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OPTIMIZATION OF BUTTERFLY PEA EXTRACT PEEL-OFF MASK WITH VARIATIONS OF POLYVINYL ALCOHOL AND IOTA CARRAGEENAN USING FACTORIAL DESIGN

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ABSTRACT

The butterfly pea flower (*Clitoria ternatea*) contains antioxidants that help combat premature skin aging. Peel-off masks are popular with consumers as topical formulations because they are unique and practical. An essential component of peel-off masks is the gelling agent Polyvinyl Alcohol (PVA), which acts as a film-forming agent. However, PVA at the wrong concentration can result poor mask physical properties. Combining PVA with other gelling agents can obtain peel-off masks with better physical properties. This study aims to identify the effect of PVA and iota carrageenan on the optimal formula of peel-off masks regarding physical properties, stability, and antioxidant activity. Butterfly pea flower extract was obtained by maceration in 70% ethanol. Optimization of PVA and iota carrageenan was conducted using a factorial design method, observing responses such as viscosity, spreadability, and drying time. Peel-off masks with butterfly pea flower extract were prepared with concentration variations of PVA (6%, 10%) and iota carrageenan (0.5%, 1%). The optimal formula was then tested for stability using cycling test method and for antioxidant activity using DPPH method. Increasing concentrations of PVA and iota carrageenan significantly affects viscosity, spreadability, and drying time, with a p-value < 0.05. The optimal peel-off mask formula contained 16% PVA and 0.5% iota carrageenan, with a viscosity of 16.473 cPs, spreadability of 5.6 cm, and drying time of 23 minutes and 11 seconds. The optimal formula demonstrated good stability during storage, with a significance p-value > 0.05, and was categorized as having weak antioxidant activity with an IC_{50} value 171.738 µg/mL.

Keywords: Butterfly Pea Flower, Polyvinyl Alcohol, Iota Carrageenan, Mask, Factorial Design

INTRODUCTION

One of issues concerning women's facial skin is aging (Rohiyati et al., 2020). Skincare products containing natural ingredients to inhibit aging are highly sought after by consumers due to their lower incidence of adverse side effects compared to chemical ingredients (Pal et al., 2017; Zakaria et al., 2018; Sunnah et al., 2019). Antioxidant compounds in skincare products can effectively prevent premature aging (Reveny & Umayah, 2016).

Butterfly pea flowers (*Clitoria ternatea*) are rich in antioxidants, primarily due to their high phenolic compound content. The 70% ethanol extract of butterfly pea flowers demonstrates potent antioxidant activity with an IC_{50} value of 41.36 ± 1.191 μg/mL. The antioxidant effect on the skin is maximized when the active substance is formulated in a topical dosage form, allowing for prolonged interaction and penetration into the skin (Andarina & Djauhari, 2017; Andriani & Murtisiwi, 2020). In previous research, butterfly pea flower extract has been utilized in facial cleansers, sunscreen creams, and micellar-based waters (Puspitasari et al., 2019; Dzakwan, 2020; Panda et al., 2020). However, formulation of butterfly pea flower extract into a peel-off mask has not been previously reported. Peel-off mask formulations contain critical gelling agents that determine the mask's physical qualities (Sulastri et al., 2016).

The gelling agent of polyvinyl alcohol (PVA) concentrations ranging from 10% to 16% serve as gelling agents capable of forming a film layer that can be easily peeled off after drying. PVA exhibits adhesive properties, forming a transparent, strong film that adheres well to the skin (Brick et al., 2014; Andini et al., 2017). However, the drawback of PVA film coatings lies in their stiffness and low flexibility. Excessive PVA concentrations in mask formulations can lead to increased viscosity, reduced spreadability, and faster drying times (Setiyadi & Qonitah, 2020). To address these issues, the combination of PVA with other gelling agents has been proposed as a solution. Previous research has demonstrated that combining PVA with HPMC influences the physical properties of peel-off masks, particularly by increasing viscosity and spreadability (Hidayati et al., 2019). However, the combination of PVA and carrageenan has not yet been explored in peel-off mask preparations. Iota carrageenan (ι-carrageenan) forms a gel from a collection of double helices that create a three-dimensional structure and contain water in the gaps, making it more elastic and stable (Akbari et al., 2020).

