Formulation and Evaluation Antioxidant Activity of Peel-off Masks from White Pomegranate Extract with Polyvinyl Alcohol and Polyethylene Glycol 1500

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Abstract:

Pomegranate is tropical fruit contains antioxidant compounds such as ellagitannin, punicalagin, and tocopherol. Punicalagin has antioxidant EC₅₀ 54.2 ppm. This study aims to formulate and evaluate peel -off mask containing pomegranate extract. Dosage formulation of peel off mask made in various concentrations of white pomegranate peel extract that F1(1%), F2 (2%) and F3 (3%) with PVA and PEG 1500 as based. Further preparation peel off mask evaluated physical and chemical characteristics and stability during storage. The Result showed that the F3 showed the highest antioxidant activity than the other formulas. The increased concentration of white pomegranate (*Punica garanatum* L.) extract had an effect on antioxidant activity of white pomegranate peel extract peel-off gel masks.

1 INTRODUCTION

Indonesia is a tropical climate with abundant sunlight. Exposure to UV from the sun continuously will cause premature aging (photoaging). Premature aging can be characterized by the appearance of wrinkles, altered pigmentation and loss of skin tone (Fisher *et al.*, 1997). Another factor for premature aging is genetic diseases. in child can have aging feathers. The three known premature aging syndromes on human being are Hutchinson-Gilford Progeria Syndrome (HGPS), Werner syndrome (WS), and Cockayne syndrome (CS) (Wang-michelitsch and Michelitsch, 2007).

Precautions for premature aging can be done by using antioxidant compounds. Antioxidants have the ability to bind to free radicals caused by oxidative stress, and may have significance in the prevention and / or treatment of various skin diseases, and slow down the process of skin aging (Dasgupta and Klein, 2014; Sadowska-Bartosz and Bartosz, 2014; Stojiljković, Pavlović and Arsić, 2014). Recently, natural antioxidants have been chosen by consumers and have become popular for treatment and food applications compared to synthetic antioxidant compounds (BHT and BHA). Plants that grow a lot in the tropics and contain natural antioxidants are

Punica granatum. Pomegranate is consumed fresh and in processed form. Commercial pomegranate juice has the highest antioxidant activities compared to other fruit juices, red wine, and green tea and currently is a high value product in the agricultural market (Prakash and Prakash, 2011; Wang, 2011).

Pomegranate consist of flavonoid, ellagitannin, punicalagin, ellagic acid, vitamin and mineral. Punical agin and Ellagitannin are compounds that play a major role in providing antioxidant effects (Viladomiu et al., 2013; Rahmani, Alsahli and Almatroodi, 2017). In this study selected gel form peel-off mask. Peel-off gel mask is a type of mask that will dry and form an occlusive film that can be peeled off after use (Ningsih, Firmansyah and Fitri, 2016). Peel-off gel mask can increase skin moisture and increase the effect of the main compound (active compound) on the epithelial part due to the occlusivity of the polymer coating (Shai, Maibach and Baran, 2009). This study uses PVA as a film former because it can produce a gel that dries quickly and forms a film layer that is transparent, strong, plastic and adheres well to the skin and PEG 1500 as a plasticize which will maintain the film's shape so that when the mask is peeled off the film remains good and not torn. Based on the description above, the research will be made a peel-off gel mask extract of white pomegranate skin extract with a concentration of 1%, 2% and 3% on the basis of PVA and PEG 1500 which have antioxidant activity.

2 METHODS

An experimental study was selected to comparing the effect various concentration of white pomegranate extract (*Punica granatum*) of peel-off gel mask on the basis of Polyvinyl Alcohol (PVA) and Polyethylene Glycol.

Preparation of the white pomegranate extract

Dry powder of white pomegranate was extracted with ethanol 70 %. The sample was macerated for 72 hours and then filtered. The filtrate obtained was concentrated using a rotary evaporator to obtain brown extract of white pomegranate.

Screening phytochemistry

The extracts were screened for the phytochemicals such as flavonoid, terpenoid, alkaloid, anthraquinone, tannin and saponin. Phytochemical screening were performed using standard procedures(Trease and Evans, 1989; Sofora, 1993).

