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Research Article

Optimization of Polyvinyl Alcohol (PVA) and Glycerin in Kolang Kaling Peel-Off Gel Mask Formula (*Arenga pinnata*.)

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Abstract: Aging is caused by free radicals, one of which is ultraviolet light which can form ROS. Galactomannan compounds have the potential as antioxidants that can counteract free radicals. Kolang kaling contains many galactomannan compounds. Kolang kaling is processed into a peel-off gel mask, where PVA and glycerin are essential ingredients. PVA can form a polymer film layer, but if the layer is not balanced, glycerin is used as a humectant. The results of the FTIR analysis showed that the functional groups were D-galactopyranose and D-mannopyranose, which were characteristic of the functional groups of galactomannan. The IC50 of kolang kaling powder is 23.8720 ppm, a powerful antioxidant. The interaction of PVA and glycerin increased drying time, viscosity, antioxidant activity, and decreased dispersion, adhesion, and pH. The optimal formula obtained was 14.47% PVA and 15.73% glycerin. The results of the One Sample T-Test validity test found that the results of the equations of each test were valid.

Keywords: glycerin; optimization; peel-off gel mask; PVA; sugar palm fruit

1. INTRODUCTION

Aging is a process experienced by the human body where body parts are decreasing, for example skin thins and wrinkles appear on the face [1]. Ultraviolet light is a potential trigger in forming free radicals Reactive Oxygen Species (ROS) on the skin. Compounds that can capture free radicals are antioxidants [2].

Polysaccharides are one of the most widely studied antioxidants today. Kolang kaling contains many galactomannan polysaccharide compounds, which are natural hydrocolloids. The galactomannan fraction in kolang kaling, as much as 10 g/mL, can show higher antioxidant activity (>50%) compared to ascorbic acid [3].

Using kolang kaling as an antioxidant will be more effective and acceptable if formulated in a topical preparation. Antioxidants in topical preparations will allow antioxidants to interact longer with the skin [4]. Peel-off gel masks are an option for various topical preparations that can improve skin quality. Peel-off gel masks have the benefit of repairing dan treating facial skin from problems with wrinkles, aging, acne, and can also shrink pores [5].

Peel-off gel masks have the principle of being able to form a polymer plastic film that is easy to peel off [6]. An important factor, namely PVA influences the formation of the film layer.

PVA plays a role in forming a film that is easy to peel off, so it can provide a peel-off effect after drying [7]. The film layer that has been formed to maintain the stability of the gel mask preparation the used glycerine which functions as a humectant. Glycerine helps maintain the stability of the gel mask dosage form by reducing the evaporation of water from the mask preparation [5]. Based on this background, it is necessary to research to optimize PVA and glycerine in the kolang kaling peel-off gel mask formula.

2. MATERIALS AND METHODS

Materials

The materials needed are kolang kaling from Gunung Pati, PVA, glycerin, HPMC, methylparaben, oleum rosae, aqua distillate, concentrated H_2SO_4, α-naphthol, NaOH, $CuSO_4$, ninhydrin, ammonium oxalate, ethanol, methanol, and DPPH

2.1. Making Kolang Kaling Powder

The kolang kaling was washed using running water and sliced thinly, and then the kolang kaling was dried using a microwave at a temperature of 40°C for ± 22 hours. The dried kolang aling was blended and sieved with a mesh sieve number 44.

2.2. Phytochemical Screening

2.2.1. Carbohydrates

The sample was given 5 drops of polish reagent and shaken carefully. Add 5 drops of concentrated H_2SO_4 slowly through the walls of the test tube. Positive results are evidenced by forming a purple ring [8].

2.2.2. Galactose and Mannose

The sample was added to Luff Schroll's solution, and a red brick precipitate formed, indicating that the sample contained galactose and mannose compounds [9].