Carrageenan, derived from seaweed and containing sulfate groups, renders the polymer hydrophilic with a polysaccharide backbone. Based on its sulfate substituents, carrageenan exists in kappa, iota, and lambda forms. Carrageenan functions as a hydrocolloid, serving as a gel base in mask formulations (Diharmi et al., 2011; Fransiska & Reynaldi, 2020; Nailufa et al., 2021). ι-carrageenan, derived from Euchema spinosum, is utilized in topical preparations at concentrations ranging from 0.1% to 0.5%. A concentration of 1% produces a thick, soft, and elastic gel consistency with excellent homogeneity, viscosity, and spreadability compared to kappa and lambda types in topical gel preparations (Chaerunisaa et al., 2020; Nailufa et al., 2021). Furthermore, formulations prepared with ι-carrageenan exhibit stability without syneresis under freezing or thawing conditions (Diharmi et al., 2011; Fransiska & Reynaldi, 2020).

Therefore, this study aims to optimize the formulation to assess the effects of PVA and ι-carrageenan concentrations as gelling agents on the film formation of butterfly pea flower extract peel-off masks. The factorial design method will be employed to achieve peel-off masks with favorable physical and chemical properties.

METHODS

Tools and Materials

The equipment used includes an analytical balance (Ohaus®), rotary evaporator (MRC Rova-110®), pH meter (pH-009(I)A®), magnetic stirrer (IKA® C-MAG HS 7), Brookfield viscometer (NDJ-8S®), UV-Vis spectrophotometer (BIOBASE® BK-UV1900PC), blender (SANYO®), oven (Memmert®), and moisture balance (æADAMR®). The ingredients used are butterfly pea flower extract (*Clitoria ternatea*), PVA (Shuangxin), ι-carrageenan (Duchefa Biochemie), propylene glycol (SK Pic Global), methylparaben and propylparaben (Salicylates & Chemicals), methanol (Merck), DPPH (Himedia), ethanol (Bratachem), gallic acid (Sigma-Aldrich), Vitamin C (Sigma-Aldrich), and a comparison peel-off mask (Jordanie®).

Research Path

1. Simplicia

Butterfly pea flower samples were obtained from Sidoarjo Village, Buduran, East Java. Plant identification was conducted at the Botany Laboratory, Faculty of Mathematics and Natural Sciences, University of Lampung. Simplicia was prepared by drying butterfly pea flowers and subsequently grinding them into finer

particles using a blender until they became powder (Chaerunisaa et al., 2020).

2. Extraction

Butterfly pea flower powder was extracted using the maceration method with 70% ethanol. 200 g of simplicia was soaked in 1 L of solvent (1:5 ratio) for three days, stirred every 24 hours for 5 minutes (Asiani et al., 2012; Andriani & Murtisiwi, 2020). After soaking, the extract was squeezed out, and the residual material was macerated again in 500 mL of solvent. The combined filtrates from maceration and remaceration were concentrated using a rotary evaporator at 55°C (Pertiwi et al., 2022).

3. Characterization

The characterization of the extract included organoleptic testing, determination of water content, drying loss, and water-soluble essence content (Kementrian Kesehatan RI, 2017).

4. Phytochemical screening

Qualitative phytochemical screening was performed to determine the secondary metabolite content in the extract, including alkaloids, flavonoids, saponins, tannins, phenolics, and terpenoids (Cahyaningsih et al., 2019; Candra et al., 2021).

5. Determination of total phenol content

A stock solution of gallic acid (1000 ppm) was diluted with methanol-water (1:1) to prepare five concentrations for a calibration curve. A solution of butterfly pea flower extract (250 ppm) was prepared by dissolving the extract in methanol-water (1:1). Next, 100 µl of each solution was mixed with 3.95 mL of distilled water and 0.25 mL of Folin-Ciocalteu reagent, homogenized, and left for 8 minutes. Then, 0.75 mL of 20% Na_2CO_3 was added, mixed, and left for 30 minutes. The absorbance of each solution was measured at a wavelength of 745 nm using a UV-Vis spectrophotometer (Mulyani, 2018; Andriani & Murtisiwi, 2020). The total phenolic content was calculated as mg gallic acid equivalent per gram of sample using the formula:

$$\text{Total phenolic content} = \frac{C \times V \times fp}{g}$$

Where:

