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OPTIMIZATION OF BUTTERFLY PEA FLOWER (*Clitoria ternatea* L.) LOADED ALGINATE-XANTHAN GUM BASED PEEL-OFF GEL MASK USING RESPONSE SURFACE METHODOLOGY

Aurum Dwi Utami, Tubagus Akmal*
Akademi Farmasi Bumi Siliwangi

*Email: tubagus.akmal93@gmail.com

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ABSTRAK

Bunga telang terbukti mengandung fenolik dan flavonoid yang bersifat sebagai antioksidan alami yang dapat dimanfaatkan sebagai *skincare* seperti masker *peel-off* gel. Penelitian ini bertujuan untuk mendapatkan formula optimal masker *peel-off* gel dengan variasi konsentrasi *sodium alginate* (SA) sebagai *gelling agent*, Xanthan Gum (XG) sebagai *viscosity modifier* dan Propilenglikol (PG) sebagai *plasticizer* yang dapat menjadi inovasi produk *skincare* berbasis bahan alami. Optimasi formula basis dilakukan dengan *Box-behnken Design* menggunakan perangkat lunak *Design Expert* v.13.0 dengan respon uji nilai pH, viskositas, daya sebar, waktu kering dan ketebalan. Pengujian stabilitas dianalisis menggunakan *Graphpad prism* v.10. Proses optimasi basis menghasilkan model penelitian yang signifikan dengan hasil formula optimum 1,5% SA, 1% XG dan 10% PG dengan nilai desirabilitas sebesar 0,933. Nilai ini menunjukkan kombinasi variabel yang digunakan menghasilkan karakteristik sediaan yang baik. Formula optimum tersebut memiliki nilai pH 5,87, viskositas 32016 cPs, daya sebar 5,25 cm, waktu kering 19.09 menit dan ketebalan 51,17 µm. Formula ini selanjutnya digunakan sebagai basis untuk masker *peel-off* gel ekstrak bunga telang. Hasil uji stabilitas menunjukkan perbedaan yang signifikan (*t-test*, $p<0,05$) untuk pH, viskositas, daya sebar, uji ketebalan dan untuk uji waktu kering menunjukkan tidak ada perbedaan yang signifikan (*t-test*, $p>0,05$) pada formula masker gel *peel-off* ekstrak bunga telang terhadap basis formula. Hasil uji stabilitas fisik menunjukkan bahwa formula masih memenuhi persyaratan gel.

Kata kunci: Antioksidan, *Box-behnken Design, Clitoria ternatea* L, Masker *Peel-off* gel, Stabilitas

ABSTRACT

Butterfly pea flowers are proven to contain phenolics and flavonoids, which are natural antioxidants that can be utilized in skincare such as peel-off gel masks. This study aims to obtain the optimal formula of peel-off gel masks with variations in the concentration of sodium alginate (SA) as a gelling agent, Xanthan Gum (XG) as a viscosity modifier and Propylene glycol (PG) as a plasticizer which can be an

innovative skincare product based on natural ingredients Optimization of the base formula was carried out by Box-behnken Design using Design Expert v.13.0 software with test responses of pH, viscosity, spreadability, dry time and thickness. The base optimization process resulted in a significant research model with the optimum formula of 1.5% SA, 1% XG and 10% PG with a desirability value of 0.933. This value indicates that the combination of variables used produces good preparation characteristics. The optimum formula has a pH value of 5.87, viscosity of 32016 cPs, spreadability of 5.25 cm, dry time of 19.09 minutes and thickness of 51.17 μm. This formula was used as the base peel-off gel mask formula of butterfly pea extract (BFE). The stability test showed significant differences (t-test, p<0.05) for the pH, viscosity, spreadability, and thickness tests. The drying time test showed no significant difference (t-test, p>0.05) in the peel-off gel mask formula of BFE against the formula base. The stability test results show the formula still meets the gel criteria.

Keywords: *Antioxidant, Box-behnken Design, Clitoria ternatea* L.*, Peel-off Gel Mask, Stability*

INTRODUCTION

The prevalence of side effects linked to synthetic ingredients supports the exploration of natural alternatives. has been popularly used in various fields due to the flavonoids content, the largest group of phenolic compounds which serve as natural antioxidants and are ideal for incorporation into skincare products (Jelantik & Cahyaningsih, 2022).

The peel-off gel mask is a skin care product that aids in enhancing facial skin imperfections, such as signs of premature aging. This product offers the advantage of forming an elastic film that, once dried, can be effortlessly removed. Additionally, it can be reapplied without the necessity of water (Anandha kifli et al., 2022).

