

The stability test process has a stable peel-off gel mask viscosity, which is seen from the results of the statistical value, which shows a Sig. (2-tailed) value of 0.1835, this value is > 0.05, which means there is no significant difference in the viscosity of the peel-off gel mask before and after the cycling test. The viscosity of the three peel-off gel mask formulas still meets the good viscosity range because the viscosity test results obtained are still within the range of good peel-off gel mask viscosity values, which is <300 dPas (Rahmawanty et al., 2015).

The dispersion test was carried out to know the ability to spread the gel on the skin layer. Based on the results of the dispersion test in table VI, it shows that the peel-off gel mask has a dispersion range of 5.10±0.10 cm-6.80±0.10 cm, where the peel-off gel mask preparation has good dispersion because it is following dispersion requirements for topical preparations are 5-7 cm (Tranggono et al., 2007). Gels with good spreadability will spread evenly on the skin to evenly distribute the effect. In addition, the viscosity of preparation also affects the extent of its distribution. The smaller the viscosity of preparation, the greater the spread. Spreadability is related to the spreading properties of the peel-off gel when used in topical preparations. The greater the spreadability, the wider the surface area of the skin in contact with the gel, and the active substance will be well distributed. A gel with great spreadability can be applied to a vast skin surface without excessive pressure.

The results of the test results of the spreadability of the peel-off gel mask preparations from the three formulas after the cycling test increased, this was due to a decrease in the viscosity of the peel-off gel mask after the cycling test conditions so that the peel-off gel mask preparation became more liquid and its spreadability increased. The results of the statistical values showed the value of Sig. (2-tailed) 0.5222, this value was >0.05. The conclusion was that there was no significant difference in the spreadability of the peel-off gel mask before and after the cycling test, and it was a stable peel-off gel mask.

Testing the speed of drying time in the peel-off gel mask preparation aims to determine the speed of the mask forming a film on the skin. Beringhs et al. (2013) reported that the principle of a peel-off mask is based on the ability to form a film that easily peels off when applied to the skin. The results of the dispersion test in table VII show that the peel-off gel mask has a spreading range of 25.41±0.16 minutes-28.62±0.13 minutes, where the three peel-off gel mask formulas still meet the drying time of the gel mask. Good peel-off is between 15-30 minutes (Beringhs et al., 2013). Polyvinyl alcohol in the mask provides a peel-off effect because it has adhesive properties so that it can form a film that is easy to peel off after drying. The proper concentration of PVA is the most critical factor influencing the performance of film formation in peel-off facial masks (Brick et al., 2014).

Table V. Viscosity Results

Formula	Peel Off Gel Mask Viscosity (dPas)	
	Before Cycling Test	**After Cycling Test**
FI	36,65 ± 2,31	35,70 ± 2,00
FII	37,60 ± 2,69	36,84 ± 2,31
FIII	39,51 ± 2,31	38,42 ± 1,21

Note : Gel Mask Peel Off Formula I (1% buas-buas leaf ethanol extract), Formula II (2% buas-buas leaf ethanol extract), and Formula III (3% buas-buas leaf ethanol extract)

Table VI. Spreadability Test Results

Formula	Spreadability of Peel Off Gel Mask (cm)	
	Before Cycling Test	**After Cycling Test**
FI	6,20 ± 0,02	6,80 ± 0,10
FII	5,80 ± 0,06	6,30 ± 0,15
FIII	5,10 ± 0,10	5,80 ± 0,12

Note : Gel Mask Peel Off Formula I (1% buas-buas leaf ethanol extract), Formula II (2% buas-buas leaf ethanol extract), and Formula III (3% buas-buas leaf ethanol extract)

Table VII. Drying Time Test Results

Formula	Drying Time of Peel Off Gel Mask (minutes)	
	Before Cycling Test	**After Cycling Test**
FI	27,20 ± 0,11	28,62 ± 0,13
FII	26,25 ± 0,03	27,64 ± 0,20
FIII	25,41 ± 0,16	26,77 ± 0,42