C = phenol concentration (x value)

V = volume of extract used (mL)

fp = dilution factor

g = weight of sample used (g)

6. Factorial design method

The mask base was optimized using the factorial design method in Design Expert software ver 12. The PVA and ι-carrageenan concentration ranges refer to research conducted by Hidayati et al. (2019) and Nailufa et al. (2021). The optimal range for low and high levels of gelling agent concentration is PVA (10% and 16%) and ι-

carrageenan (0.5% and 1%). The optimization responses included viscosity, spreadability, and drying time of the preparation. The recommended formula design for the factorial design method is listed in Table 1.

Table 1. Optimization design based on the factorial design method

Formula	PVA (%)	ι-Carrageenan (%)
F1	10	0,5
F2	10	1
F3	16	0,5
F4	16	1

7. Preparation of peel-off mask

The concentration of butterfly pea flower extract is 0.1%. The peel-off mask preparation is made using the formula shown in Table 2.

Table 2. Peel-off mask preparation formula

Composition	Formula (% b/v)			
	F1	F2	F3	F4
Butterfly pea extract	0,1	0,1	0,1	0,1
PVA	10	10	16	16
ι-Carrageenan	0,5	1	0,5	1
Propylene glycol	15	15	15	15
Methyl paraben	0,2	0,2	0,2	0,2
Propyl paraben	0,1	0,1	0,1	0,1
Aquadest	100	100	100	100

First, the PVA base is developed in distilled water at 80°C, then homogenized with a magnetic stirrer for 24 hours. Meanwhile, ι-carrageenan base is dissolved in distilled water, heated to 80°C, and stirred homogeneously. The butterfly pea flower extract is dissolved in propylene glycol and added gradually. The remaining distilled water is added to make 100 mL (Mardiyanto et al., 2018; Ratnasari & Kasasiah, 2018; Fransiska & Reynaldi, 2020).

8. Evaluation of peel-off mask

8.1 Organoleptic

Organoleptic tests include shape, color, and smell, observed directly using the five senses (Hidayati et al., 2019).

8.2 Homogeneity

0.1 g sample is spread evenly on a slide to check for any coarse grains or uneven mixing (Hidayati et al., 2019; Muflihunna et al., 2019).

8.3 pH

The pH test uses a pH meter, ensuring the preparation matches the skin pH of 4.5-6.5 (Andini et al., 2017; Sunnah et al., 2019).

9. Evaluation of optimal formula response

9.1 Viscosity

The viscosity test employs a Brookfield viscometer with spindle number 4 and a rotation speed of 30 rpm. Ideal viscosity ranges from 6000-24000 cPs (Andini et al., 2017).

9.2 Spreadability

1 g sample is placed on a transparent glass and subjected to a 125 g load for 1-2 minutes. The spread diameter is measured;

good spreadability ranges from 5-7 cm (Hidayati et al., 2019; Tanjung et al., 2021).

9.3 Drying time

0.5 g sample is spread on a 7x7 cm glass and dried in an oven at 36 ± 2°C until a 1 mm thin film forms. Ideal drying time is 15-30 minutes (Vieira et al., 2009).

10. Determination of optimal formula

Data analysis was conducted using analysis of variance (ANOVA) to determine the effect of PVA and ι-carrageenan optimization factors, which influence the response optimization with a significance value of $p < 0.005$. Interaction curves and 3D surface plots show the response analysis results. The optimal formula for PVA and ι-carrageenan is determined using goal criteria with a maximum desirability value (close to 1) and overlay plot graph. Optimization results indicate optimal viscosity, spreadability, and drying time for the butterfly pea flower extract peel-off mask.

11. Stability

The preparation is stored at 4°C for 24 hours, followed by storage at 40°C for another 24 hours, constituting one cycle. This test is repeated for up to 6 cycles. Organoleptic, syneresis, viscosity, and pH observations are conducted on preparations before and after the cycling test treatment (Rompis et al., 2009). Test results are analyzed using SPSS statistics with the paired-sample T-test method.

