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Judul artikel	:	Optimization Formula of Kolang-Kaling (Arenga Pinnata.) Peel-Off Gel Mask with Combination PVA - CMC Na as Gelling Agent on Simplex Lattice Design
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2	Review Discussions: Revisions Required and reviewer comment	11 Februari 2025	Hal. 2
3	Submit revised manuscript	14 Februari 2025	Hal. 3
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Pengusul

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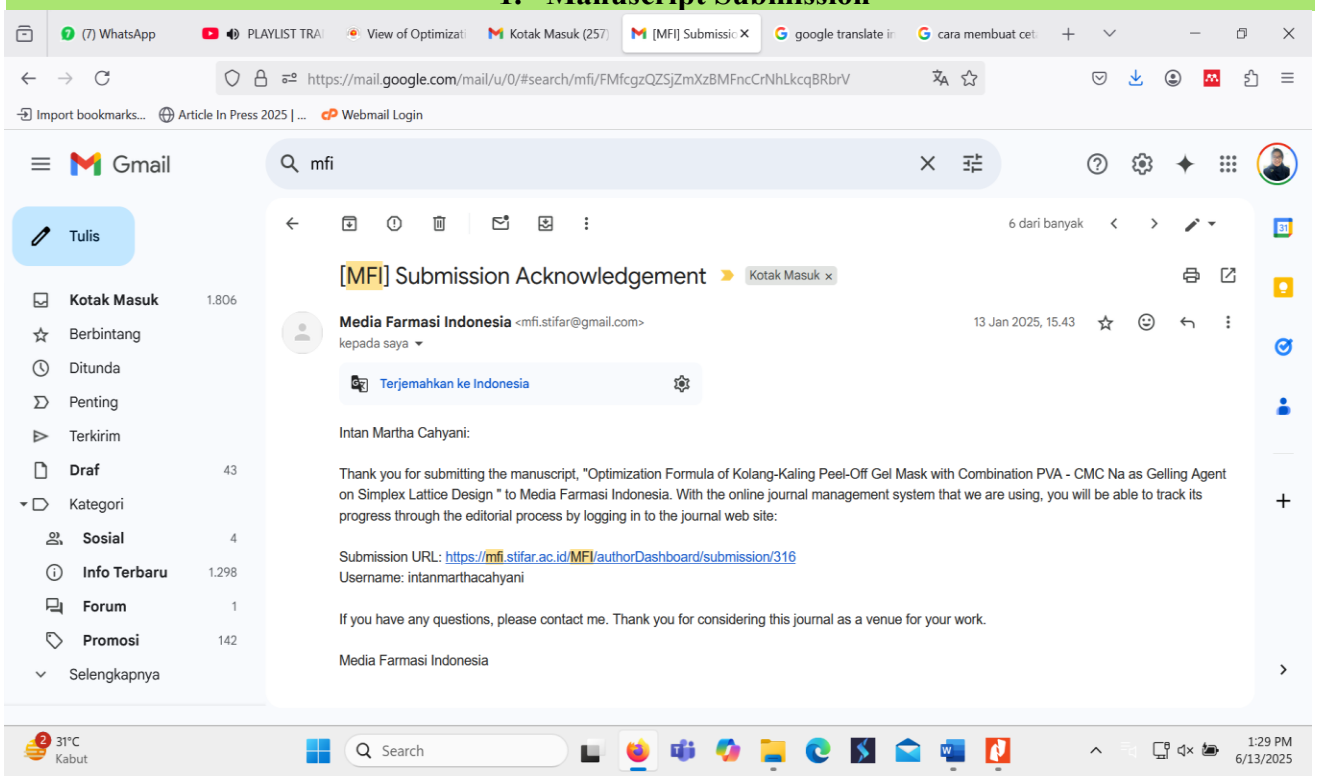
1. Manuscript Submission



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Thank you for submitting the manuscript, "Optimization Formula of Kolang-Kaling Peel-Off Gel Mask with Combination PVA - CMC Na as Gelling Agent on Simplex Lattice Design " to Media Farmasi Indonesia. With the online journal management system that we are using, you will be able to track its progress through the editorial process by logging in to the journal web site:

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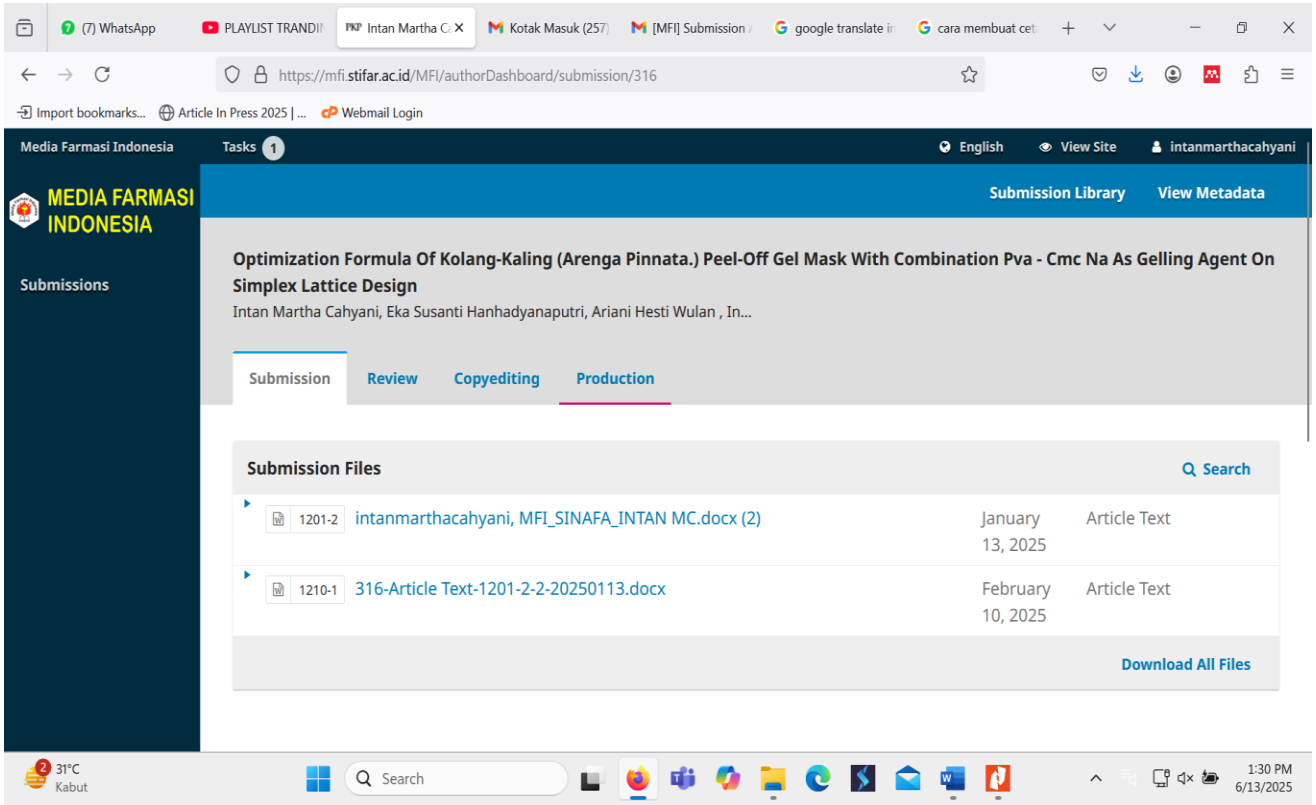

Optimization Formula Of Kolang-Kaling (Arenga Pinnata.) Peel-Off Gel Mask With Combination Pva - Cmc Na As Gelling Agent On Simplex Lattice Design
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Submission Files

1201-2	intanmarthacahyani, MFI_SINAFA_INTAN MC.docx (2)	January 13, 2025	Article Text
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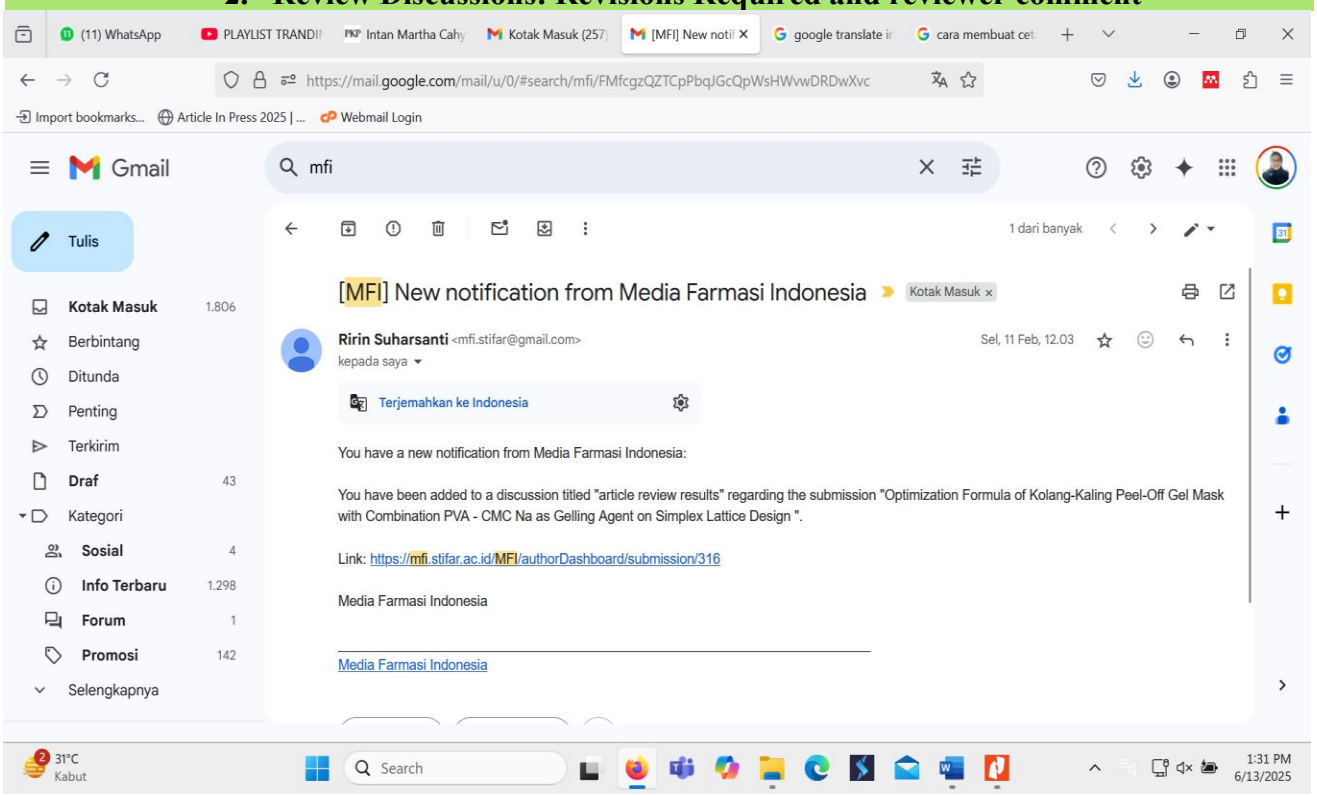

