

Antioxidant activity and irritation test of peel off gel mask of ethanol extract of pedada fruit (*Sonneratia caseolaris*)

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Abstract. Pedada (*Sonneratia caseolaris*) is a mangrove plant that lives in brackish waters and grows in many coastal areas, especially in Tanjung Jabung Timur, Province of Jambi. Pedada contains several bioactive compounds including flavonoids, luteolin, and luteolin 7-O- β -glucoside potential to stop the chain reaction of free radical formation in the body. Pedada fruit can be used as a cosmetic product such a peel off gel mask. This study aims to determine the effectiveness of peel off as an antioxidant and to determine a peel off gel mask have good physical properties, stable during storage and to can to determine the irritation of peel off gel mask. The ethanol extract of fruits used in the formulation of peel off gel mask each with a concentration of 5% with different bases (PVA, Carbomer 940, PVA+PVP). The physical properties of peel off gel mask include: organoleptic test, homogeneity, pH measurement, viscosity, flow properties, dispersion, drying time, cycling test, hedonic test, irritation test and antioxidant effectiveness test. The descriptively produced data states that the peel off gel mask have better physical properties and more stable during storage is the formula 1 with PVA base with IC50 386,23 of potency has weak antioxidant activity.

Keywords : Masker, Pedada, Antioxidant, Irritation, gel peel off.

1 Introduction

Pedada fruit (*Sonneratia caseolaris*) is a mangrove fruit that lives in the brackish water and grows widely in coastal areas, especially in Tanjung Jabung Timur coastal area of Jambi Province. Pedada fruit contains several bioactive compound as flavonoid, luteolin, luteolin-7-O-glucoside which which is able to stop the chain reaction of the formation of free radicals in the body [1].

In this study, the use of pedada fruit as a cosmetic product has been done, one of which is peel off gel mask to protect people from the danger of free radicals. his gel-shaped peel-off mask has several advantages including practical use, easy to clean and can be

removed like elastic membrane so that the face becomes smooth and tight [10].

Therefore it is used as a skin care product that gel peel off by using it for the public from the dangers of free radicals. This gel-shaped peel off mask has several practical advantages, it is easy to clean and can be removed like an elastic membrane [4].

The physical quality of peel off gel mask is influenced by the gelling agent used, such as: Polyvinyl Alcohol (PVA), Carbomer 940, Polyvinyl Piroolidone (PVP) which is a synthetic polymer. PVA is hydrophilic, not toxic, can produce a strong film layer, easy to spread and quickly dry. The mass of Carbomer 940 gel produced will give a good, clear and not cloudy

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appearance [5]. PVP as a suspending agent, stabilizers and ingredients that can increase viscosity for topical preparations [9].

The PVA, Carbomer 940 and PVP on the preparation of peel off gel mask containing pedada fruit extract is expected to produce a good preparation of peel off gel mask and has high antioxidant activity and no irritation during use.

2 Methodology

2.1 Materials

Pedada fruit obtained from Marine Village, Kuala Jambi, East Tanjung Jabung District, Province of Jambi. Pedada used is ripe fruit. The material used in this study is a 96% solutetanol, Aquadest, Carbomer 940, Sodium EDTA, glycerin, HPMC, benzoic acid, 2N NaOH, Polyvinylalcohol 72000, PVP (PolyVinylPiolidone) K30, Propylparaben, Methylparaben, Ethanol, Aqua Rosae.

The tools used in this study were stirring rod, beaker glass, measuring glass, pH meter, brookfield viscometer,

thermometer, analytical scale, evaporator cup, mortar, rotary evaporator, blender, water bath, petri dish, UV spectrophotometer, measuring flask, pipette volume, erlenmeyer, cuvette, test tube, test tube rack, refrigerator, oven, stirring rod, spatula, millimeter block paper, ruler, pipette.

2.2 Sampling and Preparation

Pedada (*Sonneratia caseolaris*) were taken as much as 50-55g. Samples are cleaned, washed, then chopped and blended.

2.3 Pedada extraction (*Sonneratia caseolaris*).

Pedada samples were extracted using maceration method with 96% ethanol solvent. Maseration is carried out by changing the solvent repeatedly extraction. The maceration results were filtered and then the extract of pedada ethanol extract was evaporated using a rotary evaporator at a temperature of 40°C and 60 rpm to obtain a thick ethanol extract of pedada fruit.

