Indonesian Journal of Pharmacy

VOL 32 (2) 2022: 261–268 | RESEARCH ARTICLE

Formulation And Optimization Peel-Off Gel Mask with Polyvinyl Alcohol and Gelatin Based Using Factorial Design from Banana Peel Flour (*Musa paradisiaca L*) As Antioxidant

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Info Article

Submitted: 16-11-2021
Revised: 24-02-2022
Accepted: 19-04-2022

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ABSTRACT

Banana peel (*Musa paradisiaca L*) contains flavonoid compounds which act as an antioxidant. It has the potential to be developed into cosmetic preparations such as peel-off gel masks. This study aims to determine how variations in the concentration of banana peel flour, PVA, and gelatin affect the physical properties of peel-off gel masks and their antioxidant activity. This study used a factorial design of 2^3 in which the factors used are the concentration of banana peel flour, PVA, and gelatin with two levels. Based on the data analysis result, there was a significant effect of each factor and the interaction between them on the spreadability and drying time of the preparation ($p<0.05$). The concentration ratio of banana peel flour, PVA, and gelatin of 5:12:5 was chosen as the optimum formula and continued to antioxidant activity test compared to F4 which had a ratio of concentrations of banana peel flour, PVA, and gelatin of 10:14:3. The antioxidant activity produced by the optimum formula was better than F4 with IC_{50} values of 525.41 and 355.64 ppm, respectively. It can be concluded that improving the dosage formula will affect the active substance's activity, but it would be preferable if the banana peel was prepared as an extract.

Keywords: Banana peel flour, peel off mask, factorial design, antioxidants

INTRODUCTION

Banana (*Musa paradisiaca L.*) is a plant native to Indonesia. It consists of 2 parts: peel and pulp. Banana production in Indonesia exceeds 7.29 million tons per year, resulting in a large amount of banana peel waste. Since people do not consume it, the skin is often discarded, although the banana peel contains many secondary metabolites that are beneficial for health. Banana peel contains high protein, dietary fiber, unsaturated fatty acids, essential amino acids, and potassium (Emaga *et al.,* 2007). In addition, it also contains antioxidant compounds like carotenoids, catecholamines, and polyphenols (Someya *et al.,* 2002, Nguyen *et al.,* 2003, Bennet *et al.,* 2010; González-Montelongo *et al.,* 2010). The polyphenolic compound contained in the banana peel is gallocatechin (Saravanan & Aradhya, 2011). The gallocatechin content contained in the banana peel is higher than in the pulp, namely 158 mg/100 g DW and 29.6 mg/100 g DW (Someya *et al.,* 2002; Sulaiman *et al.,* 2011). Flavonoid compounds such as myricetin, quercetin, cyanidin and are also contained in banana peels (Kevers *et al.,* 2007). These compounds act as antioxidants because they have a hydroxyl group attached to the carbon of the aromatic ring so it can capture free radicals (Hamid *et al.,* 2010). This makes the banana peel can be used as a cosmetic active substance.

A peel-off gel mask is a topical cosmetic that dries to form an occlusive film layer that can be peeled off after application. Rahmawanty *et al.,* 2015). Peel-off gel masks can increase skin moisture and increase the effect of active substances on the epithelium due to the occlusivity of the polymer layer formed (Vieira *et al.,* 2009; Beringhs *et al.,* 2013). Peel-off gel masks have essential components: film-forming and gelling agents. This study used Poly Vinyl Alcohol (PVA) as a film-forming and gelatin as a gelling agent. After drying, PVA causes occlusion and tensor action, softening the skin (Vieira *et al.,* 2009).

Table I. Formulation of peel-off gel mask

Ingredients	Formula (%)							
	F1	F2	F3	F4	F5	F6	F7	F8
Banana peel flour	5	10	5	10	5	10	5	10
PVA	12	12	14	14	12	12	14	14
Gelatin	3	3	3	3	5	5	5	5
HPMC	1	1	1	1	1	1	1	1
Gliserin	5	5	5	5	5	5	5	5
Methylparaben	0.18	0.18	0.18	0.18	0.18	0.18	0.18	0.18
Aquadest ad	100	100	100	100	100	100	100	100

PVA in the range of 2.5% to 17.5% can produce a gel that dries quickly, forms a transparent, strong plastic film, and adheres well to the skin (Beringhs *et al.,* 2013). According to research by Beringhs *et al*. (2013), PVA at a concentration of 13% is the optimum concentration in peel-off gel masks. Gelatin also plays a role in the manufacture of peel-off gel masks. As a gelling agent, gelatin has non-toxic properties, is flexible, has the strength to form gels well, and has a high rate of water absorption (Rahmawanty *et al.,* 2015). The banana peel used in this study was in the form of flour. Banana peel flour has a decent amount of polyphenol content, which is 29.177 mg GAE/g DW (Babbar *et al.,* 2011).