12. Antioxidant activity

The mother liquor of butterfly pea flower extract and the vitamin C comparator were made at a concentration of 1000 ppm. Meanwhile, the mother liquor of the butterfly pea flower peel-off mask and Jordaniea® peel-off mask is made at a concentration of 10,000 ppm. Each mother liquor was shaken quickly for 5 minutes, then filtered, and the filtrate was taken. Then, a calibration curve was created for each mother solution using five different concentration series. Next, 2 mL of each concentration series was taken, and 2 mL of DPPH 40 ppm was added, shaken homogeneously, and allowed to stand for the operating time. Then, the absorbance of the sample was measured using a UV-Vis spectrophotometer at a maximum wavelength of 515 nm (Izzati, 2014; Tanjung et al., 2021). The absorbance (abs) at each sample and comparison concentration was recorded to calculate the % inhibition value.

$$\%\ Inhibition = \frac{\text{abs blank} - \text{abs sample}}{\text{abs blank}} x100\%$$

The % inhibition value is plotted against concentration to calculate IC_{50} using the linear regression method, expressed with a y value of 50 and x as IC_{50} (Purwati &

Verryanti, 2016). From the linear regression equation y=a+bx, IC_{50} is calculated using the formula:

$$IC_{50}\ (x) = \frac{50-a}{b}$$

RESULTS AND DISCUSSION

1. Extraction

The extraction of butterfly pea flower simplicia powder was carried out using the maceration method because it is simple and does not require heating, thereby preventing damage to thermolabile compounds. Butterfly pea flowers contain phenolic compounds that are not heat-resistant and easily oxidize at temperatures over 90°C (Dewata et al., 2017). Ethanol at 70% was chosen as the maceration solvent because ethanol above 70% can reduce solvent polarity, thereby decreasing the yield obtained. Previous research has reported that the 70% ethanol extract of butterfly pea flowers exhibited a better IC50 value compared to the 96% ethanol extract (Andriani & Murtisiwi, 2020). The yield from the extraction of butterfly pea flowers was 40.28%, consistent with other studies reporting a yield of 46.2%. Differences in results obtained may be influenced by extraction time, solvent quantity, and sample weight used in extraction (Andriani & Murtisiwi, 2020).

2. Characterization

The results of the extract characteristics are shown in Table 3. The water content and drying shrinkage tests met the standard of less than 10% (Arifah et al., 2022). If the water content is too high, it can cause microbial growth, which affects the stability of the extract (Utami et al., 2017). Water content can affect shelf life because the presence of water can trigger various metabolic activities during storage, such as enzymatic, microbial, and chemical processes, as well as non-enzymatic reactions. These can lead to changes in the appearance, texture, taste and overall quality of the material (Nurhidayah et al., 2019). Drying shrinkage testing is conducted to provide an overview of the internal and external mineral content of an extract. The combustion process (ashing) causes organic compounds and their derivatives to break down and evaporate, leaving behind mineral elements and inorganic compounds that are closely related to the mineral content, purity, and cleanliness of the material (Najih & Nurhidajah, 2011). The water-soluble essence content is higher than the ethanol-soluble essence content because it contains more polar compounds.

Table 3. Extract characterization results

Inspection	Results
Organoleptic	Thick gel form, blue color, and distinctive odor
Water content	6,28% ± 0,74
Drying shrinkage	9,54% ± 0,10
Water soluble content	14,33% ± 0,47
Ethanol soluble content	12,67% ± 3,40

3. Phytochemical content

The results of the phytochemical screening of butterfly pea flower extract are presented in Table 4. The extract showed positive results in phytochemical screening, consistent with previous research indicating the presence of alkaloids, flavonoids, tannins, saponins, terpenoids, and phenolics (Arifah et al., 2022).

Table 4. Phytochemical screening results

Secondary Metabolites	Results
Alkaloids	+
Flavonoids	+
Tannin	+
Saponins	+
Terpenoids	+
Phenolic	+

The total phenolic content of the extract was analyzed using the Folin-Ciocalteu method. The phenol content equivalent of gallic acid was used as a standard at a maximum wavelength value of 745 nm. The total phenolic content of butterfly pea flower extract was 88 mg GAE/g sample. These results differ from the total phenolic content in other studies, which reported 61.7 mg GAE/g sample. These differences can be attributed to variations in growing regions (geography, temperature, climate), plant characteristics, and extraction solvents (Rabeta & An Nabil, 2013).