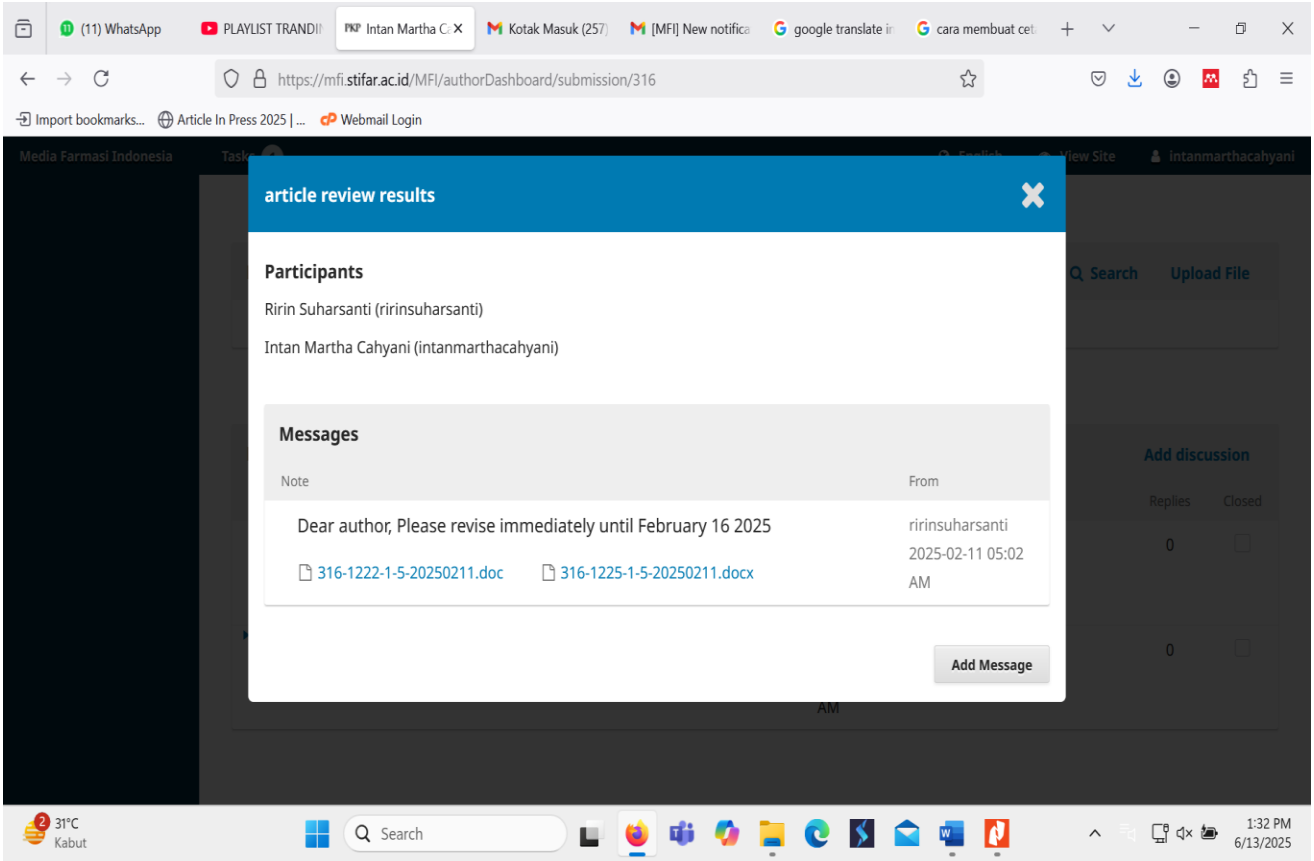

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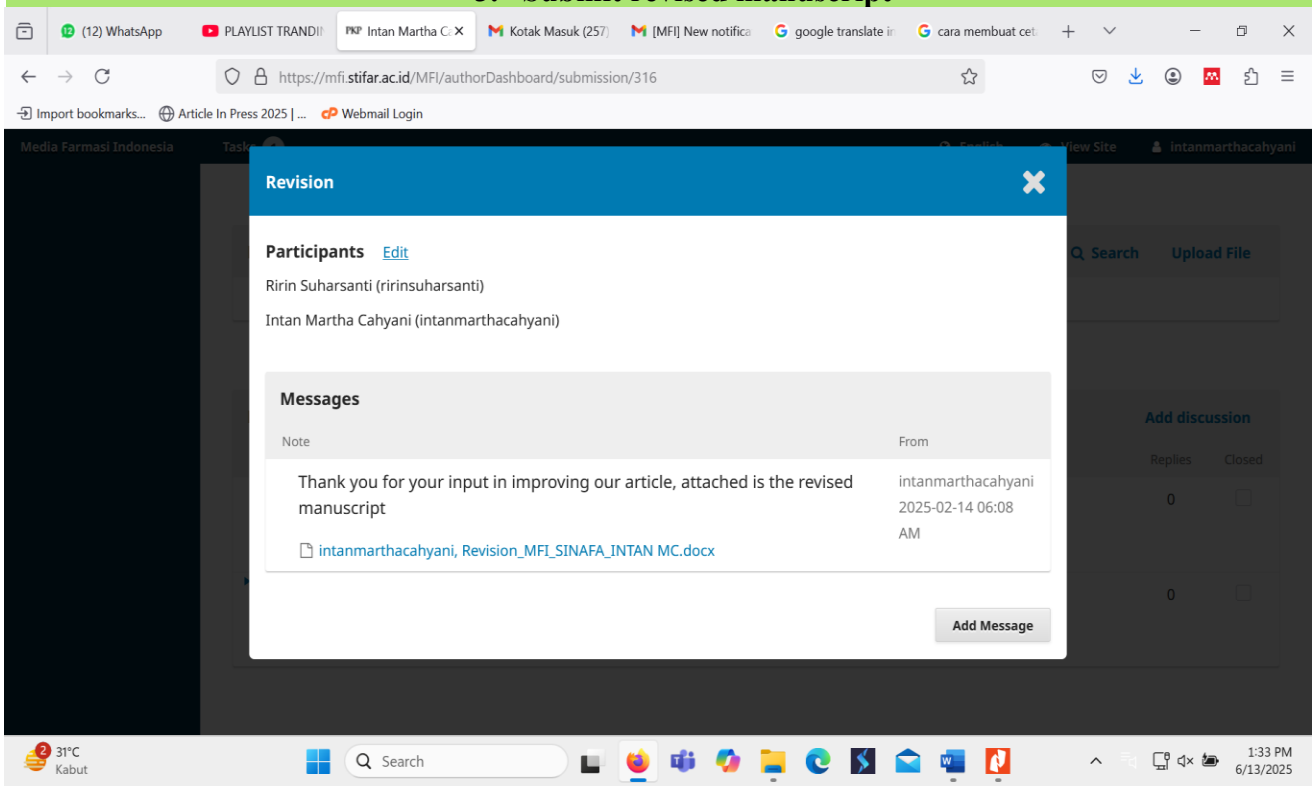

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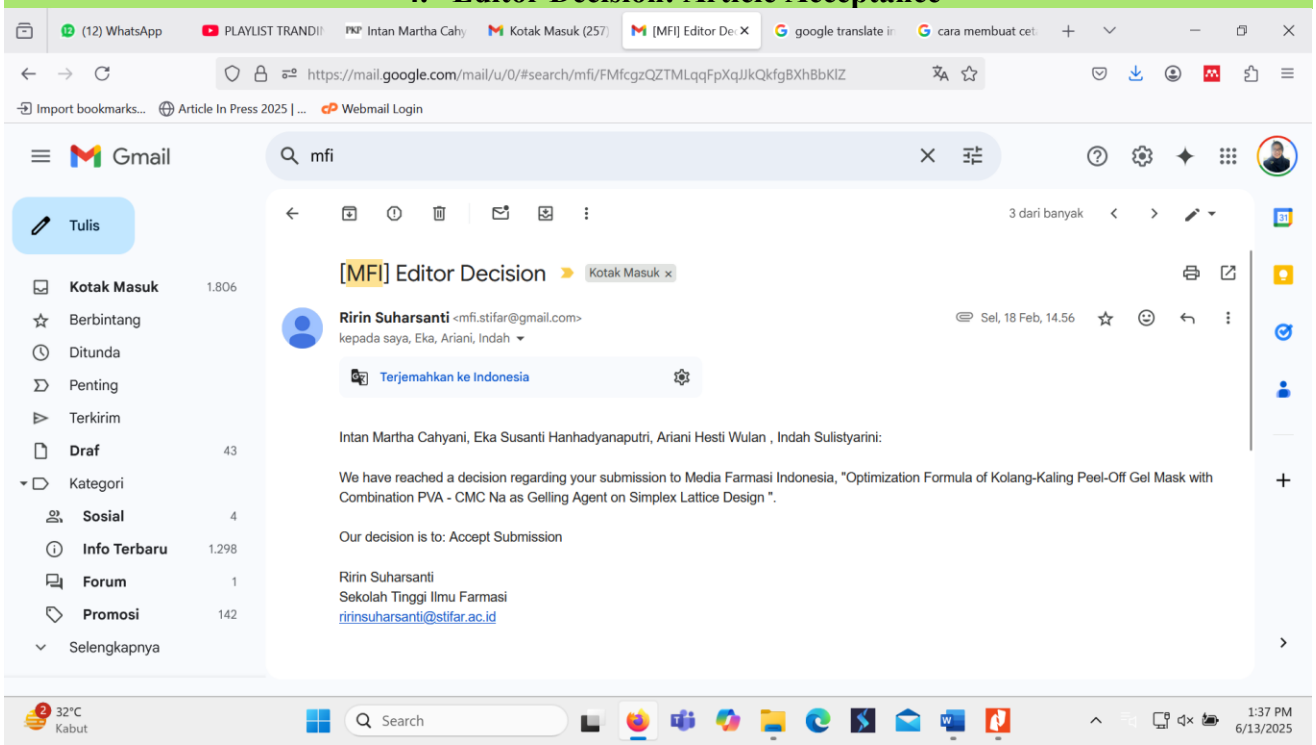