The design of the peel gel mask formula off the pedada ethanol extract on a different basis

Table 1. Formula of peel off gel mask

PVA Base (F1)	Carbomer 940 Base (F2)	PVA + PVP Base (F3)
Ethanol extract of pedada 5 gr	Ethanol extract of pedada 5 gr	Ethanol extract of pedada 5 gr
PVA 16 gr	Carbomer 940 0,5 gr	PVA 15 gr
Na EDTA 0,1 gr	Na EDTA 0,1 gr	PVP 5 gr
Glycerin 5 ml	Glycerin 5 ml	Glycerin 10 gr
HPMC 2 gr	HPMC 2 gr	Nipagin 0,2 gr
Nipagin 0,1 gr	Nipagin 0,1 gr	Nipazol 0,1 gr
Nipazol 0,1 gr	Nipazol 0,1 gr	Ethanol 15 ml
Benzoic Acid 0,1 gr	Benzoic Acid 0,1 gr	Aquadest ad 100 ml
Ethanol 5 ml	Ethanol 5 ml	
NaOH 0,2 gr	NaOH 0,2 gr	
Aquadest ad 100 ml	Aquadest ad 100 ml	

2.4 Making gel Peel Off Base PVA Gel Mask (F1)

PVA is dissolved with aquadest at 80°C until it expands, then grinded homogeneous (mass 1). HPMC

was developed with cold water (20 times of HPMC) then grinded homogeneous (mass 2). Benzoic acid is dissolved with distilled water (mass 3), add mass 2 and glycerin and crushed until homogeneous, add mass 3 and crushed homogeneous, add mass 1 homogeneous

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mixture, add Nipagin, Nipasol and Sodium EDTA which has been dissolved in distilled water. Add 70% ethanol and let stand for a moment and crushed. Check of pH, if pH is acidic, add 2N NaOH one drop pH of skin, add distilled water until forms a homogeneous gel (The mask gel peel off). Weigh ethanol extract of pedada, add the mask gel peel off and crushed.

2.5 Making Gel Peel Off Base PVA + PVP (F3) Gel Mask

PVA 0,15 g was developed (Mass 1) in a aquadest at 80°C, PVP 5 grams was developed in cold aquadest until it expands (Mass 2). Nipagins are 0.2% and Nipasol is 0.1% dissolved into 10 ml glycerol (Mass 3), stir until late. Periods B and C are inserted into material A and then stirred until homogeneous at a constant speed. 5 grams of ethanol extract added a little to a mixture of ingredients A, B and C while continuing to stir, added 96% ethanol as much as 15 ml and deodorized, stirred again, and put into container.

2.6 Antioxidant Activity Test of Peel Off Gel Mask

25 mg of the preparation was dissolved with methanol in a 25 ml volumetric flask then stirred until homogeneous to make 1000 ppm mother liquor. After that, several series of solution concentration were prepared from 1000 ppm mother liquor. Mix 2 ml of each solution of the peel off gel mask with 2 ml DPPH which has been dissolved with methanol, homogenized, then stored in a dark room for 30 minutes. Then absorbance was measured at a wavelength of 516 nm using a UV-Vis spectrophotometer.

Evaluation of Physical Properties of Peel Off Gel Masks of Pedada Fruit Extract. The evaluation of the physical properties of geel peel off mask included: organoleptic, homogeneity, measurement of pH, viscosity, flow properties, dispersion, drying time, cycling test, hedonic and irritation and antioxidant activity test.

Table 2. Recapitulation of Evaluation of Physical Properties of Peel Off Gel Masks

Characteristic	Basis PVA (F1)	Basis Carbomer 940 (F2)	Basis PVA+PVP (F3)	Parameter
Organoleptic	Light brown color, typical extract aroma, semi-solid form and thick consistency *)	Light brown color, distinctive aroma of the extract, semi-solid form and consistency rather foamy and slightly runny	Dark brown color, extract aroma, semi-solid form and very thick consistency *)	Dark brown color, typical extract aroma, semi-solid form and very thick consistency*
Homogeneity	Homogeneous*)	Inhomogeneous	Homogeneous*)	Homogeneous (Rowe et al, 2006)
pH	4,92*)	4,43*)	4,44*)	pH 4-6,8 (stawish, 1994)
Viscosity	6,567918Pa.s (6567,918 cps)	2,979966 Pa.s (2979,966 cps)	4.015881 Pa.s (4015,881 cps)	7100 – 83144 cps (Chandira et al, 2010).
Flow properties	Pesudoplastic *)	Dilatant*)	Pseudoplastic*)	non-Newton flow properties are plastic, pseudoplastic, or dilatant (Martin et al, 1993).
Spread power	6,39 cm*)	7,75 cm	4,36 cm	between 5-7cm (Yuliani, 2010).
Drying time	24,55 min *)	21,19 min *)	16,48 min *)	15-30 min (Vieira, 2009).
Irritation test (10 Panelist)	Not irritating	10 % irritant (redness)	10% irritant (redness)	No reaction (-), skin redness (+),