The materials that support these characteristics can utilize SA, which serves as both a gelling agent and a film-forming agent, exhibiting biocompatible and biodegradable properties. Furthermore, XG serves as a viscosity modifier that demonstrates compatibility with various ingredients and exhibits excellent stability, contributing to the formulation of a high-quality product (Ariani et al., 2023; Nugrahaeni et al., 2021). Additional materials that may be utilized include plasticizers. PG is chosen as a plasticizer to enhance the flexibility and elasticity of the product

formulation (Krisnadi et al., 2019).

A contemporary experimental design methodology can be employed to validate the optimal formulation. The Box-Behnken design is used to formulate a BFE-based peel-off gel mask with SA and XG, ensuring compliance with the required parameters for natural mask approval.

RESEARCH METHOD

This study utilized dried butterfly pea flowers from the local market, and all materials are of pharmaceutical grade.

Extraction

The extraction of butterfly pea flower was carried out in accordance with the methodology outlined by Akmal et al., (2024) with modification using Ultrasonic Assisted Extraction (UAE), utilizing a ratio of 1:20 for simplicia powder and 96% ethanol concentration he sample is centrifuged and concentrated using a water bath.

Preparation Peel-off gel mask

Table. 1 Design of Experiment for Optimization of Peel-off gel mask Formula

Independent Variables (X)	Min (%)	Max (%)
SA	1	2
XG	0,5	1
PG	5	10
Dependent Variables (Y)	**Constraints**	
pH	In range	
Viscosity	In range	
Spreadability	In range	
Drying time	Minimize	
Thickness	In range	

Ascorbic acid (0.5g) was dissolved in distilled water, followed by the gradual incorporation of SA and XG into the ascorbic acid solution, which was stirred gently for 45 minutes using an overhead stirrer. A separate beaker was utilized to stir PG, phenoxyethanol (1g), and TEA (0.19g) with a magnetic stirrer at a speed of 400 rpm for a duration of 5 minutes. The solution was subsequently incorporated into the expanded gel base and stirred for a duration of 10 minutes to achieve a homogeneous gel (Narayanan et al., 2022). The peel-off gel mask was assessed for various parameters including organoleptic properties, homogeneity, pH, viscosity, spreadability, drying time, and thickness to identify the optimal formulation, which was achieved by incorporating 3% BFE.

Evaluation Peel-Off Mask

The purpose of the organoleptic test is to examine the alterations in consistency, odor, and discoloration. The homogeneity test revealed a

uniform arrangement, with no coarse grains detected when utilizing a pressed glass object. The pH test is conducted utilizing a pH meter along with its electrodes. The electrode is immersed in the sample, and the pH meter will display the corresponding pH value (Lestari et al., 2022). A viscosity test was conducted utilizing a Brookfield LV Viscometer. To assess spreadability, the sample was positioned between two acrylic glass slides, onto which weights were applied (Azizah et al., 2024). The drying time test involved recording the duration required for the gel to dry on a hotplate maintained at 37°C (Hariyadi et al., 2021). The final thickness test is conducted by measuring the formed gel at various points using a digital thickness gauge (Bushair P et al., 2023). The optimum formula underwent a physical stability test over a 28-day storage period, with assessments conducted on days 0, 7, 14, 21, and 28 at a controlled room temperature (15 to 30 °C) (Wijayanti et al., 2015).

Statistical Data Analysis

The BBD design method was used to formulate experimental data using Design-Expert v13.0 which used to assess the influence of independent variables as factors (X) and dependent variables as responses (Y). BBD is an optimization method that uses three independent variables and offers the advantage of excluding extreme combinations of factor levels, which reduces the risk of experimental failure (Zahara et al., 2023). The data obtained from the dependent variable results in the stability test were analyzed using GraphPad Prism v10.

RESULT AND DISCUSSION

Extraction

The extract yield achieved was 35.4%. The requirement for the yield of thick extracts is that the value is not less than 10% (Indriaty et al., 2022).

Optimization Peel-off Gel Mask

To assess formulation accuracy and identify the best formula, 15 experiments with three center points were conducted using the Box-behnken Design. Results are presented in Table 3.