Based on the description above, as the formulation and optimization of peel-off gel masks from banana peel flour, this study used a 2^3 factorial design which used three factors with two levels. The factors and levels used in the factorial design were as follows: Banana Peel Flour (5 and 10%), PVA (12 and 14%), and Gelatin (3 and 5%). To find the best peel-off gel mask formula, researchers analyzed data on its spreadability and drying time. The optimum formula will be tested for antioxidant activity using the diphenylpicrylhydrazyl (DPPH) method. This study is critical because the characteristics of the preparation can affect the release of the active substance, thereby affecting the antioxidant's effectiveness.

MATERIALS AND METHODS

The ingredients used in this study include banana peel flour from South Sumatra, Indonesia. Polyvinyl alcohol (PVA), Gelatin, Glycerin, methylparaben, 70% ethanol, magnesium powder, hydrochloric acid mayer reagent, bouchardat reagent, dragendorf reagent, Lieberman buchard reagent, n-hexane, iron (III) chloride, DPPH powder, and ascorbic acid ordered from Bratachem, Indonesia. Distilled water was from Merck, Indonesia.

Preparation of Banana Peel Flour

Banana peels were cleaned, steamed, and chopped. After that, they were dried for 24 hours at a temperature of 50^0C. Ground using a blender and sieve with a 60-mesh sieve. The flour was then kept at room temperature in a tightly sealed container.

Phytochemical Screening

The phytochemical screening covered the examination of flavonoids, alkaloids, steroids and triterpenoids, saponins, and tannins. The test was qualitatively using color reagents following the Ministry of Health of the Republic of Indonesia's regulation (Ministry of Health of the Republic of Indonesia, 2000).

Design Formula

The formula used was formula 2^3 factorial design, which consists of two levels and three factors. The factors and levels used in the factorial design were the concentrations of banana peel flour (5% and 10%), PVA (12% and 14%), and gelatin (3% and 5%). Based on the 2^3-factorial design, eight formulas were obtained for peel-off gel masks (Table I).

Preparation of Peel-Off Gel Mask

The peel-off gel mask was made following the concentration (Table I), PVA was dissolved at the temperature of 600C in water using a magnetic stirrer for 24 h (Mass I). Gelatin was dissolved in warm distilled water until it swelled completely, then was added HPMC gradually and then stirred until homogeneous (Mass II). Methylparaben was dissolved in glycerin and then added to Mass II, which was mixed until it was completely homogeneous. Then, mass I to mass II were mixed

until homogeneous (Mass III). Banana peel flour was added in mass III little by little and mixed until homogeneous. Finally, the remaining distilled water was gradually added until a gel mass was formed.

Evaluation of Peel-Off Gel Mask

The evaluation of the peel-off gel mask preparation included organoleptic, homogeneity, pH, spreadability, and drying time. Organoleptic test was directly observed the color, shape, and smell of the mask. The homogeneity test was observed visually. pH test measured with pH meter. Spreadability was determined by measuring the spreading diameter of 1 g of the sample between two horizontal glass plates (10 cm × 20 cm) after one minute (Apriani *et al*., 2018). The drying time was observed for 30 min until the drying process was completed.

Data Analysis of Peel-Off Gel Mask Evaluation

The spreadability and drying time data were analyzed according to the factorial design method using software *Design Expert 11*® to determine the influence of factors and factor interactions on the response.

Formula Optimization

Formula optimization was carried out using software *Design Expert 11*®. The formula was expected to provide maximum spreadability response and minimal drying time.

Antioxidant Activity Test

The testing procedure was carried out based on the DPPH method. A 6 ml test solution containing a peel-off gel mask with concentrations 50, 100, 150, and 200 µg/mL mixed with 3.8 ml of DPPH 1 mM. The mixture was incubated in a dark room for 35 minutes. After that, its absorbance was measured at a wavelength of 515 nm with a UV-Visible spectrophotometer (Indarti *et al.,* 2019). Measurements were made three times. The percent value of radical scavenging is calculated by following the formula 1:

$$\% \text{ Radical Scavenging} = \frac{A_{blank} - A_{test}}{A_{test}} x\ 100\% \quad ....1$$

Where: A Blank = Absorbance of DPPH Solution; A Test = Absorbance of Sample; IC_{50} values are calculated when the % radical scavenging value is 50%.