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ISBN: 978-979-98417-5-9 | Universitas Islam Indonesia

				reddish skin and itching (++) swollen skin (+++) (Voight, 1995: MOH RI, 1985)
Hedonic test - color - Sensation - cosiness	80 % like 70 % like 90% like *)	0% 0% 0%	20% like 30% like 10% like	Of the 10 panelists, they felt very like (4), rather like (3), disliked (2) and disliked (1)
Cycling test Organoleptic and homogeneity	Light brown color, typical extract aroma, semi-solid form and thick consistency *) Homogeneous *)	Light brown color, distinctive aroma of the extract, semi-solid form and consistency rather foamy and slightly runny Separate	Dark brown color, typical extract aroma, semi-solid shape and very thick consistency Homogeneous*)	Stable color, odor and shape changes do not occur (Rowe et al, 2006) Homogeneous (Rowe et al, 2006)
Sedimentation Degree (F) temperature of 25 °C temperature of 40 °C temperature of 40 °C	F = 1 F = 1 F = 1*)	F = 1 F = 0,8 F = 0,8	F = 1 F = 1 F = 1*)	F = 1 [1]
pH temperature of 25 °C temperature of 40 °C temperature of 4 °C	4,91 4,80 5,04*)	4,44 4,40 4,41*)	4,28 4,26 4,25*)	Deodorant roll on pH 4-6,8 (stawiski, 1994)
Spreading capability after storage	5,31cm *)	7,75 cm	3,61 cm	between 5-7cm (Yuliani, 2010).

Table 3. Test results of antioxidant activity

Results of antioxidant activity	Pedada Fruit Ethanol Extract	Formula 1 with the best PVA base from physical properties and stable storage	Parameter
IC50	881,25 ppm	386,23 ppm	Very strong <50 ppm, strong 50-100 ppm, medium 101-150 ppm, weak > 150 ppm (Kresnawaty et al, 2012)

3 Results and discussion

3.1 Organoleptic

Organoleptic tests were carried out to determine the color, odor and shape of peel off gel masks that had been made. Organoleptic results obtained from the three formulas of peel off gel mask are relatively the same, namely light brown color, distinctive aroma extract, semi-solid form, whereas different consistency is thick, very thick, slightly foamy and dilute, this is due to the influence of gelling agent viscosity used, where PVA viscosity, PVP is greater than the viscosity of carbomer 940 [9].

3.2 Homogeneity

Homogeneity test is carried out to determine uniformity of particles and even distribution of particles on the preparation. The homogeneity test results are the absence of solid particles contained in the gel peel off mask preparation from each formula. From the evaluation it is known that two formulas with PVA base and PVA + PVP are homogeneous where the active substance is evenly dispersed in the preparation. While one formula with carbomer940 base separation occurs because the pedada fruit extract is not mixed with the gelling agent base.

3.3pH

According to Tranggono and Latifah (2007) a good preparation of peel off gel masks has a pH that matches

the physiological pH of the skin which is 4.5 to 6.5, if it has a pH smaller than 4.5 it can irritate the skin while if the pH is greater from 6.5 can cause scaly skin [8]. The research data above shows that all formulations of peel off gel masks are produced according to the physiological pH of the skin, which is still in the pH range of 4.5 to 6.5. So that the three formulas are said to have met the criteria that correspond to the physiological pH of the human skin.

3.4 Viscosity

Viscosity test is carried out to determine the thickness of a preparation. Viscosity in the gel is influenced by an increase in humectant concentration and gelling agent [15]. According to Chandira et al (2010) the viscosity of the gel peel off mask preparation should be in the range of 7,100 - 83,144 cps. In this study the resulting viscosity of the dosage ranged from 2,979,966 cps -6,567,918 cps. None of the formulas that meet the viscosity of the peel off gel mask.

3.5 Flow properties

The resulting flow properties are pseudoplastic flow and dilatan. Flow properties in pharmaceutical preparations that are non-Newtonian in nature are plastic, pseudoplastic, or dilatan [7]. Flow flow shows an increase in inhibitory power to flow with increasing rate of shear. Pseudoplastic flow when this flow curve passes point (0,0), in contrast to plastic flow so that the pseudoplastic flow does not have the yield value. The pseudoplastic substance decreases with increasing rate of shear.