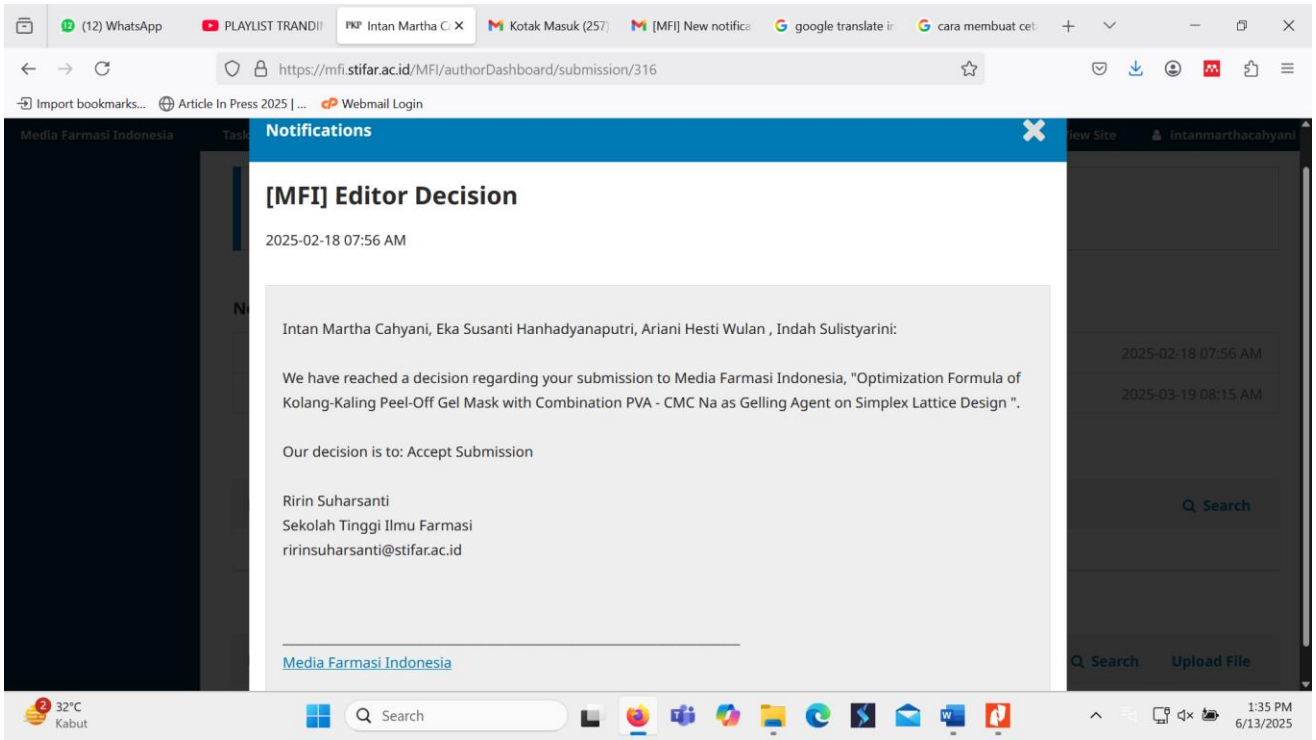

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Our decision is to: Accept Submission

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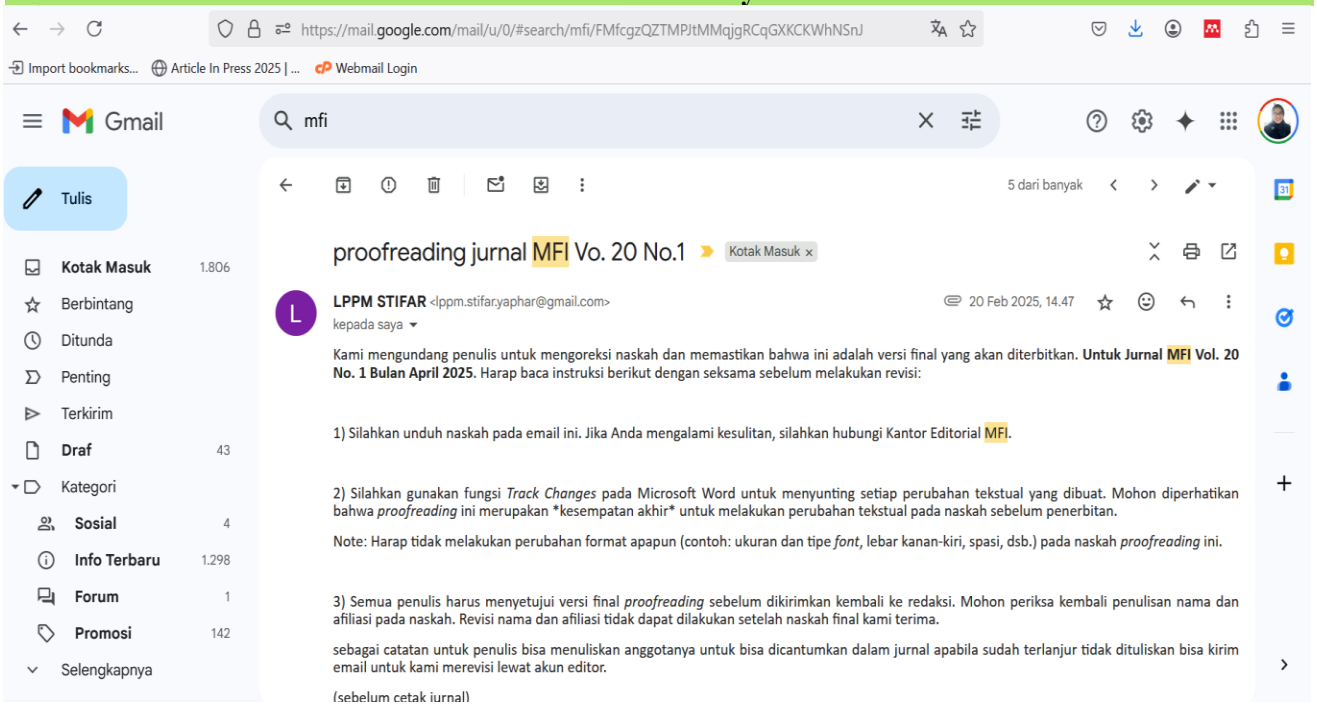

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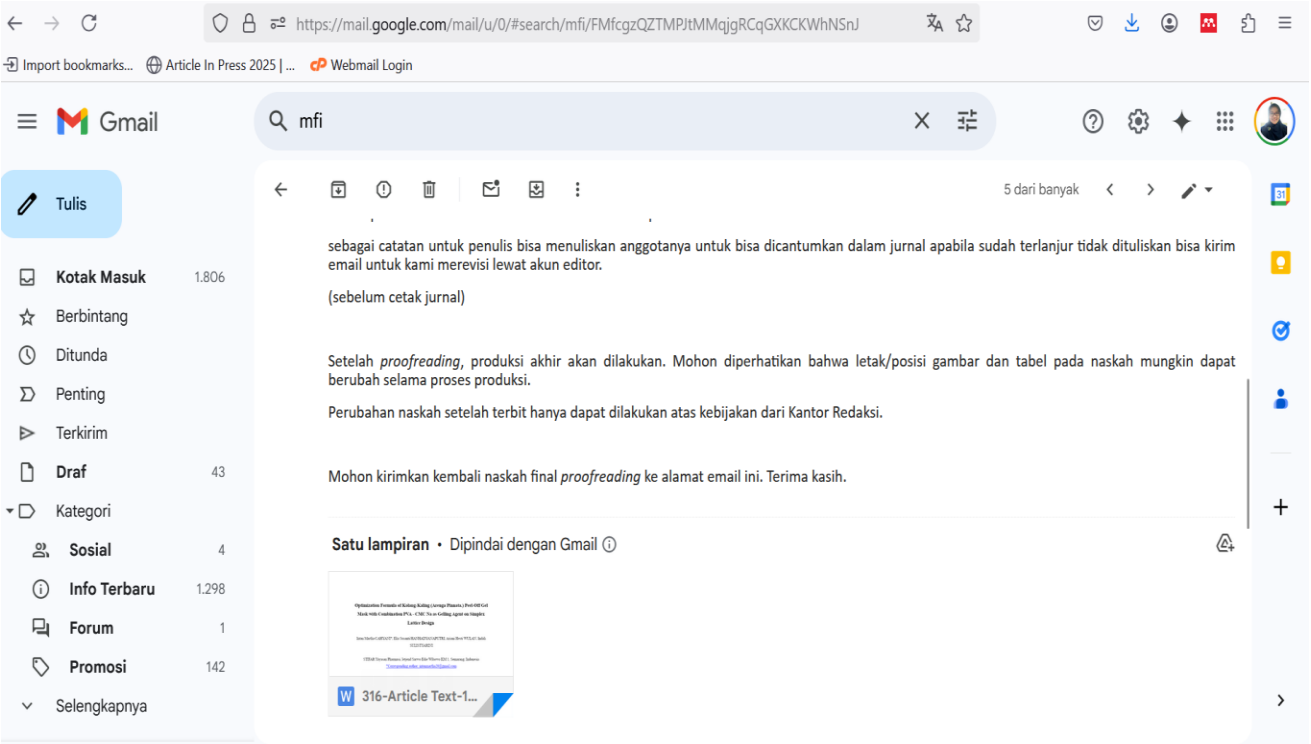

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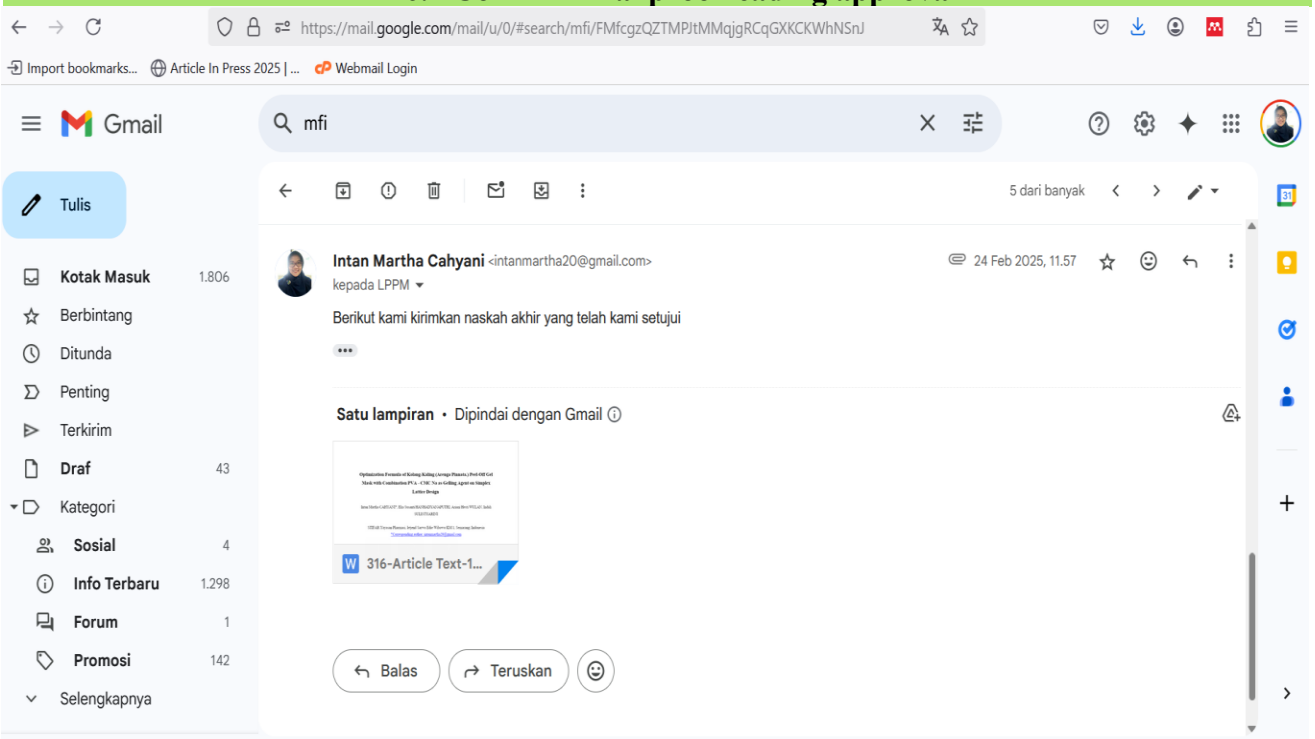