2.2.3. Protein

In the biuret test, the sample was added with dilute NaOH and diluted $CuSO_4$. Positive results are indicated by the formation of purple or blue-violet spots or solutions [10]. The ninhydrin test was carried out by adding the sample with ninhydrin powder. Positive results are indicated by a blue solution [11].

2.2.4. Calcium

The sample is added with ammonium oxalate, and the formation of a brownish-white precipitate means that the sample is positive for calcium[12].

2.3. Preparation of Peel-Off Gel Mask Preparations

The peel-off gel mask was prepared by expanding PVA over a water bath using an aquadest in a porcelain cup. Kolang kaling and HPMC were developed with aquadest. The PVA that had been developed was mixed with the expanded HPMC and the added kolang kaling powder. Methylparaben, glycerine, and perfume are poured, mixed with distilled water up to 40 grams, stirred until homogeneous, and placed in a container. All formula peel-off gel mask preparation was made based on Run Design Expert 10.0.1 using Simplex Lattice Design. The peel-off gel mask formula for kolang kaling can be seen in Table 1.

Table 1. Kolang Kaling Peel-Off Gel Mask Formula

Formula	Run							
	I	**II**	**III**	**IV**	**V**	**VI**	**VII**	**VIII**
Kolang Kaling Powder (%)	2	2	2	2	2	2	2	2
PVA (%)	11	11	12	13	13	14	15	15
Glycerin (%)	19	19	18	17	17	16	15	15
HPMC (%)	4	4	4	4	4	4	4	4
Methylparaben (%)	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
Perfume	5	5	5	5	5	5	5	5
Aquadest ad (g)	40	40	40	40	40	40	40	40

2.4. Physical Characteristics Test

2.4.1. Organoleptic Test

The organoleptic test was carried out by paying attention to changes in colour, aroma, shape, and texture of the peel-off gel mask preparation [13].

2.4.2. Homogeneity Test

The preparation of 0.1 grams was placed on an object of glass and covered with another glass object. The object glass is pressed and observed under light [14]. The preparation is said to be homogeneous if there are no coarse grains in the peel-off gel mask preparation [15].

2.4.3. pH test

The pH test was carried out by dipping the pH meter into a peel-off gel mask, and the pH meter was allowed to stand until the screen showed a constant number [16].

2.4.4. Dry Time Test

The peel-off gel mask is applied as much as 0.1 grams with a smearing area of 2.5X2.5 cm evenly on the arms. Observe the time required for the preparation to dry [17].

2.4.5. Spreadability Test

The dosage of 1 gram is placed on glass and covered with another glass of the same size. Put a weight on it, as much as 50 grams, and let it stand for 1 minute, then measure the diameter of the preparation. The ballast is added periodically, as much as 50 grams, until the diameter of the preparation is constant [18].

2.4.6. Adhesion Test

250 grams of the preparation is placed on a slide, and another slide is placed on top. 1 kg of weight is placed on the slide for 5 minutes. The slide is mounted on the test instrument, and 150 grams of the load is hung on the left side, then the load is released, and the time it takes for the two slides to be released is recorded.

2.4.7. Viscosity Test

A sample of 15 grams of peel-off gel mask was put into a container, then spindled 64 until wholly submerged. The speed is set using 1.0 rpm, press enters, and observe the numbers that appear on the Brookfield viscometer [18].

2.5. Antioxidant Test by DPPH Method (2.2-diphenyl-1-picrylhydrazyl)

2.5.1. Preparation of 30 ppm DPPH Solution

1.5 mg of DPPH was weighed and dissolved with methanol p.a to the limit mark in a 50 mL volumetric flask.

2.5.2. Determination of Wavelength

The 30 ppm DPPH solution was poured into a cuvette, and then the absorbance was measured at a wavelength of 400-800 nm using a UV-Vis spectrophotometer.

2.5.3. Determination of Operating Time

A total of 2.0 mL of the middle standard sample solution was added to 2.0 mL of 30 ppm DPPH solution. The absorption was measured using a UV-Vis spectrophotometer at a wavelength obtained for 60 minutes.