Note : Gel Mask Peel Off Formula I (1% buas-buas leaf ethanol extract), Formula II (2% buas-buas leaf ethanol extract), and Formula III (3% buas-buas leaf ethanol extract)

Table VIII. Test Results of Antioxidant Activity

Formula	Percent Inhibition ± (%)	
	Before Cycling Test	**After Cycling Test**
FI	52,41 ± 0,26	51,76 ± 0,13
FII	65,18 ± 0,52	64,07 ± 0,20
FIII	77,63 ± 0,13	76,77 ± 0,42

Note : Gel Mask Peel Off Formula I (1% buas-buas leaf ethanol extract), Formula II (2% buas-buas leaf ethanol extract), and Formula III (3% buas-buas leaf ethanol extract)

The results of the drying time of the peel-off gel mask preparations from the three formulas after the cycling test increased, this was due to a decrease in the viscosity of the peel-off gel mask after the cycling test conditions so that the peel-off gel mask preparation became more liquid and the drying time became longer. The results of the statistical value showed the value of Sig. (2-tailed) 0.5285, this value was >0.05, so it could be concluded that there was no significant difference in the drying time of the peel-off gel mask before and after the cycling test, and it was a stable peel-off gel mask.

The antioxidant activity test used in this study was the DPPH method (2,2-Diphenyl-1-Pikrylhydrazil). The DPPH method is a simple, fast, sensitive, and reproducible method for testing antioxidant activity. DPPH is a stable free radical at room temperature and is often used to assess the antioxidant activity of several compounds or extracts of natural ingredients. This method is suitable for evaluating activity antioxidants from ethanol extract of buas-buas leaves. The principle of the DPPH test is to remove color for antioxidants that directly reach the DPPH radicals by monitoring the absorbance using a UV-Vis

spectrophotometer. The DPPH radical with concentrated organic nitrogen is a stable free radical with a dark purple color that becomes a yellow color when reduced to a non-radical form by antioxidants (Bendra, 2012).

The antioxidant activity test results in table VIII show that the peel-off gel mask has a percent inhibition value ranging from 51.76±0.13% - 77.63±0.13%. Each formula's DPPH free radical inhibitory activity before and after the cycling test conditions increased along with the increase in the concentration of the extract contained in the mask formula. This is follows the research conducted by Hanani et al. (2005), which stated that the inhibition of DPPH radicals increased with increasing extract concentration. Regina et al. (2008) noted that the higher the concentration, the higher the antioxidant activity.

The antioxidant activity test showed a decrease in the antioxidant activity of the peel-off gel mask. However, there was no significant difference before and after the cycling test with a Sig. (2-tailed) value of 0.0820. The highest average percentage of inhibition before and after the cycling test, namely in Formula III, was 77.20±0.28%. The decrease in antioxidant activity of the peel-off gel mask preparation after storage can be influenced by environmental factors such as light which can cause the oxidation process to reduce the antioxidant activity of the peel-off gel mask preparation. Then the treatment at the time of sample preparation until the peel-off gel mask preparation can also affect the decrease in antioxidant activity. The poor treatment makes the practice more in contact with the environment to reduce the antioxidant activity of the preparation.

CONCLUSION

The conclusion based on the results of the research show of the physical stability test on the three peel-off gel mask formulas of ethanol extract of *Premna serratifolia* L. leaves (FI: 1%, FII: 2%, FIII: 3%) showed good physical stability because there was no difference significant for six storage cycles (P>0.05) with the result that the preparation has a characteristic aroma of oleum citri, brownish-green in color, has a thick, homogeneous texture, meets the requirements of pH, viscosity, spreadability, and drying time. The stability test results showed the antioxidant activity of the peel-off gel mask ethanol extract of *Premna serratifolia* L. leaves. The three formulas showed no significant change (P>0.05) in antioxidant activity for six storage cycles, with the highest average percent inhibition before and after the cycling test, namely in Formula III is 77.20 ± 0.28%.

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