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Optimization Formula Of Kolang-Kaling (Arenga Pinnata.) Peel-Off Gel Mask With Combination Pva - Cmc Na As Gelling Agent On Simplex Lattice Design

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Optimization Formula of Kolang-Kaling Peel-Off Gel Mask with Combination PVA - CMC Na as Gelling Agent on Simplex Lattice Design

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Abstract

Kolang-kaling fruit in topical preparations increase its effectiveness as an antioxidant. The peel-off gel mask is as an effective topical preparation for skin health and user comfort. Its formulation is determined by quality parameters, particularly forming a plastic film of PVA and CMC Na as polymers. This study aims to determine the effect of these combination and the ratio of both concentrations based on the physical characteristics and IC50 value. The optimization on the simplex lattice design was obtained in 8 runs with PVA concentrations (10% -15%) and CMC Na (0.5% -5.5%). The pH value, spreadability, adhesion, drying time, and IC50 were used as parameters to determine optimal formula. The results showed that the difference in PVA and CMC Na concentrations had a significant effect on all optimization parameters (p-value <0.05) and the lack of fit for each replication was not significant (p-value> 0.05). The optimal formula obtained PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spread power of 20.34 cm; drying time of 18 minutes, and IC50 of 25.5 ppm (desirability of 0.954), which is not significantly different from the observation results indicating the optimization is valid.

Keyword: Kolang-kaling, Optimization, Peel-off gel mask, PVA, CMC Na.

INTRODUCTION

Kolang-kaling is a type of *Aren* fruit with a chewy texture and a fresh taste, it is widely used as an ingredient in drinks or traditional foods. Several studies have stated that kolang-kaling has numerous health benefits because of the content of compounds, including 9.1% calcium (bones) (Holly et al., 2018) flavonoids (joint pain) (Yolanda & Febriyanti, 2021) crude fiber 10.52% (digestion), and galactomannan, which is widely used as an emulsifier and thickener in ice cream.(Ihsan et al., 2022)

Galactomannan, is a natural hydrocolloid (Hasna, 2020) the content of kolang-kaling is 10µg/mL with antioxidant activity >50% higher than vitamin C (Yanti & Ali, 2017). In another study, it was stated that the antioxidant activity of galactomannan was included in the very strong category with IC50 (20.45 ppm) (Tarigan, 2012). The process of processing and drying

Kolang-kalinginto powder with several heating methods can affect the antioxidant activity of glucomannan. The drying method that can maintain antioxidant activity in the very strong category is a microwave at a temperature of 40°C for 22 hours with an IC50 value of 25.30 ppm (Hanhadyanaputri et al., 2023) showing the potential to be developed as a peel-off gel mask preparation to increase its effectiveness and comfort.

Peel-off gel mask is the right dosage form for use on facial skin with an elastic membrane and is easy to remove after use.(Sulastri & Dewi, 2018) In its formulation, gelling agent plays an important role for this purpose. Polyvinyl Alcohol (PVA) can function as a gelling agent because it is a hydrophilic polymer with adhesive properties that will help provide a film layer effect called the peel-off effect and can be easily peeled off after drying (Anung Anindhita et al., 2023). Not only does PVA have advantages as a gelling agent, but there are also disadvantages that must be considered in its use, namely the formation of a rigid and easily broken film layer (Sinambela & Telaumbanua, 2022). Carboxymethyl Cellulose Sodium (CMC Na) is a hydrophilic polymer with a more stable viscosity over a long period of time(Eryani et al., 2023) thus it can increase the elasticity of the PVA layer formed. This study aims to determine the effect of the combination of PVA and CMC Na and the ratio of these concentrations to obtain the optimal peel-off gel mask formula for kolang-kaling.

MATERIALS AND METHODS

a) Materials and Tools

The materials needed are kolang-kaling taken from Gunung Pati Semarang PVA, CMC Na, propilenglycol, methylparaben, 2.2-diphenyl-1-picrylhydrazyl (DPPH), and Vitamin C. In this research process, tools are needed, including a blender, microwave, pH meter, and UV-Vis spectrophotometer.

b) Methods

Preparation of Kolang-Kaling Powder

Soak the Kolang-kaling in clean water for 1 x 24 hours, wash it clean, drain, thinly sliced, and dried in a microwave at 40ºC for 22 hours, blended and sieved through mesh No. 44.

Preparation of Kolang-Kaling Peel-Off Gel Mask

PVA, CMC Na, and palm sugar powder were each developed with 80ºC aqua destillata, then mixed and homogenized. The mixture was added with methylparaben that had been dissolved in propylene glycol, and stirred until homogeneous. The proportion of PVA and CMC Na was determined according to the optimization design with the simplex lattice design method according to the formula for each run as seen in Table 1.

Table 1. Design Optimization of Kolang-Kaling Peel-Off Gel Mask

Formula	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
Kolang-Kaling Powder (%)	2	2	2	2	2	2	2	2	2	2
PVA (%)	**10**	**12.5**	**15**	**10**	**13.75**	**11.25**	**10**	**12.5**	**15**	**15**
CMC Na (%)	**5.5**	**3**	**0.5**	**5.5**	**1.75**	**4.25**	**5.5**	**3**	**0.5**	**0.5**
Propylene Glycol (%)	10	10	10	10	10	10	10	10	10	10
Methyl Paraben (%)	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Aquadest (%)	Ad 100									

Physical Characteristics Test of Kolang-Kaling Peel-Off Gel Mask

pH Test

This was done by dipping the pH meter electrode into the preparation, leaving it until showing a constant pH value (Andini et al., 2017). A good preparation has a pH that matches the skin's pH, namely in the range of 4.5-6.5 so that when the preparation is applied, it does not cause irritation or cause dry and scaly skin (Silvia et al., 2021).

Drying Time Test

A preparation of 0.1 grams was applied to an area of 2.5 x 2.5 cm on the arm and the time required for the preparation to dry is observed (Andini et al., 2017). The drying time for a good peel-off gel mask spent 15-30 minutes (Silvia et al., 2021).

Spreadability Test

Of 0.5 grams preparation was placed in the center of the spreadability tester, covered with a glass plate that had been previously weighed and its weight recorded, left for one minute, then the four sides of the spread diameter were measured and a load of 50 grams was added, and left again for one minute. The spread diameter of the preparation was then recorded as before. The addition of the load was stopped when the spread diameter of the preparation is constant (Cahyani & Putri, 2017). The spread of a good peel off gel mask is in the range of 5-7 cm.(Silvia et al., 2021)

Adhesion Test

It was carried out by placing 0.25 grams of the preparation on the object glass, given pressure with the addition of a load of 100 grams for 5 minutes and mounted on the adhesion tester. The time for the two object glasses to be released was then recorded. The adhesion test of a good peel-off gel mask was more than 1 second.(Syam et al., 2021)

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

Vitamin C was produced in a series with concentrations of 2,4,6,8,10 ppm. Gel mask samples were made in concentrations of 20,40,60,70,80 ppm. A 2.0 ml sample was added with 2.0 ml of DPPH solution, placed in a test tube that had been coated with aluminum foil. The samples were incubated at room temperature according to the OT obtained by each sample. Measure the absorbance using a UV-Vis spectrophotometer using the maximum wavelength obtained. Perform testing on all runs and determine the IC50 value of each run (Diah et al., 2022).

RESULTS AND DISCUSSION

The peel-off gel mask preparation of kolang-kaling powder obtained was then tested for physical characteristics, including pH, drying time, spreadability, adhesion test and antioxidant activity (IC50) with the results of each run can be seen in Table 2.

Table 2. Optimization Response to Characterization of Kolang-Kaling Peel-Off Gel Mask

Response	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
pH	5.98	5.70	5.63	5.81	5.69	5.72	5.90	5.68	5.51	5.49
Drying Time (minute)	19	16	17	18	18	17	19	20	18	19
Spreadability (cm)	4.25	6.48	5.45	3.50	8.48	5.02	4.45	6.56	6.78	6.98
Adhesion (second)	37	27	22	28	20	29	31	28	15	25
IC50 (ppm)	33.94	34.97	33.24	39.52	38.69	42.00	36.38	35.95	20.51	20.43

pH Test

Based on the optimization equation of the pH test parameters, it shows that PVA and CMC Na both have an effect on increasing the pH value with coefficients of +0.36 and +0.47 respectively (Table 3), but the effect of the increase was dominated by CMC Na because CMC Na has a higher pH (7.0 - 8.5) (Wijayanti et al., 2005) compared to PVA pH (5.0 – 8.0) (Arijani & Ariani, 2019). The results are in accordance with the pH test profile of the kolang-kaling

peel-off gel mask preparation shown in Figure 1a. where there is an increase in the curve that is proportional to the increase in CMC Na concentration.

Dry Time Test

Table 3 shows the optimization equation showing that the increase in drying time is significantly influenced by the increase in PVA and CMC Na concentrations (+1.18 and +2.15) with the increase in the curve being dominated by the influence of CMC Na (Figure 1b), while the combination of both has the opposite effect (-0.09). These results indicate that increasing the concentration of PVA can accelerate the drying time due to its adhesive properties (Septiani et al., 2021) so that it forms a dry layer more quickly, as report (Diah et al., 2022).