2.5.4. Preparation of Peel-Off Gel Mask Main Liquor

A total of 0.05 grams of the sample was dissolved in methanol p.a, then put into a 50 mL volumetric flask and diluted to the limit mark.

2.5.5. Antioxidant Activity Test

Vitamin C was made in series with concentrations of 2, 4, 6, 8, and 10 ppm. Kolang kaling powder was made in series with concentrations of 10, 20, 30, 40, and 50 ppm. Gel mask samples were made in concentrations in 20, 40, 60, 70, and 80 ppm. The 2.0 mL sample was added with 2.0 mL of DPPH solution and placed in a test tube coated with aluminium foil. Samples were incubated at room temperature according to the operating time obtained for each sample. Absorption test using a UV-Vis spectrophotometer using the maximum wavelength obtained.

2.5.6. IC50 Determination

Determination of IC50 is carried out from the results of absorbance measurements from 5 concentration series to produce % attenuation.

3. RESULTS AND DISCUSSION

3.1 Kolang Kaling Powder

Kolang kaling from Gunung Pati was washed using running water and sliced thinly, and then the kolang kaling was dried using a microwave at a temperature of 40°C for ± 22 hours. The dried kolang kaling was blended and sieved with a mesh sieve number 44 until a fine powder forms.

3.2. Phytochemical Screening

The results of the phytochemical screening test showed that the kolang kaling powder contained carbohydrates, galactose, mannose, and calcium compounds.

3.3 Peel-Off Gel Mask Preparations

The peel-off gel mask was prepared by expanding PVA in a porcelain cup. Kolang kaling and HPMC were developed with aquadest. The PVA that had been developed was mixed with the expanded HPMC and add kolang kaling powder. Methylparaben, glycerine, and perfume are poured, mixed with distilled water up to 40 grams, stirred until homogeneous, and placed in a container. All formula peel-off gel mask preparation was made based on Run Design Expert 10.0.1 using Simplex Lattice Design.

3.4 Physical Characteristics Test

3.4.1. Organoleptic test

The organoleptic test showed that all the runs had a gel form, soft texture, cream colour, and rose scent. Adding kolang kaling powder makes the peel-off gel mask have a cream colour, while the aroma of roses is due to the formula containing the ingredients oleum rosae.

3.4.2 Homogenity Test

In the homogeneity test, it was found that all runs were homogeneous, as indicated by the distribution of the preparation evenly on the object glass, and there were no coarse particles.

3.4.3 pH Test

Higher concentrations of PVA were able to lower the pH value, while glycerine increased the pH of the mask preparation. The coefficient value of the PVA component (+0.51504), which is greater than the glycerin component (+0.39948), indicates that the PVA component effect on lowering pH than glycerin. The interaction between PVA and glycerine can reduce the pH of the peel-off gel mask preparations with a coefficient value of (-0.038333). PVA has a pH value between 5-8 and glycerin with a pH of 7, so variations in the concentration of PVA and glycerin can affect the pH of the peel-off gel mask preparation.

3.4.4 Dry Time Test

The results of the drying time test showed that the higher the concentration of PVA, the faster the drying time, while the higher the glycerin, the slower the drying time. The value of the PVA component (-4.25752) is greater than the value of glycerin (-0.31987), indicating that the PVA component has a more significant effect on accelerating the drying time of the kolang kaling peel-off gel mask preparation. The interaction of PVA and glycerin components can increase the drying time of the preparation with a coefficient value (+0.36275). Glycerin is hygroscopic and can bind water in the surrounding environment so that it slows down the drying time. PVA acts as a gelling agent, where PVA has adhesive properties or can form a film that is easily peeled off after drying [13]. The greater the concentration of PVA can cause the formation of the gel mask film layer to be faster, accelerating the drying time of the preparation.