Table 3. Box Behnken design with their observed responses of peel-off gel mask

Run	pH	Viscosity (cPs)	Spreadability (cm)	Drying Time (minutes)	Thickness (µm)
1	5.25	2466.67±103.28	7.80±0.05	21.47	38

Run	pH	Viscosity (cPs)	Spreadability (cm)	Drying Time (minutes)	Thickness (µm)
2	5.42	4433.33±81.65	7.79±0.09	20.12	47
3	5.51	19083.33±386.87	6.18±0.16	20.57	51
4	5.48	10900.00±167.33	6.69±0.04	22.88	42
5	5.41	3516.67±75.28	7.75±0.05	24.92	49
6	5.43	7800.00±167.33	6.00±0.17	22.23	36
7	5.40	9800.00±189.74	6.58±0.21	23.68	47
8	5.27	64000.00±565.68	4.90±0.13	19.20	50
9	5.39	17250.00±408.66	6.00±0.18	21.77	34
10	5.53	12716.70±348.81	5.90±0.09	21.02	51
11	5.41	45900.00±374.16	4.87±0.08	19.85	53
12	5.31	9666.67±81.65	6.27±0.10	23.10	46
13	5.33	25533.33±136.63	5.57±0.16	22.57	45
14	5.53	9633.33±51.64	5.93±0.05	24.18	49
15	5.31	26000.00±299.44	5.22±0.04	23.08	54

Two models were generated, quadratic and linear models. The resulting correlation value (R^2) for the pH, viscosity, spreadability, drying time and thickness tests were 0.6039; 0.9847; 0.7589; 0.9685; and 0.7351.

Effect of Independent Variables on Dependent Variables

Dependent variables are shown in Table 3. In the pH test, SA significantly influences the results, SA increases pH by contributing OH^- ions from its carboxyl group (Sunarharum et al., 2020).

Viscosity is influenced by SA and PG, with higher concentrations reducing viscosity. This occurs because ascorbic acid makes the solution acidic, destabilizing SA and hindering gel formation, while PG adds elasticity to the gel (Maharani et al., 2017).

XG significantly affects spreadability, which inversely correlates with viscosity: higher viscosity reduces spreadability, and vice versa (Nugrahaeni et al., 2021).

Drying time results show PG has the greatest impact, slowing drying due to its hygroscopic nature, which absorbs moisture and reduces water evaporation (Ardana et al., 2015).

In the thickness test, PG as a plasticizer increased the thickness of the film. This is because plasticizer will increase the constituent polymers in the film matrix, thus increasing the film thickness (Madu et al., 2022).

Confirmation Optimum Formula

Optimization of the peel-off gel mask formula and results shown in Table 5. Desirability and predicted response values are also illustrated in Figure 1.

Table 5. Optimum Formula and Test Response Results

SA (%)	XG (%)	PG (%)	Desirability
1.5	1	10	0.933
Responses	**Predicted Values**	**Actual Values**	**%Error**
pH	5.46	5.87 ± 0.00	7.50%
Viscosity (cPs)	40136	32016 ± 325.06	-20.23%
Spreadability (cm)	5.19	5.25 ± 0.12	1.15%
Drying time (minutes)	19.79	19.09 ± 0.31	-3.53%
Thickness (µm)	53.76	51.17 ± 3.92	4,81%

The desirability value that is close to 1 is the expected value because the optimum point that has a high desirability value or is close to 1 is the best formula (Sopyan et al., 2022). Three responses were lower and two higher than predicted values. While viscosity showed the highest error, all results remained within acceptable ranges. These outcomes indicate the formula's effectiveness as a base for developing BFE peel-off gel masks, which underwent stability tests over 28 days at 15–30°C

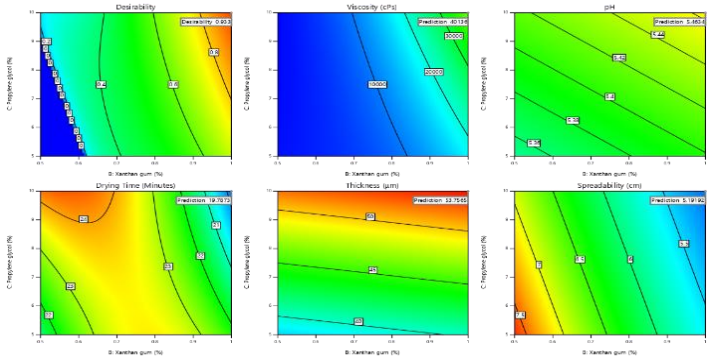

Figure 1. Desirability index plot for optimum formula conditions of peel-off gel mask

Stability Test Results of Peel-off Gel Mask

The observed organoleptic tests included assessments of consistency, odor, and color. The formula base exhibits a viscous consistency, characterized by a unique odor and a subtle yellow hue. On the 21st day, a color change to yellow was observed; this occurred as a result of the degradation of vitamin C in the formula, leading to a gradual shift towards a more yellow hue (Tonthawi & Musfiroh, 2023). The formula incorporating extract results in a thicker consistency owing to the reduced water content, exhibits a unique extract aroma, presents a dark green hue, and remains unchanged in the organoleptic evaluations.