Table 3. Optimization Equation of Kolang-Kaling Peel-Off Gel Mask

Response	Equation	Model (p-value)	Lack of fit (p-value)
pH	$Y=0.36\ A + 0.47\ B - 4.97.10^{-3}\ AB$	0.0015	0.5356
Drying Time	$Y=1.18\ A + 2.15\ B - 0.09\ AB$	0.0268	0,7580
Spreadability	$Y=0.40\ A - 2.44\ B + 0.24\ AB$	0.0054	0.1025
Adhesion	$Y=1.23\ A + 3.56\ B + 4.21.10^{-3}\ AB$	0.0195	0.7032
IC50	$Y=1.35\ A - 7.53\ B + 1.18\ AB$	0.0268	0.4315

Note: ($P<0.05$ means significantly different)

Spreadability Test

The analysis results showed that PVA gave a synergistic effect (+0.40) while CMC Na was anatogonistic (-2.44) on the spreadability of the peel-off gel mask preparation of kolang-kaling powder (Figure 1c). This is influenced by the adhesive properties of PVA which can form resistance so that it provides resistance to mass spread (Diah et al., 2022).

Adhesion Test

The synergistic effect is presented in the increase in the concentration of PVA (+1.23) and CMC Na (+3.56) on the adhesion power of the peel-off gel mask preparation of kolang-kaling powder. Figure 1d shows that the effect of PVA is lower than CMC Na because CMC Na is a hydrophilic polymer, which can expand with the presence of water so that the binding power between particles and viscosity increases (Kusuma et al., 2018). Meanwhile, PVA in high concentrations forms a thinner liquid so that the resulting synergistic effect is lower (Wibowo et al., 2022).

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

The results of the antioxidant activity test analysis showed that PVA gave a synergistic effect (+1.35) while CMC Na had an antagonistic effect (-7.53) on the IC50 value of the peel-off gel mask preparation of kolang-kaling powder as seen in the equation Table 3 and reinforced by Figure 1e. Increasing the concentration of CMC Na in the preparation can increase the dispersion of galactomanan the form of molecular dispersion. It can bind to DPPH free radicals so that increasing its antioxidant activity as indicated by a decrease in the IC50 value (Zahara et al., 2020).

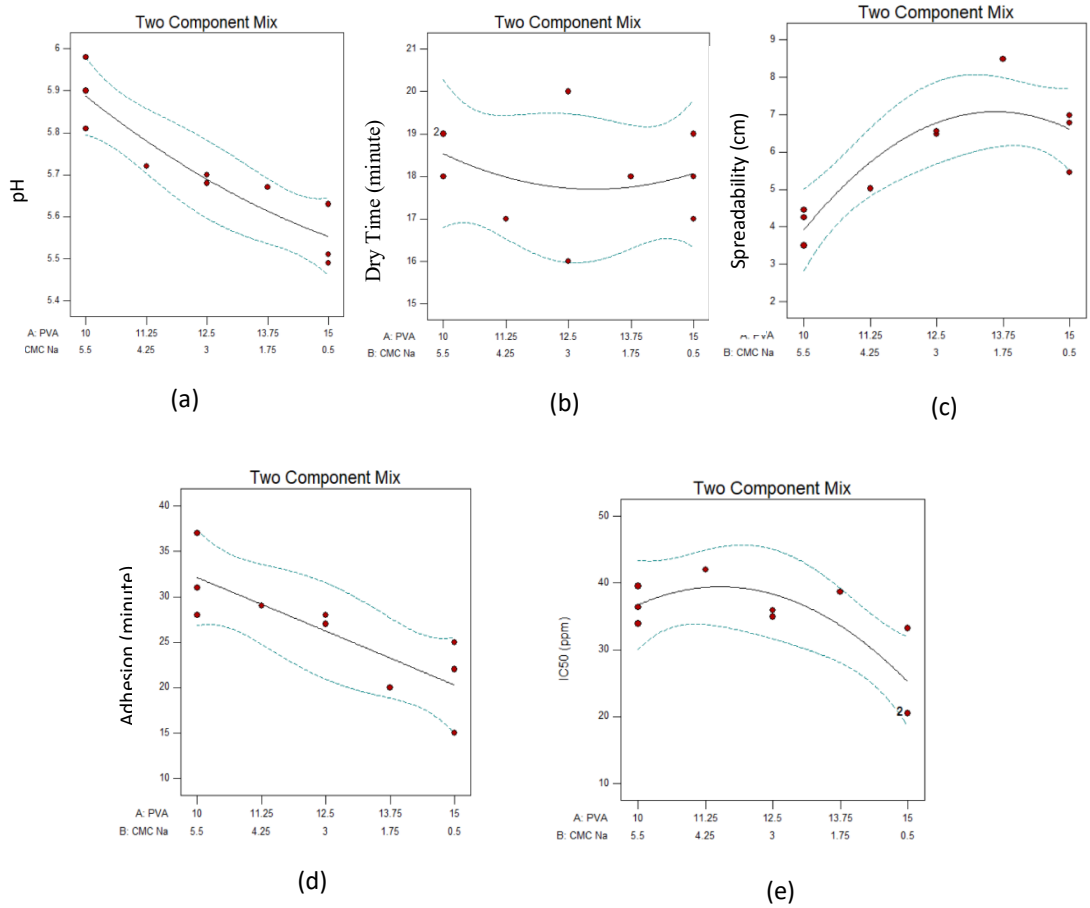

Figure 1. Contour Plot Effect Concentration of PVA and CMC Na on (a) pH; (b) Dry Time; (c) Spreadability; (d) Adhesion; and (e) IC50

Optimization

The optimum formula of peel-off gel mask preparation of kolang-kaling powder was obtained based on the target regression model of all response, namely pH, adhesion, spreading, drying time and IC50.

Table 4. Comparative Analysis of Prediction and Observed Data

Response	Prediction	Observation ±SD	p-value

pH	5.55	5.60 ± 0.03	0.06
Drying Time	18.06	18 ± 0.00	0.09
Spreadability	6.63	6.54 ± 0.03	0.09
Adhesion	20.34	20.28 ± 0.07	0.20
IC50	25.5	25.55 ± 0.73	0.91

Note: ($P<0.05$ means significantly different)

The selected formula is expected to produce peel-off gel mask preparation of kolang-kaling powder with optimal results for each parameter by determining the priority scale at a weight of 0.1-1. Furthermore, the optimization analysis will produce a desire function in which this study each parameter response is expected as an objective function and the desire function is developed to obtain optimum conditions (Mesa et al., 2017). The results of the model analysis show that the optimum formula is obtained at PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spreadability of 20.34 cm; drying time of 18 minutes and IC50 of 25.5 ppm. The formula has a desirability value of 0.954. The high desirability value indicates that the system will produce predicted values that are close to ideal (Amdoun et al., 2018) To ensure the predicted value, a one-sample t-test analysis was performed with the observation results. The data results show that the observation results are not significantly different from the predicted values ($P>0.05$) for all optimization parameters as shown in Table 4.

CONCLUSIONS

The optimal formula was achieved with a PVA concentration of 14.97% and CMC Na of 0.53%, resulting a predicted pH 5.55, adhesion 6.63 seconds, spreadability 20.34 cm, drying time 18 minutes, and IC50 25.5 ppm (desirability of 0.954). These values showed no significant differences from the observation results, indicating the validity of the optimization equations used for each parameter.

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BUKTI KORESPONDENSI CATATAN REVIEWER 1

Optimization Formula of Kolang-Kaling Peel-Off Gel Mask with Combination PVA - CMC Na as Gelling Agent on Simplex Lattice Design

Abstract

Kolang-kaling fruit in topical preparations increase its effectiveness as an antioxidant. The peel-off gel mask is as an effective topical preparation for skin health and user comfort. Its formulation is determined by quality parameters, particularly forming a plastic film of PVA and CMC Na as polymers. This study aims to determine the effect of these combination and the ratio of both concentrations based on the physical characteristics and IC50 value. The optimization on the simplex lattice design was obtained in 8 runs with PVA concentrations (10% -15%) and CMC Na (0.5% -5.5%). The pH value, spreadability, adhesion, drying time, and IC50 were used as parameters to determine optimal formula. The results showed that the difference in PVA and CMC Na concentrations had a significant effect on all optimization parameters (p-value <0.05) and the lack of fit for each replication was not significant (p-value> 0.05). The optimal formula obtained PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spread power of 20.34 cm; drying time of 18 minutes, and IC50 of 25.5 ppm (desirability of 0.954), which is not significantly different from the observation results indicating the optimization is valid.

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Keyword: Kolang-kaling, Optimization, Peel-off gel mask, PVA, CMC Na.

INTRODUCTION

Kolang-kaling is a type of *Aren* fruit with a chewy texture and a fresh taste, it is widely used as an ingredient in drinks or traditional foods. Several studies have stated that kolang-kaling has numerous health benefits because of the content of compounds, including 9.1% calcium (bones) (Holly et al., 2018) flavonoids (joint pain) (Yolanda & Febriyanti, 2021) crude fiber 10.52% (digestion), and galactomannan, which is widely used as an emulsifier and thickener in ice cream.(Ihsan et al., 2022)

Galactomannan, is a natural hydrocolloid (Hasna, 2020) the content of kolang-kaling is 10µg/mL with antioxidant activity >50% higher than vitamin C (Yanti & Ali, 2017). In another study, it was stated that the antioxidant activity of galactomannan was included in the very strong category with IC50 (20.45 ppm) (Tarigan, 2012). The process of processing and drying Kolang-kalinginto powder with several heating methods can affect the antioxidant activity of glucomannan. The drying method that can maintain antioxidant activity in the very strong category is a microwave at a temperature of 40°C for 22 hours with an IC50 value of 25.30 ppm (Hanhadyanaputri et al., 2023) showing the potential to be developed as a peel-off gel mask preparation to increase its effectiveness and comfort.

BUKTI KORESPONDENSI CATATAN REVIEWER 1

Peel-off gel mask is the right dosage form for use on facial skin with an elastic membrane and is easy to remove after use.(Sulastri & Dewi, 2018) In its formulation, gelling agent plays an important role for this purpose. Polyvinyl Alcohol (PVA) can function as a gelling agent because it is a hydrophilic polymer with adhesive properties that will help provide a film layer effect called the peel-off effect and can be easily peeled off after drying (Anung Anindhita et al., 2023). Not only does PVA have advantages as a gelling agent, but there are also disadvantages that must be considered in its use, namely the formation of a rigid and easily broken film layer (Sinambela & Telaumbanua, 2022). Carboxymethyl Cellulose Sodium (CMC Na) is a hydrophilic polymer with a more stable viscosity over a long period of time(Eryani et al., 2023) thus it can increase the elasticity of the PVA layer formed. This study aims to determine the effect of the combination of PVA and CMC Na and the ratio of these concentrations to obtain the optimal peel-off gel mask formula for kolang-kaling.

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MATERIALS AND METHODS

a) Materials and Tools

The materials needed are kolang-kaling taken from Gunung Pati Semarang PVA, CMC Na, propilenglycol, methylparaben, 2.2-diphenyl-1-picrylhydrazyl (DPPH), and Vitamin C. In this research process, tools are needed, including a blender, microwave, pH meter, and UV-Vis spectrophotometer.

b) Methods

Preparation of Kolang-Kaling Powder

Soak the Kolang-kaling in clean water for 1 x 24 hours, wash it clean, drain, thinly sliced, and dried in a microwave at 40ºC for 22 hours, blended and sieved through mesh No. 44.