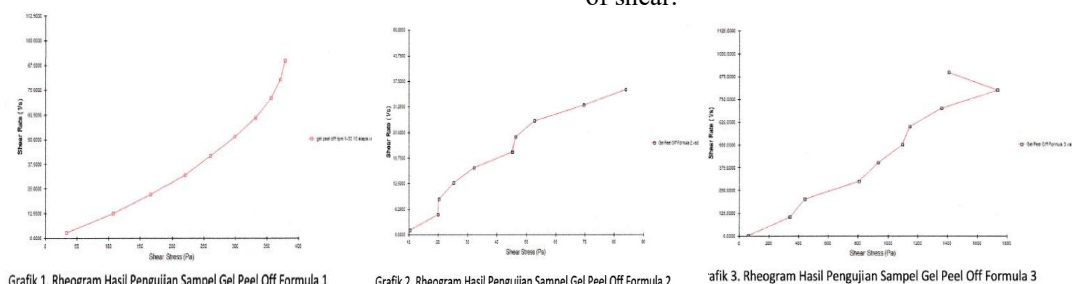


Fig 1. Flow properties of sample

3.6 Spread Power

Spread power testing aims to see the ability to spread the gel over the surface of the skin during use [13]. Spread in the dosage form is inversely proportional to its viscosity. The lower the viscosity, the higher the

dispersal power [2]. The desired range of dispersive power for topical preparations aimed at facial skin is 5-7 cm [15]. The dispersibility that meets the requirements is the Formula with a PVA base of 6.39 cm.

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3.7 Drying Time Test

Testing when drying the peel off gel mask preparation aims to find out how long the gel preparation will dry on the surface of the skin and form a layer of film. When dry the preparation of peel off gel mask is good between 15-30 minutes [12]. This time is the ideal time to apply masks in general. All formulas meet the time parameter to dry.

3.8 Irritation test

The irritation test was carried out on 10 female panelists aged 20-30 years. The attachment of the test material is carried out on the arm closed (patch test). Test results from the formula based on carbomer 940 and PVA + PVP showed that each panelist experienced irritation in the form of redness on the skin after the use of the preparation. Irritations that occur are reversible because redness disappears within minutes. While the PVA-based formula none of the panelists who experienced irritation can be said that formula 2 is safer to use.

3.9 Stability test for cycling test method

Stable preparations are preparations that are within acceptable limits during storage and use, where the properties and characteristics are the same as those they had when they were made. Viewed from an organoleptic perspective, the results of the stability test of the cycling test method did not show the difference between before and after the test. The pH value after the stability test has increased and some have decreased. Changes in the pH value are affected by the media which is decomposed by the temperature at which storage produces acids or bases, this is due also to the formula not being added to the buffer or buffer. But changes in pH that occur still meet the physiological pH range of the skin. The effect of the scatter test obtained a decrease in spreading power in each preparation after a stability test, but formula 1 still can be qualified before or after storage that is 5-7 cm. The decrease in spreading power is due to the influence of the physical properties of the gelling agent.

3.10 Activity Test of Antioxidant Mask of Peel Off Gel

The value of antioxidant activity using DPPH method is stated with IC₅₀. IC₅₀ is large, the antioxidant activity is classified as weak, whereas if the IC₅₀ value is small of the antioxidant activity is strong. Testing of

antioxidant activity is carried out in the formula with PVA which has the best physical properties and is safe for use. The IC₅₀ value in the formula with PVA base is 386.23 ppm while the pedada ethanol extract has an IC₅₀ value of 88.25 ppm. The level of antioxidant strength from the preparation of peel off gel mask and ethanol extract of pedada fruit is weak antioxidant (IC₅₀ > 150ppm). While the IC₅₀ value of vitamin C as a comparison is 7.248 ppm which means that the antioxidant strength is very strong. The antioxidant activity of the peel off gel mask is lower than of vitamin C. This is because the preparation of peel off gel mask of ethanol extract of pedada fruit does not have a pure active compound and still has all the components of the chemical compound both primary metabolites or secondary metabolites.

Conclusion

The peel off gel mask with PVA has best physical properties, more stable during storage and safe for use and non irritating compared to Carbomer 940 and PVA + PVP base which after use can be irritation to skin. The an antioxidant activity test and the gel peel off mask with PVA have IC₅₀ 386.23 and antioxidant potential activity has weak because is not pure compounds.

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