RESULT AND DISCUSSION

Preparation of Banana Peel Flour

The study used banana peel obtained from South Sumatra, Indonesia. The results obtained are blackish-brown flour with a distinctive odor.

Phytochemical Screening

Phytochemical screening of banana peel flour was to determine the compounds contained in the flour. The compounds identified were flavonoids, alkaloids, steroids/triterpenoids, saponins, and tannins. The results of phytochemical screening proved that banana peel flour contains flavonoids, alkaloids, steroids, saponins, and tannins.

The presence of flavonoids in banana peel flour is related to their antioxidant activity. It consists of flavonoid compounds such as myricetin, quercetin, and cyanidin (Kevers *et al.,* 2007). These compounds act as antioxidants because they have a hydroxyl group attached to the carbon of the aromatic ring so it can capture free radicals (Hamid *et al.,* 2010).

Evaluation of Peel-Off Gel Mask

In a peel-off gel mask preparation, a 2^3-factorial-design was used to create eight formulas from banana peel flour. Evaluation of the peel-off gel mask of banana peel flour has been carried out on eight formulas to determine the physical properties of the preparation. Organoleptic tests, homogeneity tests, pH tests, spreadability tests, and drying time tests were all used to evaluate the peel-off gel mask made from banana peel flour (Table II).

The peel-off gel mask produced has the characteristics as follows: yellowish-brown to brown color, homogeneous, pH around 5, spreadability in the range of 4.2 to 5.8 cm, and a drying time of 14 to 24 min. Peel-off gel masks have pH requirements ranging from 4.5 to 6.5, spreadability ranging from 5 to 7 cm, and a drying time of 15 to 30 min (Grace *et al.,* 2015; Rum *et al.,* 2021). F2, F4, F6, and F8 produced a spreadability of less than 5 cm. Thus, this formula did not meet the requirement. F6 also produces a drying time that is less than 15 min, which does not meet the requirements.

Data Analysis of Peel-Off Gel Mask Evaluation

The spreadability and drying time data were further analyzed with the software *Design Expert 11*® to see the impact of factors and interactions on the response and to find the best peel-off gel mask formula. Based on the results of the ANOVA

Table II. Evaluation of Peel-Off Gel Mask Result

Test	Result			
	F1	**F2**	**F3**	**F4**
Organoleptic	The yellowish-brown, semisolid, distinctive odor	Brown, semisolid, distinctive odor	Brown, semisolid, distinctive odor	Brown, semisolid, distinctive odor
Homogeneity	Homogeneous	Homogeneous	Homogeneous	Homogeneous
pH	5.42	5.30	5.36	5.42
Spreadability (cm)	5.78±0.55	4.88±0.39	5.15±0.30	4.22±0.08
Drying Time (min)	23.8	16.37	15.14	22.5
Test	**F5**	**F6**	**F7**	**F8**
Organoleptic	Brown, semisolid, distinctive odor	Brown, semisolid, distinctive odor	The yellowish-brown, semisolid, distinctive odor	Brown, semisolid, distinctive odor
Homogeneity	Homogeneous	Homogeneous	Homogeneous	Homogeneous
pH	5.38	5.40	5.62	5.54
Spreadability (cm)	5.63±0.38	4.33±0.21	5.35±0.57	4.43±0.50
Drying Time (min)	16.23	14.23	18.9	15.28

Table III. The Results of ANOVA Analysis

Source	p-value	
	Spreadability	**Drying Time**
Model	0.0009*	< 0.0001*
A-Banana Peel Flour	< 0.0001*	0.0010*
B-PVA	0.0477*	0.6717
A- Gelatin	0.6414	< 0.0001*
AB	0.5641	< 0.0001*
AC	0.5415	0.0002*
BC	0.1080	0.0002*
ABC	0.5195	< 0.0001*

*p value <0.05 indicates that the factor has a significant effect on the response