4. Preparation

Peel-off masks are formulated by dissolving PVA in distilled water at 80℃ using a magnetic stirrer to enhance solubility. PVA is sensitive to overheating, which can cause water evaporation and lead to the formation of a thick layer on the solution's surface (Mardiyanto et al., 2018). Meanwhile, ι-carrageenan is dissolved first in distilled water and heated to 80℃ with constant stirring to prevent clumping, as direct addition of hot water can cause ι-carrageenan to become sticky and clump. The solutions of both bases are then mixed and heated at 80℃ for 10-15 minutes to ensure thorough combination. Peel-off mask gel preparations contain a significant water component; therefore, a combination of preservatives is added to enhance antimicrobial activity and prevent bacterial growth. Preservatives with a concentration of 0.2% methyl paraben and 0.1% propyl paraben are used because they have broad-spectrum antimicrobial activity within a pH range of 4-8. The combination is necessary because it provides a synergistic effect,

enhancing antimicrobial activity. The effectiveness of methyl paraben can also be increased if propylene glycol is added at a concentration of 2-5% (Rowe et al., 2009). Increased antimicrobial activity is required in gel preparations containing hydrophilic PVA and ι-carrageenan polymers. Propylene glycol acts as a humectant to retain water and minimize excessive evaporation during storage and application on the skin.

5. Evaluation of peel-off mask

Organoleptic evaluation aims to assess the physical properties of peel-off masks related to consumer comfort during use. Organoleptic observations for each formula showed consistent results in terms of shape, color, odor, and homogeneity. The butterfly pea flower extract peel-off mask is illustrated in Figure 1.

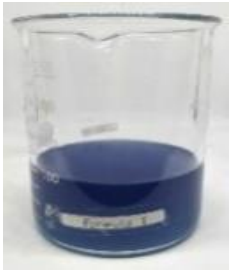

Figure 1. Peel-off mask containing the extract

All formulations exhibit a sticky gel texture due to the presence of PVA and ι-carrageenan. PVA's adhesive properties impart a sticky texture to the mask gel, skin to glue. The blue color of the mask comes from butterfly pea flower extract, and its scent is dominated by the aroma of PVA. Homogeneity testing ensures even distribution of the gel mask preparation, with no coarse particles or lumps found in any formulation. pH testing is conducted to ensure that the preparation's pH aligns with the skin's pH. Masks with either a very low or highly acidic pH can increase the risk of irritation, while those with a high or excessively alkaline pH can lead to dry, flaky skin. Based on pH test results, all peel-off mask formulations meet the pH requirements suitable for skin, ranging from 5.67 ± 0.19 (F1), 5.77 ± 0.05 (F2), 5.50 ± 0.22 (F3), to 5.27 ± 0.29 (F4). The pH value of the preparation can be influenced by the PVA content, which typically ranges between pH 5 and 8 in high-concentration mask formulations (Arinjani & Ariani, 2019).

6. Response data analysis

Viscosity is a fluid's resistance to flow and is essential in mask preparations for ease of application to the skin. A low viscosity mask gel is easier to apply but may cause it to run off the face due to its fluidity. Conversely, high viscosity makes application difficult (Sulastri et al., 2016). Viscosity results indicate that all formulas meet the requirement of 6000-24000 cPs. The spreadability test assesses how well the preparation spreads over the skin's surface.

Only formulas F2, F3, and F4 meet the optimal spreadability of 5-7 cm. Additionally, only formulas F3 and F4 meet the drying time requirement of 15-30 minutes (Andini et al., 2017; Rum et al., 2021). Detailed evaluation results including viscosity, spreadability, and drying time are presented in Table 5.

Table 5. Evaluation results of response to peel-off mask with butterfly pea flower extract

Formula	Response		
	Viscosity (cPs)	**Spreadability (cm)**	**Drying Time (minutes)**
F1	0,1	0,1	0,1
F2	10	10	16
F3	0,5	1	0,5
F4	100	100	100

Response data optimization of formulas uses an optimization model with R2, adjusted R2, predicted R2, and adequate precision to evaluate model suitability. A good model is indicated by an R2 value close to 1 for desired results (Ramadhani et al., 2017). The difference between adjusted R2 and predicted R2 values should be less than 0.2 to indicate model feasibility (Othman et al., 2017). ANOVA data confirms primary effects models with significant p-values ($p < 0.05$) for all responses. Detailed response data analysis is presented in Table 6.