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Preparation of Kolang-Kaling Peel-Off Gel Mask

PVA, CMC Na, and palm sugar powder were each developed with 80ºC aqua destillata, then mixed and homogenized. The mixture was added with methylparaben that had been dissolved in propylene glycol, and stirred until homogeneous. The proportion of PVA and CMC Na was determined according to the optimization design with the simplex lattice design method according to the formula for each run as seen in Table 1.

Table 1. Design Optimization of Kolang-Kaling Peel-Off Gel Mask

Formula	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
Kolang-Kaling Powder (%)	2	2	2	2	2	2	2	2	2	2

BUKTI KORESPONDENSI CATATAN REVIEWER 1

PVA (%)	**10**	**12.5**	**15**	**10**	**13.75**	**11.25**	**10**	**12.5**	**15**	**15**
CMC Na (%)	**5.5**	**3**	**0.5**	**5.5**	**1.75**	**4.25**	**5.5**	**3**	**0.5**	**0.5**
Propylene Glycol (%)	10	10	10	10	10	10	10	10	10	10
Methyl Paraben (%)	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Aquadest (%)	Ad 100									

Physical Characteristics Test of Kolang-Kaling Peel-Off Gel Mask

pH Test

This was done by dipping the pH meter electrode into the preparation, leaving it until showing a constant pH value (Andini et al., 2017). A good preparation has a pH that matches the skin's pH, namely in the range of 4.5-6.5 so that when the preparation is applied, it does not cause irritation or cause dry and scaly skin (Silvia et al., 2021).

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Drying Time Test

A preparation of 0.1 grams was applied to an area of 2.5 x 2.5 cm on the arm and the time required for the preparation to dry is observed (Andini et al., 2017). The drying time for a good peel-off gel mask spent 15-30 minutes (Silvia et al., 2021).

Spreadability Test

Of 0.5 grams preparation was placed in the center of the spreadability tester, covered with a glass plate that had been previously weighed and its weight recorded, left for one minute, then the four sides of the spread diameter were measured and a load of 50 grams was added, and left again for one minute. The spread diameter of the preparation was then recorded as before. The addition of the load was stopped when the spread diameter of the preparation is constant (Cahyani & Putri, 2017). The spread of a good peel off gel mask is in the range of 5-7 cm.(Silvia et al., 2021)

Adhesion Test

It was carried out by placing 0.25 grams of the preparation on the object glass, given pressure with the addition of a load of 100 grams for 5 minutes and mounted on the adhesion tester. The time for the two object glasses to be released was then recorded. The adhesion test of a good peel-off gel mask was more than 1 second.(Syam et al., 2021)

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

Vitamin C was produced in a series with concentrations of 2,4,6,8,10 ppm. Gel mask samples were made in concentrations of 20,40,60,70,80 ppm. A 2.0 ml sample was added with 2.0 ml of DPPH solution, placed in a test tube that had been coated with aluminum foil. The

samples were incubated at room temperature according to the OT obtained by each sample. Measure the absorbance using a UV-Vis spectrophotometer using the maximum wavelength obtained. Perform testing on all runs and determine the IC50 value of each run (Diah et al., 2022).

RESULTS AND DISCUSSION

The peel-off gel mask preparation of kolang-kaling powder obtained was then tested for physical characteristics, including pH, drying time, spreadability, adhesion test and antioxidant activity (IC50) with the results of each run can be seen in Table 2.

Table 2. Optimization Response to Characterization of Kolang-Kaling Peel-Off Gel Mask

Response	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
pH	5.98	5.70	5.63	5.81	5.69	5.72	5.90	5.68	5.51	5.49
Drying Time (minute)	19	16	17	18	18	17	19	20	18	19
Spreadability (cm)	4.25	6.48	5.45	3.50	8.48	5.02	4.45	6.56	6.78	6.98
Adhesion (second)	37	27	22	28	20	29	31	28	15	25
IC50 (ppm)	33.94	34.97	33.24	39.52	38.69	42.00	36.38	35.95	20.51	20.43

pH Test

Based on the optimization equation of the pH test parameters, it shows that PVA and CMC Na both have an effect on increasing the pH value with coefficients of +0.36 and +0.47 respectively (Table 3), but the effect of the increase was dominated by CMC Na because CMC Na has a higher pH (7.0 - 8.5) (Wijayanti et al., 2005) compared to PVA pH (5.0 – 8.0) (Arijani & Ariani, 2019). The results are in accordance with the pH test profile of the kolang-kaling peel-off gel mask preparation shown in Figure 1a. where there is an increase in the curve that is proportional to the increase in CMC Na concentration.

Dry Time Test

Table 3 shows the optimization equation showing that the increase in drying time is significantly influenced by the increase in PVA and CMC Na concentrations (+1.18 and +2.15)

with the increase in the curve being dominated by the influence of CMC Na (Figure 1b), while the combination of both has the opposite effect (-0.09). These results indicate that increasing the concentration of PVA can accelerate the drying time due to its adhesive properties (Septiani et al., 2021) so that it forms a dry layer more quickly, as report (Diah et al., 2022).

Table 3. Optimization Equation of Kolang-Kaling Peel-Off Gel Mask

Response	Equation	Model (p-value)	Lack of fit (p-value)
pH	$Y=0.36\ A + 0.47\ B - 4.97.10^{-3}\ AB$	0.0015	0.5356
Drying Time	$Y=1.18\ A + 2.15\ B - 0.09\ AB$	0.0268	0,7580
Spreadability	$Y=0.40\ A - 2.44\ B + 0.24\ AB$	0.0054	0.1025
Adhesion	$Y=1.23\ A + 3.56\ B + 4.21.10^{-3}\ AB$	0.0195	0.7032
IC50	$Y=1.35\ A - 7.53\ B + 1.18\ AB$	0.0268	0.4315

Note: ($P<0.05$ means significantly different)

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Spreadability Test

The analysis results showed that PVA gave a synergistic effect (+0.40) while CMC Na was anatogonistic (-2.44) on the spreadability of the peel-off gel mask preparation of kolang-kaling powder (Figure 1c). This is influenced by the adhesive properties of PVA which can form resistance so that it provides resistance to mass spread (Diah et al., 2022).

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Adhesion Test

The synergistic effect is presented in the increase in the concentration of PVA (+1.23) and CMC Na (+3.56) on the adhesion power of the peel-off gel mask preparation of kolang-kaling powder. Figure 1d shows that the effect of PVA is lower than CMC Na because CMC Na is a hydrophilic polymer, which can expand with the presence of water so that the binding power between particles and viscosity increases (Kusuma et al., 2018). Meanwhile, PVA in high concentrations forms a thinner liquid so that the resulting synergistic effect is lower (Wibowo et al., 2022).

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

The results of the antioxidant activity test analysis showed that PVA gave a synergistic effect (+1.35) while CMC Na had an antagonistic effect (-7.53) on the IC50 value of the peel-off gel mask preparation of kolang-kaling powder as seen in the equation Table 3 and reinforced by Figure 1e. Increasing the concentration of CMC Na in the preparation can increase the dispersion of galactomanan the form of molecular dispersion. It can bind to DPPH free radicals so that increasing its antioxidant activity as indicated by a decrease in the IC50 value (Zahara et al., 2020).

BUKTI KORESPONDENSI CATATAN REVIEWER 1

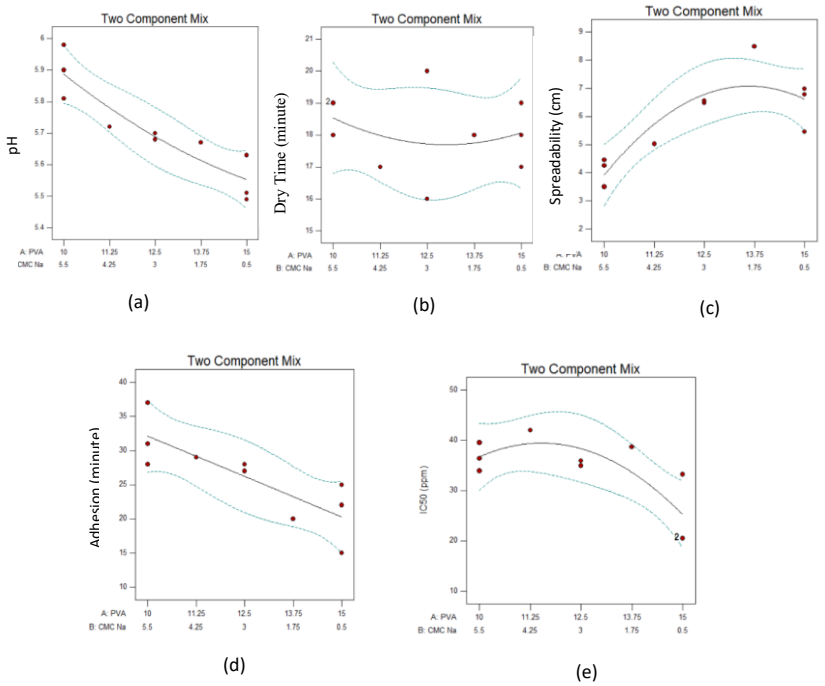

Figure 1. Contour Plot Effect Concentration of PVA and CMC Na on (a) pH; (b) Dry Time; (c) Spreadability; (d) Adhesion; and (e) IC50

Optimization

The optimum formula of peel-off gel mask preparation of kolang-kaling powder was obtained based on the target regression model of all response, namely pH, adhesion, spreading, drying time and IC50.

Table 4. Comparative Analysis of Prediction and Observed Data

Response	Prediction	Observation ±SD	p-value
pH	5.55	5.60 ± 0.03	0.06
Drying Time	18.06	18 ± 0.00	0.09
Spreadability	6.63	6.54 ± 0.03	0.09
Adhesion	20.34	20.28 ± 0.07	0.20
IC50	25.5	25.55 ± 0.73	0.91

Note: ($P<0.05$ means significantly different)

BUKTI KORESPONDENSI CATATAN REVIEWER 1

The selected formula is expected to produce peel-off gel mask preparation of kolang-kaling powder with optimal results for each parameter by determining the priority scale at a weight of 0.1-1. Furthermore, the optimization analysis will produce a desire function in which this study each parameter response is expected as an objective function and the desire function is developed to obtain optimum conditions (Mesa et al., 2017). The results of the model analysis show that the optimum formula is obtained at PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spreadability of 20.34 cm; drying time of 18 minutes and IC50 of 25.5 ppm. The formula has a desirability value of 0.954. The high desirability value indicates that the system will produce predicted values that are close to ideal (Amdoun et al., 2018) To ensure the predicted value, a one-sample t-test analysis was performed with the observation results. The data results show that the observation results are not significantly different from the predicted values (P>0.05) for all optimization parameters as shown in Table 4.