The peel-off gel mask formulations, whether based on specific formulas or enriched with extracts, exhibit excellent homogeneity, as evidenced by the even distribution of all particles observed on the object glass, with no clumping present. In alignment with earlier studies carried out by Lestari et al., (2022). Peel-off gel masks that exhibit excellent homogeneity are defined by the lack of coarse particles within the formulation.

The pH in both formulas decreased (Figure 2), and this reduction in pH during storage was affected by CO_2, as the CO_2 interacts with water, resulting in an acidic preparation (Akmal et al., 2022). Despite a decrease in pH during preparation, the final pH value stayed within the acceptable range for topical products (4.5—6.5). pH must be considered as it affects the safety and comfort of the product, which has the potential to cause irritation (Lestari et al., 2022).

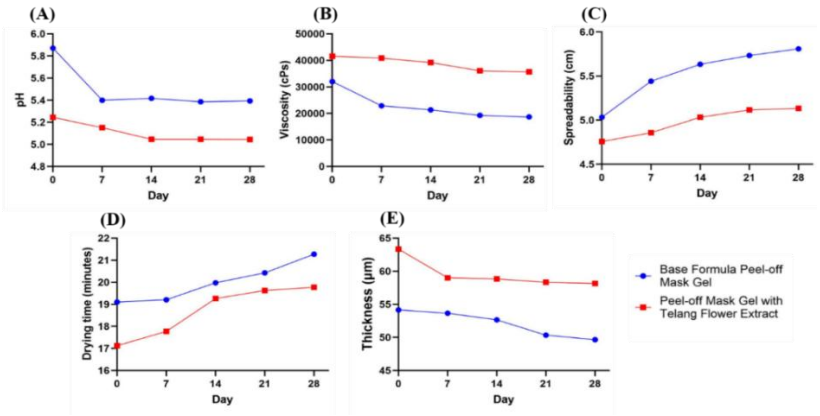

Figure 2. The values of pH (A), viscosity (B), spreadability (C), drying time (D) and thickness (E) for 28 days

Viscosity affects the ease of use of the product. The viscosity test of formula bases and formulas with extracts indicates a reduction in viscosity (Figure 2). External influences such as storage temperature or humidity can impact viscosity. Inadequate airtight packaging can lead to an increase in the gel's water content, as it tends to absorb moisture from the surrounding environment. Additionally, it is important to recognize that the duration of storage influences viscosity; as storage time increases, there is a higher likelihood of a reduction in the viscosity of the formulation. Despite the reduction in viscosity, all samples continue to meet

the gel viscosity standards (2000-50000 cPs) (Akmal et al., 2022; Samsul et al., 2022).

The increase in spreadability (Figure 2) resulted from a drop in viscosity; lower viscosity correlates with increased spreadability. Changes in spreadability remain within the stipulated range of 5-7 cm (Akmal et al., 2022). Spreadability is related to the product's ability to be evenly applied to the face. Good spreadability ensures optimal distribution of active ingredients.

Both formulas exhibited an extended drying time (Figure 2). The drying time of peel-off masks is influenced by viscosity; a reduction in viscosity results in an extended drying period. Prolonged storage may also contribute to this issue, as the formula has significant water content, hence prolonging the drying time of the preparation. The extended duration remains within the stipulated range of 15-30 minutes (Ermawati et al., 2022). The primary reason for the reduction in thickness (Figure 2) may be attributed to the decline in viscosity resulting from the prolonged gel storage time, which consequently elevates the water content in the gel. The rise results in diminished gel strength, thereby yielding reduced thickness. The thickness influences the removability of the peel-off gel mask (Wati et al., 2023).

The statistical analysis (t-test) stability test data showed insignificant differences for the drying time test, and the other tests showed significant differences.

CONCLUSIONS

SA and XG serve as natural bases, butterfly pea flower acts as an antioxidant, and PG adds elasticity in peel-off gel masks. The optimal formula, determined by Box-Behnken design, consists of 1.5% SA, 1% XG, and 10% PG with a desirability of 0.933. The stability test showed significant differences (t-test, $p<0.05$) for the pH, viscosity, spreadability, thickness tests and showed no significant difference (t-test, $p>0.05$) for the drying time test between the two formulas. Both the base formula and BFE formulas, during the stability test met all gel evaluation criteria. These findings suggest the potential application of the optimized peel-off

gel mask in the skincare industry, particularly as a natural skincare.

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