Antioxidant activity of white pomegranate extract

Stock solution of DPPH were prepared in methanol and methanol buffered with acetic acid buffer (0.1 M, pH 5.5), respectively. Buffered methanol was prepared by mixing 40 mL of 0.1 M acetate buffer (pH 5.5) with 60 mL methanol. Ascorbic acid is used as control positive (2, 4, 6, 8, 10 ppm). Control

positive was prepared by mixing 200 ppm of DPPH and methanol. The Solvents and other chemicals wrapped in aluminum foil and kept at 30°C for 30 min in dark. All measurement were done at 517 nm using Spectrophotometry. The data are mean \pm SD. The percentage of decolourisation was plotted against the concentration of the sample, and the IC50 values were determined. The DPPH absorbance decreases with an increase in DPPH radical scavenging activity. Results were expressed as IC50 concentration where 50% inhibition of the DPPH radical is obtained. This activity is given as the percent of DPPH radical scavenged, which is calculated with the equation:

DPPH radical scavenging activity (%) =
$$\left[\frac{\text{(Abs control - Abs sample)}}{\text{Abs control}} \times 100\right]$$
 (1)

Absorabance of control is the absorbance of DPPH radical + methanol and Abs sample is absorbance DPPH radical + sample.

The formulation of peel-off gel mask

To develop a suitable peel-off gel mask, the formulations were developed within determination range with different concentrationpercentage of extract. According to the formula given in the table 1, 3 different formulations were fabricated and then evaluated characteristic and stability in order to select the best formulation. For preparation of peeloff gel mask formulation, the concentrated extract of white pomegranate were used. Initially stoke 80% PVA was prepared. A certain amount of PVA powder was weighted and added to the water on the magnetic stirrer at 80°C for 15 minute and then placed in freezer for 24 hours.

Table	1. Form	ila of ne	el-off gel	mask from	white Po	megranate extract.
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In andiant	Formula (%)			
Ingredient	FB	FI	FII	FIII
Extract of White Pomegranate	-	1	2	3
Polyvinyl Alcohol	15	15	15	15
Polyethylene Glycol (PEG) 1500	2,5	2,5	2,5	2,5
Vitamin C	0,01	0,01	0,01	0,01
Propylene Glycol	10	10	10	10
Methyl Paraben	0,18	0,18	0,18	0,18
Propyl Paraben	0,02	0,02	0,02	0,02
Aquadest ad	100	100	100	100
White pomegranate extract	-	1	2	3

Evaluation parameters

Organoleptic: The consistency and the colour was checked visually. The odour was evaluated manually by smelling the product.

Homogeneity: This parameter was checked visually.

pH: 1% solution was prepared and checked for the pH using pH meter.

Observation of physical stability: at room and cold temperatures by observing organoleptic during storage

Spread ability: A small amount of the mask was applied on paper charts coated transparent glass given the particular load (1g) and then left to stand for 60 seconds. Then calculate the area given by the dosage is calculated.

Peeling time: A small amount of the mask was applied on the skin, left for few minutes and the time was noted until it formed a mask.

Antioxidant activity: This parameter was checked with calculate percent inhibition (DPPH radical scavenging activity (%).

Antioxidant test for gel peel-off mask mask using DPPH method

DPPH solution was made with a concentration of 200 ppm and 40 ppm. Then the preparation of the test solution was made with a concentration of 10,000 ppm and filtered, then the filtrate obtained was pipette as much as 1.0 ml mixed with a DPPH solution of 200 ppm and stirred with Methanol p.a ad 10.0 ml. Then absorbance was measured with a UV-Vis spectrophotometer at a maximum wave-length of 517 nm.

3 RESULTS AND DISCUSION

In this study the sample used was pomegranate skin extract, which has a high antioxidant effect. Sample extraction has been carried out by maceration method using 70% ethanol solvent. Ethanol is a semi polar compound that can attract both polar and nonpolar compounds. So that the compounds obtained are more and have the potential to be able to have antioxidant activity.

Screening phytochemistry

Phytochemical screening tests can be seen in Table 2. The results obtained indicate that pomegranate skin extracts found several secondary metabolites including terpenoids, flavonoids, sterols and steroids, saponins, anthraquinones and tannins.

Table 2: Phytochemical screening of Pomegranate extract.

No	Metabolite seconder	Result
110		resur
1	Terpenoid	+
2	Alkaloid	-
3	Flavonoid	+
4	Sterol dan Steroid	+
5	Saponin	+
6	Antrakinon	+
7	Fenol & tanin	+

Antioxidant activity determination of white pomegranate extract

Antioxidant test, the reaction between DPPH and white pomegranate rind extract that occurs because DPPH can react with compounds that can donate hydrogen atoms, one of which is the antioxidant contained in white pomegranate rind extract. The reaction mechanism that occurs is the antioxidant extract of a white pomegranate rind reducing DPPH compounds resulting in a reduction in the intensity of the color of the DPPH solution which was originally purple to fade (yellow). Color fading will cause a decrease in the absorbance value of spectrophotometer (Prakash, 2001) as in the antioxidant test that has been done it is known that the higher the concentration of the extract where the more antioxidants are added the more yellow the color of the solution and the absorbance decreases.