Table 6. ANOVA results and statistical response parameters

Response data	Parameter				
	p-value	**R^2**	***Adj.* R^2**	***Pred.* R^2**	***Ad. Pre***
Viscosity	0,0022*	1	1	0,999	667,17
Spreadability	0,0276*	0,999	0,997	0,987	55,425
Drying Time	0,0034*	1	1	0,999	327,96

Note: * = Significant; Adj = Adjusted; Pred = Predicted; Ad. Pre = Adequate Precision

Half-normal plot standard effects are insignificant if points align on a straight line (Secula et al., 2013). Figure 2 displays half-normal plots where factors (PVA and ι-carrageenan) deviate significantly from the straight line, impacting viscosity, spreadability, and drying time responses. Optimization response ANOVA results are detailed in Table 7.

Table 7. Coefficient value, p-value, and percent (%) contribution to optimization response

Response	PVA	ι-Carrageenan
Viscosity		
Coefficient	+ 2,787x10^5	+ 98726,25
p-value	0,0015*	0,0042*
% Contribution	88,8525%	11,147%
Spreadability		
Coefficient	-0,8250	-0,3750
p-value	0,0193*	0,0424*
% Contribution	82,8317%	17,1103%
Drying Time		
Coefficient	-14,80	-1,35
p-value	0,0022*	0,0236*
% Contribution	99,1737%	0,825165%

Note: * = Significant influencing factors ($p < 0.05$)

All optimization responses with influencing factors have p-values < 0.05. PVA has a more significant influence than ι-carrageenan, contributing a higher

percentage to viscosity than ι-carrageenan. A positive coefficient for viscosity indicates a direct relationship where concentration increases lead to higher viscosity. Conversely, negative coefficients for spreadability and drying time suggest an inverse relationship where higher concentrations reduce spreadability and increase drying time.

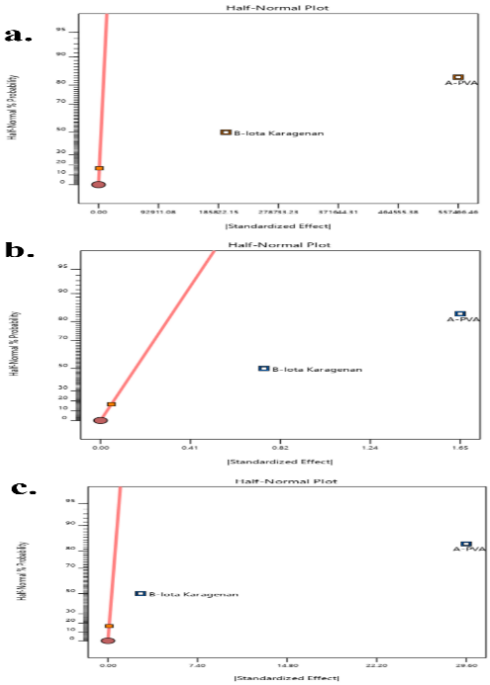

Figure 2. Half-normal plot of optimization response (a) viscosity; (b) spreadability; and (c) drying time

Predicted vs. actual curve results are shown in Figure 3, demonstrating a strong correlation between actual and predicted responses.