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CONCLUSIONS

The optimal formula was achieved with a PVA concentration of 14.97% and CMC Na of 0.53%, resulting a predicted pH 5.55, adhesion 6.63 seconds, spreadability 20.34 cm, drying time 18 minutes, and IC50 25.5 ppm (desirability of 0.954). These values showed no significant differences from the observation results, indicating the validity of the optimization equations used for each parameter.

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BUKTI KORESPONDENSI CATATAN REVIEWER 1

BUKTI KORESPONDENSI CATATAN REVIEWER 2

Optimization Formula of Kolang-Kaling Peel-Off Gel Mask with Combination PVA - CMC Na as Gelling Agent on Simplex Lattice Design

Abstract

Kolang-kaling fruit in topical preparations increase its effectiveness as an antioxidant. The peel-off gel mask is as an effective topical preparation for skin health and user comfort. Its formulation is determined by quality parameters, particularly forming a plastic film of PVA and CMC Na as polymers. This study aims to determine the effect of these combination and the ratio of both concentrations based on the physical characteristics and IC50 value. The optimization on the simplex lattice design was obtained in 8 runs with PVA concentrations (10% -15%) and CMC Na (0.5% -5.5%). The pH value, spreadability, adhesion, drying time, and IC50 were used as parameters to determine optimal formula. The results showed that the difference in PVA and CMC Na concentrations had a significant effect on all optimization parameters (p-value <0.05) and the lack of fit for each replication was not significant (p-value> 0.05). The optimal formula obtained PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spread power of 20.34 cm; drying time of 18 minutes, and IC50 of 25.5 ppm (desirability of 0.954), which is not significantly different from the observation results indicating the optimization is valid.

Keyword: Kolang-kaling, Optimization, Peel-off gel mask, PVA, CMC Na.

INTRODUCTION

Kolang-kaling is a type of *Aren* fruit with a chewy texture and a fresh taste, it is widely used as an ingredient in drinks or traditional foods. Several studies have stated that kolang-kaling has numerous health benefits because of the content of compounds, including 9.1% calcium (bones) (Holly et al., 2018) flavonoids (joint pain) (Yolanda & Febriyanti, 2021) crude fiber 10.52% (digestion), and galactomannan, which is widely used as an emulsifier and thickener in ice cream.(Ihsan et al., 2022)

Galactomannan, is a natural hydrocolloid (Hasna, 2020) the content of kolang-kaling is 10µg/mL with antioxidant activity >50% higher than vitamin C (Yanti & Ali, 2017). In another study, it was stated that the antioxidant activity of galactomannan was included in the very strong category with IC50 (20.45 ppm) (Tarigan, 2012). The process of processing and drying Kolang-kalinginto powder with several heating methods can affect the antioxidant activity of glucomannan. The drying method that can maintain antioxidant activity in the very strong category is a microwave at a temperature of 40°C for 22 hours with an IC50 value of 25.30 ppm (Hanhadyanaputri et al., 2023) showing the potential to be developed as a peel-off gel mask preparation to increase its effectiveness and comfort.

BUKTI KORESPONDENSI CATATAN REVIEWER 2

Peel-off gel mask is the right dosage form for use on facial skin with an elastic membrane and is easy to remove after use.(Sulastri & Dewi, 2018) In its formulation, gelling agent plays an important role for this purpose. Polyvinyl Alcohol (PVA) can function as a gelling agent because it is a hydrophilic polymer with adhesive properties that will help provide a film layer effect called the peel-off effect and can be easily peeled off after drying (Anung Anindhita et al., 2023). Not only does PVA have advantages as a gelling agent, but there are also disadvantages that must be considered in its use, namely the formation of a rigid and easily broken film layer (Sinambela & Telaumbanua, 2022). Carboxymethyl Cellulose Sodium (CMC Na) is a hydrophilic polymer with a more stable viscosity over a long period of time(Eryani et al., 2023) thus it can increase the elasticity of the PVA layer formed. This study aims to determine the effect of the combination of PVA and CMC Na and the ratio of these concentrations to obtain the optimal peel-off gel mask formula for kolang-kaling.

MATERIALS AND METHODS

a) Materials and Tools

The materials needed are kolang-kaling taken from Gunung Pati Semarang PVA, CMC Na, propilenglycol, methylparaben, 2.2-diphenyl-1-picrylhydrazyl (DPPH), and Vitamin C. In this research process, tools are needed, including a blender, microwave, pH meter, and UV-Vis spectrophotometer.

b) Methods

Preparation of Kolang-Kaling Powder

Soak the Kolang-kaling in clean water for 1 x 24 hours, wash it clean, drain, thinly sliced, and dried in a microwave at 40ºC for 22 hours, blended and sieved through mesh No. 44.

Preparation of Kolang-Kaling Peel-Off Gel Mask

PVA, CMC Na, and palm sugar powder were each developed with 80ºC aqua destillata, then mixed and homogenized. The mixture was added with methylparaben that had been dissolved in propylene glycol, and stirred until homogeneous. The proportion of PVA and CMC Na was determined according to the optimization design with the simplex lattice design method according to the formula for each run as seen in Table 1.

Table 1. Design Optimization of Kolang-Kaling Peel-Off Gel Mask

Formula	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
Kolang-Kaling Powder (%)	2	2	2	2	2	2	2	2	2	2

BUKTI KORESPONDENSI CATATAN REVIEWER 2

PVA (%)	**10**	**12.5**	**15**	**10**	**13.75**	**11.25**	**10**	**12.5**	**15**	**15**
CMC Na (%)	**5.5**	**3**	**0.5**	**5.5**	**1.75**	**4.25**	**5.5**	**3**	**0.5**	**0.5**
Propylene Glycol (%)	10	10	10	10	10	10	10	10	10	10
Methyl Paraben (%)	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Aquadest (%)	Ad 100									

Physical Characteristics Test of Kolang-Kaling Peel-Off Gel Mask

pH Test

This was done by dipping the pH meter electrode into the preparation, leaving it until showing a constant pH value (Andini et al., 2017). A good preparation has a pH that matches the skin's pH, namely in the range of 4.5-6.5 so that when the preparation is applied, it does not cause irritation or cause dry and scaly skin (Silvia et al., 2021).

Drying Time Test

A preparation of 0.1 grams was applied to an area of 2.5 x 2.5 cm on the arm and the time required for the preparation to dry is observed (Andini et al., 2017). The drying time for a good peel-off gel mask spent 15-30 minutes (Silvia et al., 2021).

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Spreadability Test

Of 0.5 grams preparation was placed in the center of the spreadability tester, covered with a glass plate that had been previously weighed and its weight recorded, left for one minute, then the four sides of the spread diameter were measured and a load of 50 grams was added, and left again for one minute. The spread diameter of the preparation was then recorded as before. The addition of the load was stopped when the spread diameter of the preparation is constant (Cahyani & Putri, 2017). The spread of a good peel off gel mask is in the range of 5-7 cm.(Silvia et al., 2021)

Adhesion Test

It was carried out by placing 0.25 grams of the preparation on the object glass, given pressure with the addition of a load of 100 grams for 5 minutes and mounted on the adhesion tester. The time for the two object glasses to be released was then recorded. The adhesion test of a good peel-off gel mask was more than 1 second.(Syam et al., 2021)

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

Vitamin C was produced in a series with concentrations of 2,4,6,8,10 ppm. Gel mask samples were made in concentrations of 20,40,60,70,80 ppm. A 2.0 ml sample was added with 2.0 ml of DPPH solution, placed in a test tube that had been coated with aluminum foil.

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The samples were incubated at room temperature according to the OT obtained by each sample. Measure the absorbance using a UV-Vis spectrophotometer using the maximum wavelength obtained. Perform testing on all runs and determine the IC50 value of each run (Diah et al., 2022).

Commented [2]: write the OT clearly

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Commented [4]: write down the formula for calculating % inhibition

RESULTS AND DISCUSSION

The peel-off gel mask preparation of kolang-kaling powder obtained was then tested for physical characteristics, including pH, drying time, spreadability, adhesion test and antioxidant activity (IC50) with the results of each run can be seen in Table 2.

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Table 2. Optimization Response to Characterization of Kolang-Kaling Peel-Off Gel Mask

Response	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
pH	5.98	5.70	5.63	5.81	5.69	5.72	5.90	5.68	5.51	5.49
Drying Time (minute)	19	16	17	18	18	17	19	20	18	19
Spreadability (cm)	4.25	6.48	5.45	3.50	8.48	5.02	4.45	6.56	6.78	6.98
Adhesion (second)	37	27	22	28	20	29	31	28	15	25
IC50 (ppm)	33.94	34.97	33.24	39.52	38.69	42.00	36.38	35.95	20.51	20.43

pH Test

Based on the optimization equation of the pH test parameters, it shows that PVA and CMC Na both have an effect on increasing the pH value with coefficients of +0.36 and +0.47 respectively (Table 3), but the effect of the increase was dominated by CMC Na because CMC Na has a higher pH (7.0 - 8.5) (Wijayanti et al., 2005) compared to PVA pH (5.0 – 8.0) (Arijani & Ariani, 2019). The results are in accordance with the pH test profile of the kolang-kaling peel-off gel mask preparation shown in Figure 1a. where there is an increase in the curve that is proportional to the increase in CMC Na concentration.

Dry Time Test

Table 3 shows the optimization equation showing that the increase in drying time is significantly influenced by the increase in PVA and CMC Na concentrations (+1.18 and +2.15)

with the increase in the curve being dominated by the influence of CMC Na (Figure 1b), while the combination of both has the opposite effect (-0.09). These results indicate that increasing the concentration of PVA can accelerate the drying time due to its adhesive properties (Septiani et al., 2021) so that it forms a dry layer more quickly, as report (Diah et al., 2022).

Table 3. Optimization Equation of Kolang-Kaling Peel-Off Gel Mask

Response	Equation	Model (p-value)	Lack of fit (p-value)
pH	$Y=0.36\ A + 0.47\ B - 4.97.10^{-3}\ AB$	0.0015	0.5356
Drying Time	$Y=1.18\ A + 2.15\ B - 0.09\ AB$	0.0268	0,7580
Spreadability	$Y=0.40\ A - 2.44\ B + 0.24\ AB$	0.0054	0.1025
Adhesion	$Y=1.23\ A + 3.56\ B + 4.21.10^{-3}\ AB$	0.0195	0.7032
IC50	$Y=1.35\ A - 7.53\ B + 1.18\ AB$	0.0268	0.4315

Note: ($P<0.05$ means significantly different)

Spreadability Test

The analysis results showed that PVA gave a synergistic effect (+0.40) while CMC Na was anatogonistic (-2.44) on the spreadability of the peel-off gel mask preparation of kolang-kaling powder (Figure 1c). This is influenced by the adhesive properties of PVA which can form resistance so that it provides resistance to mass spread (Diah et al., 2022).