3.4.5 Spreadability Test

PVA with a greater concentration can reduce spreadability, while a greater concentration of glycerin can increase spreadability. The coefficient value of the PVA component (+0.16287) is smaller than the coefficient of glycerin (+0.32398) which indicates that glycerin is more influential than PVA in increasing the spreadability of peel-off gel masks. The interaction of PVA and glycerin components can reduce the spreadability of the preparation with a coefficient value (-0.012500). PVA has adhesive properties that can inhibit resistance to flow and spread.

3.4.6 Adhesion Test

On adhesion, the greater the concentration of PVA can increase the adhesion, while the more significant the concentration of glycerin can decrease the adhesion of the peel-off gel mask. The coefficient value of the PVA component (+386.60959) is greater than the coefficient value of the glycerin component (143.53442), which indicates that PVA has a more significant influence on increasing the adhesion of gel mask preparations. The interaction between the two components can reduce the adhesiveness of the mask with a coefficient value (of-32.75490). PVA has adhesive properties (adhesive), increasing the adhesion of peel-off gel mask preparations.

3.4.7 Viscosity test

In viscosity testing, it was found that the greater the concentration of PVA can increase the viscosity, the more significant the concentration of glycerin can decrease the viscosity. The interaction between PVA and glycerin can increase the viscosity, as indicated by the coefficient value (+4803.92517). The increased viscosity can be influenced by the concentration of PVA, because it increases the number of polymer fibers so that more liquid is retained and bound by the gelling agent, which causes the viscosity of the preparation [7].

The optimal formula obtained was a formula with a ratio of 14.470% PVA and 15.530% glycerin. The optimal formula was made 5 times of replication and then tested for physical characteristics and antioxidant activity. The results obtained are the optimal formula for the peel-off gel mask has a cream color, rose smell, soft texture, and gel form, homogeneous, drying time 16.8 minutes, spreadability 4.76 cm, adhesion 447.6 seconds, pH 5,1, viscosity 558800 cps, and IC50 34,8327 ppm.

The parameter results were validated using the One Sample T-Test technique. The significance value obtained is a significance value of p> 0.005, which indicates that the results obtained between the theoretical and experimental results are not significantly different, which means that the equations of each optimization parameter are valid.

3.5. The Results of Antioxidant

Testing the antioxidant activity of kolang kaling powder with a maximum wavelength of 516.60 nm. The operating time (OT) of vitamin C was 16 minutes, OT for powder kolang kaling was 23 minutes, and OT for gel mask preparation was 11 minutes. Vitamin C, a comparison compound, has an IC50 value of 4.8187 ppm, including intense antioxidant activity. The IC50 of kolang kaling powder is 23.8720 ppm, which means that kolang kaling powder has powerful antioxidant activity but is not more potent than vitamin C.

PVA, which is used as a wetting agent in galactomannan, can cause an increase in the molecular dispersion of galactomannan, affecting the viscosity level. Galactomannan dispersion can bind to free radicals DPPH, which causes an increase in antioxidant activity if the molecular dispersion of galactomannan increases. Based on the above, it can be concluded that the higher the PVA, the more galactomannan dispersion, so the viscosity of the preparation increases, and the antioxidant activity is more potent.

In the antioxidant activity test, the greater the concentration of PVA can reduce the IC50, while the more significant the concentration of glycerin can increase the IC50. The interaction of PVA and glycerin can increase IC50 with a coefficient value of (+0.35294). The correlation value between viscosity and IC50 was calculated from the obtained r value. The value of r obtained is -0.87612, indicating that viscosity and IC50 have a powerful correlation. The higher the viscosity, the lower the IC50 so that the antioxidant activity of the kolang kaling peel-off gel mask becomes stronger.

4 CONCLUSION

Based on the research that has been done, the differences in the concentration of PVA and glycerin in the preparation significantly affect the physical characteristics and antioxidant activity of the kolang kaling peel-off gel mask preparation. The optimal formula for kolang kaling peel-off gel mask was obtained at a concentration ratio of 14.47% PVA and 15.5% glycerin.

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