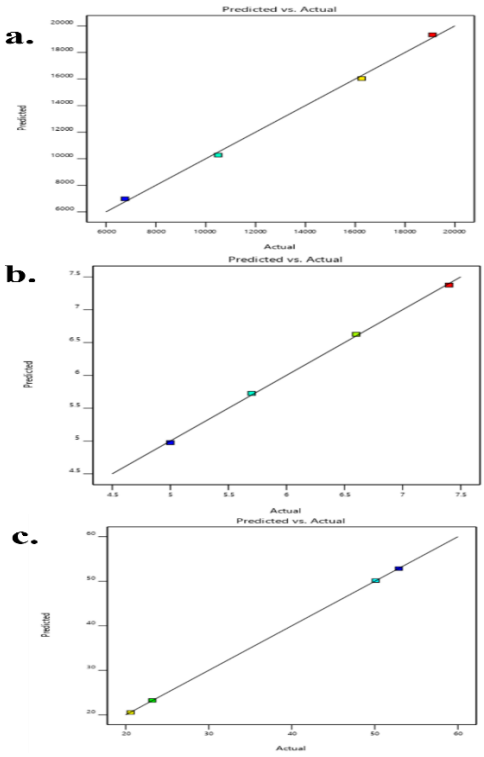

Figure 3. Predicted vs. actual curve graph of optimization response (a) viscosity; (b) spreadability; and (c) drying time

A 3D surface plot in Figure 4 illustrates interactions between factors to determine optimal points for viscosity, spreadability, and drying time. The literature suggests that increasing PVA concentration thickens gel viscosity due to enhanced polymer fiber binding (Noviani et al., 2016; Arinjani & Ariani, 2019). Conversely, higher concentrations may reduce spreadability and hasten drying times due to increased molecular size and faster evaporation (Arinjani & Ariani, 2019; Purnamasari et al., 2021).

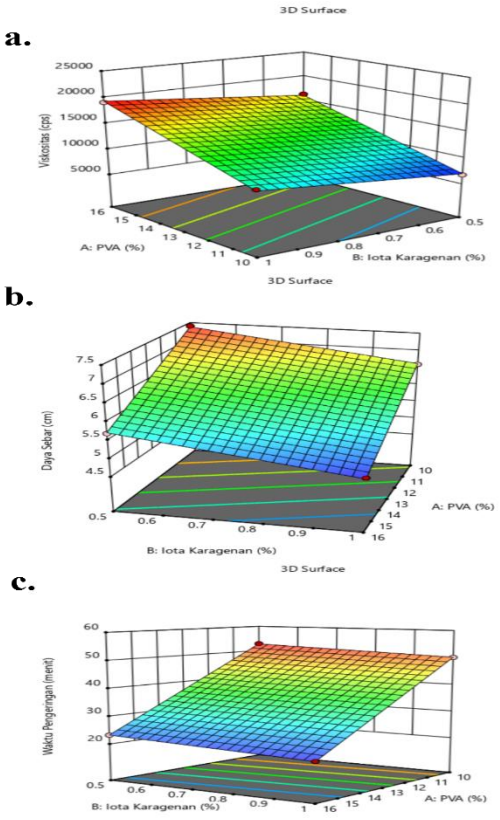

Figure 4. 3D response surface plot of optimization (a) viscosity; (b) spreadability; and (c) drying time

7. Optimize peel-off mask formula

Viscosity and spreadability responses are maximized, while drying time response is minimized. Maximizing viscosity reduces drying time, while enhancing spreadability facilitates easier application on the skin, thereby minimizing drying time according to the criteria. Based on these criteria, it is evident that the excellent optimum formula is F3, with a desirability value close to 1 (0.921), achieved using 16% PVA and 0.5% ι-carrageenan, as shown in the overlay plot graph in Figure 5. The recommended optimization area is highlighted in yellow, while gray areas indicate zones that do not meet the optimization response criteria.

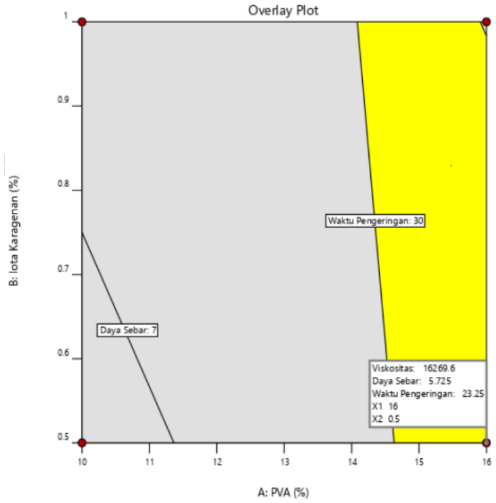

Figure 5. Overlay plot graph

Evaluation of the optimal peel-off mask formula provides response results, as detailed in Table 8. The actual response values closely match the predicted responses. Confidence Interval (CI) and Prediction Interval (PI) values validate the optimal formula response data. The 95% CI represents the range between the two values of the sample, while the 95% PI denotes the lower and upper limits of the prediction results. The actual response results fall within both the 95% CI and 95% PI ranges (Pratiwi et al., 2021).