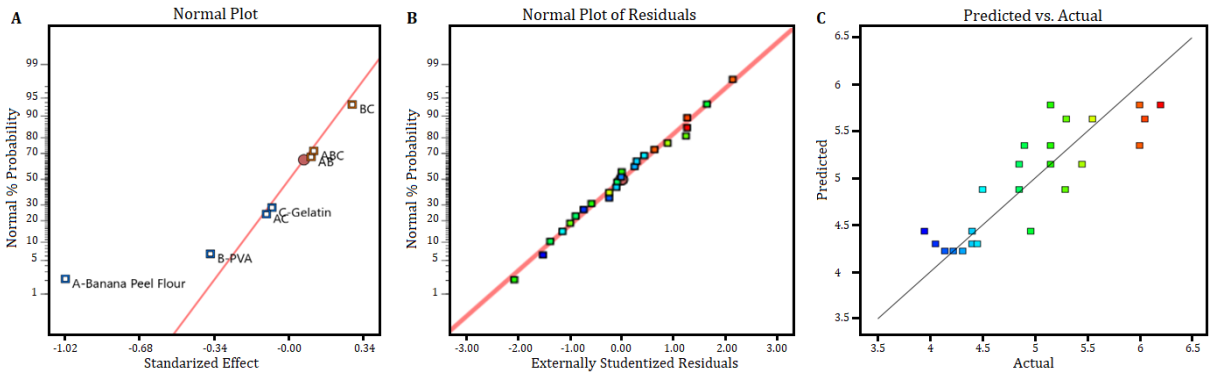

Figure 1. The Graph of Normal Plot (A), Normal Plot of Residual (B), and Predicted vs Actual (C) from Spreadability Data

analysis on the response of spreadability and drying-time (Table III) there is an influence of factors on the response. The banana peel flour (A) and PVA (B) influenced the spreadability response, whereas the banana peel flour (A), gelatin (C), the interaction of the banana peel flour and PVA (AB), the interaction of the banana peel flour and gelatin (AC), the interaction of PVA and gelatin (BC), and the interaction of the banana peel flour, PVA, and gelatin (ABC) influenced the drying time response.

The data generated on spreadability and drying time responses are normally distributed (Figure 1 and 2). Predicted and actual values can be seen in the Predicted VS Actual graphs (Figure 1 and 2). The spreadability had the Predicted R^2 of 0.4182, and is not as close to the actual R^2 of 0.6283 as one might normally expect; i.e. the difference is more than 0.2. It could be a sign of a large block effect or a problem with the model and/or data. But Adeq Precision for the spreadability test was 6.580, indicating an adequate signal. This model can be used to navigate the design space. The drying time response has Predicter R2 and Adjusted R2 values of 0.9322 and 0.9567, respectively, indicating that the resulting data is somewhat decent.

The influence of factors on the response can have positive and negative effects (Figures 1 and 2). Besides, it can also be seen from the response equations obtained based on data analysis. In the normal plot graph on the spreadability response, the banana peel flour (A) and PVA (B) harm the response with the equation y = 4.96938 – 0.50979 A – 0.17979 B. This negative effect illustrates that the greater the concentration of the banana peel flour (A) and PVA (B) used, the smaller the spreadability value produced. Banana peel flour contains starch with a fairly high concentration of 16 to 48.5% (Hernández-Carmona *et al.,* 2017). The presence of starch will affect the viscosity of the preparation (Hadisoewignyo *et al.,* 2017). Starch will be swelling when it contacts water, so the higher the concentration of banana peel flour used, the viscosity will increase. The viscosity of the preparation will impact the spreadability value. The higher the preparation viscosity value, the higher value of spreadability (Douguet *et al.,* 2017). PVA will also harm the spreadability response. The higher the concentration of PVA used, the more viscosity will increase because the hydrogen bonds that occur between the OH group in PVA and the amount of water used in the preparation will increase (Marin *et al.,* 2014).

The normal plot graph on the drying time response shows that the factors of banana peel flour (A), gelatin (B), the interaction of banana peel flour and gelatin (AC), and the interaction of the banana peel flour, PVA, and gelatin (ABC) had a negative effect. While the interaction of banana peel flour and PVA (AB) and the interaction of PVA with gelatin had a positive effect. This can also be seen from the resulting response equation, namely y = 1068.88 – 32.2917A – 94.9583C + 94.7917AB – 37.7917 AC + 39.2917 BC – 105.042 ABC. Banana peel flour harms the drying time response that the higher the concentration of banana peel flour used, the drying time value will decrease. It is because the concentration of water in the preparation will be smaller so that the drying time will be faster. Gelatin also gives the same effect. The higher the concentration of gelatin, the drying time will decrease. It supports the research of Rahmawanty *et al*. (2015) that increasing the concentration of gelatin causes the preparation to dry faster. Likewise, the results of the interaction between banana peel flour and gelatin and the interaction between banana peel flour, PVA, and gelatin will harm the drying time response. It means that when the concentration of these factors increases, the drying time of the preparation will decrease. However, when banana peel flour or gelatin interacted with PVA, there was a positive effect on the drying time response. It is because PVA acts as a plasticizer and film-forming. When PVA interacts with water, PVA will absorb the liquid and form a compact mass resulting in the liquid being retained and difficult to evaporate, causing the drying time of the preparation becomes longer or increase (Kathe & Kathpalia, 2017).