Measurements of antioxidant activity were conducted at wavelength 517 nm. Based on testing the antioxidant activity IC_{50} values obtained to red rice bran extract is 18.11 ppm and percent inhibition is 95.61 %, at a concentration of 80 ppm. Based on the classification carried out by Blois (Blois, 1958), if the IC_{50} value <50 μg / ml is categorized as a strong antioxidant, 101-150 μg / ml is a medium of antioxidant, while a weak antioxidant with $IC_{50}\!\!>\!150$ μg / ml. in this study pomegranate peel extract and positive control (vitamin C) included in the category of strong antioxidants.

Table 3: Pomegranate extract antioxidant test results.

Commlo	Sample conc.	Absorbance		Percent	IC ₅₀
Sample	(ppm)	Sample	DPPH	Inhibition (%)	$(\mu g/ml)$
	5	0.647		36.94	
White	10	0.319		68.91	
Pomegranate	20	0.069		93.27	18.11
Extract	40	0.069		93.27	
	80	0.045	1.026	95.61	
	2	0.747	1.026	27.19	
	4	0.471		54.09	
Vitamin C	6	0.184		82.07	3.67
	8	0.069		93.27	
	10	0.054		94.74	

NI.	D (Observation				
No	Parameters		F1	F2	F3	
		Consistency: semisolid	Consistency: semisolid	Consistency: semisolid	Consistency: semisolid	
1	Organoleptic	Colour: white Odor: specific	Colour: brownish yellow	Colour: brownish yellow	Colour: light brown	
			Odor: specific	Odor: specific	Odor: specific	
2	Homogeneity	Homogeneity	Homogeneity	Homogeneity	Homogeneity	
3	pН	6.46	5.38	5.03	4.83	
4	Physical stability (freeze thaw)	Stable	Stable	Stable	Stable	
5	Spread ability at load 1 g (cm/g)	10.43	9.73	9.47	9.05	
6	Peeling time	28 minute, 30 second	27 minute, 17 second	26 minute, 40 second	26 minute, 20 second	
		0,51 %	51.69 %	80.72 %	87.74 %	
7	Percent inhibition	(1st week)	(1st week)	(1st week)	(1st week)	
		2.05 %	40.61%	76.23 %	85.14 %	
		(4th week)	(4th week)	(4th week)	(4th week)	

Table 4: The result of characteristic evaluation of peel-off gel mask.

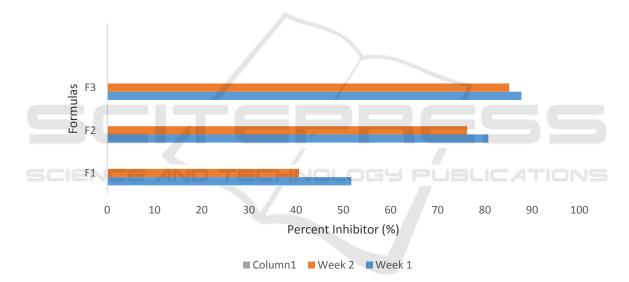


Figure 1: Antioxidant activity at 1st week and 4th week of peel-off gel mask of white Pomegranate extract.

Evaluation Parameters

Based on the evaluation of peel-off gel mask of organoleptic, homogeneity can be seen in the table 4. The result showed that all formulas is stable and homogeneity during 4 weeks of storage. The evaluation results obtained pH range from 4.83 to 5.38, where the normal skin pH range is 4.5 to 7. It can be that the pH of the formulas excluded in the pH range of normal skin (Table 4).

The observation of the physical stability by using freeze thaw during 8 week of storage indicates that all of formulas is not separation and preparation is stable. Spread ability is done by using weights, the goal is to see how much power and the ability of spreading on the surface of the skin. Based on observations obtained F1 9.73 cm/g, F2 9.47 cm/g dan F3 9.05 cm/g. Based on result F3 have spread power more undilute than F1 and F3.

Based on determination of antioxidant activity that percent inhibition of control formula showed no antioxidant activity while F1 showed their antioxidant activity 51.69 % (the first week) and 40.61% of fourth week, F2 in the first week 80.72% and 76.23% of fourth week and F3 on the first week amounted to 87.74% and the fourth week amounted to 85.14%. The result showed that F3 is the higher antioxidant activity more than F1 and F2 (Fig.1). This

because in F3 containing the highest concentration of white pomegranate extract.

4 CONCLUSIONS

Various levels of white pomegranate extract in peel-off gel mask ($Punica\ granatum\ L$.) has an influence on antioxidant activity that have been made both before and after the stability test. Increasing concentration of fruit peel extract will significantly increase the antioxidant activity with a value (p = 0.00 < (0.05). After the T test on antioxidant activity, there was a significant difference between before and after the stability test in the form of a decrease in antioxidant activity.

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