Table 8. Predicted, actual, and optimal formula verification values

Response	Pred	Act	95% CI		95% PI	
			Low	High	Low	High
Viscosity (cPs)	16,26	16,47	16,05	16,48	16,26	16,89
Spreadability (cm)	5,7	5,6	5,2	6,3	4,8	6,5
Drying Time (minutes)	23,25	23,11	22,15	24,35	21,32	24,59

Note: Pred = Predicted; Act = Actual

8. Evaluation of peel-off mask formula

The stability test was conducted on the optimal formula peel-off mask to assess its stability during storage. Organoleptic observations of the mask preparations indicated stability, with no changes observed in shape, color, or odor before and after the cycling test. This stability suggests that the use of 16% PVA and 0.5% ι-carrageenan effectively absorbs water in the gel during the stability test. Table 9 presents the results of the stability test parameters viscosity and pH.

Table 9. Viscosity and pH stability test results

Stability	Average ± SD	
	Viscosity	pH
Before	16,473 ± 0,025	5,4 ± 0,05
After	16,433 ± 0,040	5,7 ± 0,12

Viscosity decreased after the cycling test, consistent with previous research attributing this to temperature effects on the polymer used. Higher temperatures cause polymer chains to disentangle, increasing particle distance and lowering viscosity. Conversely, lower temperatures lead to chain entanglement, reducing particle distance and increasing viscosity (Reveny & Umayah, 2016). The pH increased post-cycling test, aligning with studies attributing pH changes to media decomposition producing acids or bases during the test (Limbong et al., 2021; Syam et al., 2021). Statistical data analysis pre and post-cycling test in the paired-sample T-test showed non-significant changes ($p > 0.05$) in viscosity (0.196) and pH (0.073), indicating good stability of the peel-off mask during storage.

9. Antioxidant Activity

Antioxidant activity was assessed using the DPPH method on butterfly pea flower extract, vitamin C, butterfly pea flower extract peel-off mask, and Jordaniea® peel-off mask. Table 10 presents the results of IC_{50} levels and antioxidant activity categories.

Table 10. Results of IC_{50} levels and antioxidant activity categories

Sample	IC_{50} values (µg/mL)	Category
Butterfly pea flower extract	40,735 µg/mL	Very strong
Vitamin C	5,142 µg/mL	Very strong
Peel-off mask of butterfly pea flower extract	171,738 µg/mL	Weak
Jordaniea®	117,669 µg/mL	Moderate

The IC_{50} value denotes the concentration of an antioxidant compound required to reduce radical compounds by 50%, indicating its antioxidant activity

capability. Antioxidant activity categories are classified as very strong (IC_{50} < 50 µg/mL), strong (50-100 µg/mL), moderate (100-150 µg/mL), and weak (150-200 µg/mL) (Rum et al., 2021). Butterfly pea flower extract and vitamin C exhibit extremely strong antioxidant activity, with vitamin C demonstrating better activity due to its lower IC_{50} value. However, the optimal formula for the butterfly pea flower extract peel-off mask falls into the weak antioxidant activity category. Jordaniea® peel-off mask, containing green tea with antioxidant properties, is categorized as moderate. The antioxidant activity of butterfly pea flower extract in the peel-off mask formulation is reduced, possibly due to other added components affecting the IC_{50} value. Moreover, the high viscosity of the optimal formula peel-off mask could hinder the escape of butterfly pea flower extract from polymer entrapment, thereby affecting its interaction with the DPPH compound and resulting in lower antioxidant activity.

CONCLUSIONS

The research findings demonstrate that PVA and ι-carrageenan significantly influence optimal responses in terms of viscosity, spreadability, and drying time ($p < 0.05$). The optimal peel-off mask of butterfly pea flower extract formula, with 16% PVA and 0.5% ι-carrageenan, exhibits stability during storage ($p > 0.05$) and falls into the weak antioxidant activity category, with an IC_{50} value of 171.738 µg/mL.

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