Formula Optimization

Formula optimization was also carried out using the software *Design Expert 11*® based on the results of the spreadability and drying time response. When the optimization was performed, the researchers chose the criteria for the optimum formula: wide spreadability and fast drying time. The program recommended an optimum formula with a desirability value of 0.970 and a concentration of banana peel flour of 5%, PVA of 12%, and gelatin of 5%. A desirability value close to 1 indicates that the formula is close to the criteria desired by the researcher (Kalariya *et al.,* 2017).

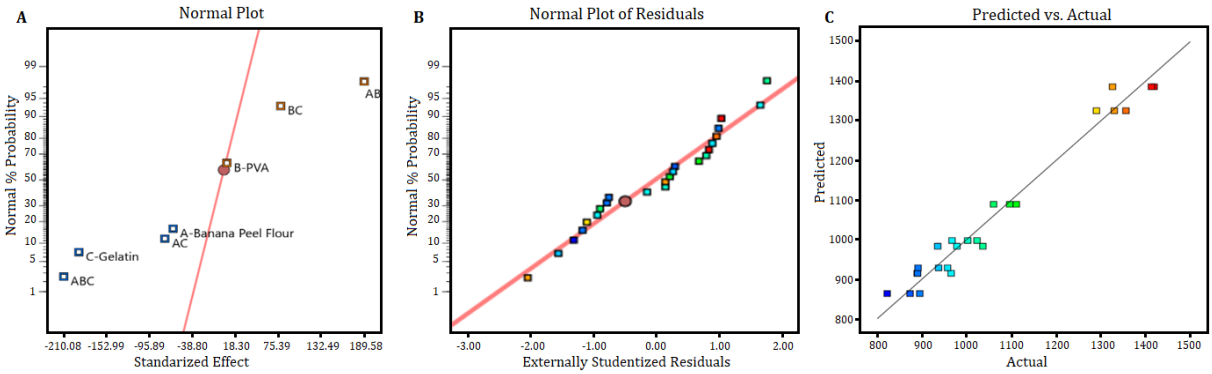

Figure 2. The Graph of Normal Plot (A), Normal Plot of Residual (B), and Predicted vs Actual (C) from Drying Time Data

Antioxidant Activity Test

In this test, the concentration of 5% banana peel flour, 12% PVA, and 5% gelatin was chosen as the optimum formula and F4 as a comparison which had the opposite concentration level with F5, namely 10%, 14%, and 3% respectively (Table IV).

Based on the result of antioxidant activity test (Table IV), both F5 and F4 showed a percent inhibition of DPPH. It proves that the peel-off gel mask can indeed act as an antioxidant. However, when viewed from the IC_{50} value, which is more than 200 ppm, it indicates that the antioxidant activity of the peel-off gel mask of banana peel flour is in the "very- weak" category (Molyneux, 2004). The banana peel flour was not extracted so the active substances were not fully extracted. However, this study aims to make cosmetic preparation so the result of the research is fairly acceptable. F5 gives a better activity than F4. It proves that an optimum base can help increase the antioxidant activity of banana peel flour. F5 contains 5% of banana peel flour while F4 contains 10% of banana peel flour concentration. Theoretically, the concentration of the active substance will be related to the expected activity. The higher the concentration of the active substance, the greater the activity of the active substance. However, the right concentration of the dosage base will also affect the release of the active substance. The release of the active substance will be effective when the concentration of the base used is optimum, increasing the active substance's activity. When comparing the drying times of F5 and F4, F5 has a faster drying time than F4, implying that the active substance's ability to penetrate the skin is faster and the effect is better.

CONCLUSION

The peel-off gel mask produced in this study has good physical properties, such as yellowish-brown to brown color, pH of around 5, homogeneity, and spreadability and drying time. Data analysis shows an effect of the use of the concentration of banana peel flour, PVA, and gelatin and the interaction between factors on the response of spreadability and drying time. The optimum formula produced based on the software *Design Expert 11*® is the concentration of banana peel flour was 5%, PVA was 12%, and gelatin was 5%; with a desirability value of 0.970. The results of the antioxidant activity test showed that there was an effect of using the optimum base for the release of the active substances. The IC_{50} of the optimum formula with 5% banana peel flhttps://doi.org/10.5897/AJPAC.9000020http://dx.doi.org/10.5530/pj.2019.11.122https://doi.org/10.1016/j.ajps.2017.07.004our was higher than that of F4 with 10% kepok banana peel flour, but it would be better if the banana peel was extracted.

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