Adhesion Test

The synergistic effect is presented in the increase in the concentration of PVA (+1.23) and CMC Na (+3.56) on the adhesion power of the peel-off gel mask preparation of kolang-kaling powder. Figure 1d shows that the effect of PVA is lower than CMC Na because CMC Na is a hydrophilic polymer, which can expand with the presence of water so that the binding power between particles and viscosity increases (Kusuma et al., 2018). Meanwhile, PVA in high concentrations forms a thinner liquid so that the resulting synergistic effect is lower (Wibowo et al., 2022).

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

The results of the antioxidant activity test analysis showed that PVA gave a synergistic effect (+1.35) while CMC Na had an antagonistic effect (-7.53) on the IC50 value of the peel-off gel mask preparation of kolang-kaling powder as seen in the equation Table 3 and reinforced by Figure 1e. Increasing the concentration of CMC Na in the preparation can increase the dispersion of galactomanan the form of molecular dispersion. It can bind to

DPPH free radicals so that increasing its antioxidant activity as indicated by a decrease in the IC50 value (Zahara et al., 2020).

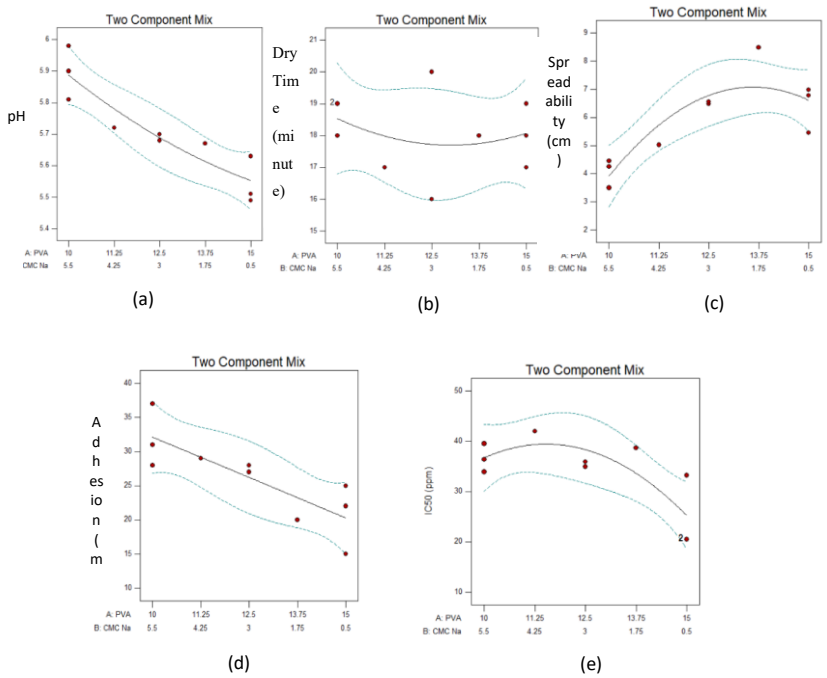

Figure 1. Contour Plot Effect Concentration of PVA and CMC Na on (a) pH; (b) Dry Time; (c) Spreadability; (d) Adhesion; and (e) IC50

Optimization

The optimum formula of peel-off gel mask preparation of kolang-kaling powder was obtained based on the target regression model of all response, namely pH, adhesion, spreading, drying time and IC50.

Table 4. Comparative Analysis of Prediction and Observed Data

Response	Prediction	Observation ±SD	p-value
pH	5.55	5.60 ± 0.03	0.06
Drying Time	18.06	18 ± 0.00	0.09
Spreadability	6.63	6.54 ± 0.03	0.09
Adhesion	20.34	20.28 ± 0.07	0.20
IC50	25.5	25.55 ± 0.73	0.91

Note: ($P<0.05$ means significantly different)

BUKTI KORESPONDENSI CATATAN REVIEWER 2

The selected formula is expected to produce peel-off gel mask preparation of kolang-kaling powder with optimal results for each parameter by determining the priority scale at a weight of 0.1-1. Furthermore, the optimization analysis will produce a desire function in which this study each parameter response is expected as an objective function and the desire function is developed to obtain optimum conditions (Mesa et al., 2017). The results of the model analysis show that the optimum formula is obtained at PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spreadability of 20.34 cm; drying time of 18 minutes and IC50 of 25.5 ppm. The formula has a desirability value of 0.954. The high desirability value indicates that the system will produce predicted values that are close to ideal (Amdoun et al., 2018) To ensure the predicted value, a one-sample t-test analysis was performed with the observation results. The data results show that the observation results are not significantly different from the predicted values (P>0.05) for all optimization parameters as shown in Table 4.

CONCLUSIONS

The optimal formula was achieved with a PVA concentration of 14.97% and CMC Na of 0.53%, resulting a predicted pH 5.55, adhesion 6.63 seconds, spreadability 20.34 cm, drying time 18 minutes, and IC50 25.5 ppm (desirability of 0.954). These values showed no significant differences from the observation results, indicating the validity of the optimization equations used for each parameter.

ACKNOWLEDGEMENTS

The author expresses gratitude to the Semarang Pharmacy Foundation for supporting this research through the lecturer grant program, funded by the Research and Community Service Institute (LPPM) of the Semarang Pharmacy Foundation, which has provided funding facilities to ensure the good execution of the study.

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BUKTI KORESPONDENSI CATATAN REVIEWER 2

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Intan Martha CAHYANI*, Eka Susanti HANHADYANAPUTRI, Ariani Hesti WULAN, Indah SULISTYARINI

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Abstract

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	I	II	III	IV	V	VI	VII	VIII	IX	X
Kolang-Kaling Powder (%)	2	2	2	2	2	2	2	2	2	2
PVA (%)	**10**	**12.5**	**15**	**10**	**13.75**	**11.25**	**10**	**12.5**	**15**	**15**
CMC Na (%)	**5.5**	**3**	**0.5**	**5.5**	**1.75**	**4.25**	**5.5**	**3**	**0.5**	**0.5**
Propylene Glycol (%)	10	10	10	10	10	10	10	10	10	10
Methyl Paraben (%)	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Aquadest (%)	Ad 100									

Physical Characteristics Test of Kolang-Kaling Peel-Off Gel Mask

pH Test

This was done by dipping the pH meter electrode into the preparation, leaving it until showing a constant pH value (Andini et al., 2017). A good peel-off gel mask has a pH that matches the skin's pH, namely in the range of 4.5-6.5 so that when the peel-off gel mask is applied, it does not cause irritation or cause dry and scaly skin (Silvia et al., 2021).

Drying Time Test

A peel-off gel mask of 0.1 grams was applied to an area of 2.5x2.5 cm on the arm and the time required for the preparation to dry is observed (Andini et al., 2017). The drying time for a good peel-off gel mask spent 15-30 minutes (Silvia et al., 2021).

Spreadability Test

Of 0.5 grams peel-off gel mask was placed in the center of the spreadability tester, covered with a glass plate that had been previously weighed and its weight recorded, left for one minute, then the four sides of the spread diameter were measured and a load of 50 grams was added, and left again for one minute. The spread diameter of the peel-off gel mask was then recorded as before. The addition of the load was stopped when the spread diameter of the peel-off gel mask is constant (Cahyani & Putri, 2017). The spread of a good peel off gel mask is in the range of 5-7 cm.(Silvia et al., 2021)

Adhesion Test

It was carried out by placing 0.25 grams of the peel-off gel mask on the object glass, given pressure with the addition of a load of 100 grams for 5 minutes and mounted on the

adhesion tester. The time for the two object glasses to be released was then recorded. The adhesion test of a good peel-off gel mask was more than 1 second.(Syam et al., 2021)

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

Vitamin C was produced in a series with concentrations of 2,4,6,8,10 ppm. Gel mask samples were made in concentrations of 20,40,60,70,80 ppm. A 2.0 ml sample was added with 2.0 ml of DPPH solution, placed in a test tube that had been coated with aluminum foil. Samples were incubated at room temperature for 23 minutes. Measure the absorption using a UV-Vis spectrophotometer at the maximum wavelength (516,60 nm). Perform testing on all runs and determine the IC50 value of each run (Diah et al., 2022).

RESULTS AND DISCUSSION

The peel-off gel mask preparation of kolang-kaling powder obtained was then tested for physical characteristics, including pH, drying time, spreadability, adhesion test and antioxidant activity (IC_{50}) with the results of each run can be seen in Table 2.

Table 2. Optimization Response to Characterization of Kolang-Kaling Peel-Off Gel Mask

Response	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
pH	5.98	5.70	5.63	5.81	5.69	5.72	5.90	5.68	5.51	5.49
Drying Time (minute)	19	16	17	18	18	17	19	20	18	19
Spreadability (cm)	4.25	6.48	5.45	3.50	8.48	5.02	4.45	6.56	6.78	6.98
Adhesion (second)	37	27	22	28	20	29	31	28	15	25
IC50 (ppm)	33.94	34.97	33.24	39.52	38.69	42.00	36.38	35.95	20.51	20.43

pH Test

Based on the optimization equation of the pH test parameters, it shows that PVA and CMC Na both have an effect on increasing the pH value with coefficients of +0.36 and +0.47 respectively (Table 3), but the effect of the increase was dominated by CMC Na because CMC Na has a higher pH (7.0 - 8.5) (Wijayanti et al., 2005) compared to PVA pH (5.0 – 8.0) (Arijani & Ariani, 2019). The results are in accordance with the pH test profile of the kolang-kaling

peel-off gel mask preparation shown in Figure 1a. where there is an increase in the curve that is proportional to the increase in CMC Na concentration.

Dry Time Test

Table 3 shows the optimization equation showing that the increase in drying time is significantly influenced by the increase in PVA and CMC Na concentrations (+1.18 and +2.15) with the increase in the curve being dominated by the influence of CMC Na (Figure 1b), while the combination of both has the opposite effect (-0.09). These results indicate that increasing the concentration of PVA can accelerate the drying time due to its adhesive properties (Septiani et al., 2021) so that it forms a dry layer more quickly, as report (Diah et al., 2022).

Table 3. Optimization Equation of Kolang-Kaling Peel-Off Gel Mask

Response	Equation	Model (p-value)	Lack of fit (p-value)
pH	$Y=0.36\ A + 0.47\ B - 4.97.10^{-3}\ AB$	0.0015	0.5356
Drying Time	$Y=1.18\ A + 2.15\ B - 0.09\ AB$	0.0268	0.7580
Spreadability	$Y=0.40\ A - 2.44\ B + 0.24\ AB$	0.0054	0.1025
Adhesion	$Y=1.23\ A + 3.56\ B + 4.21.10^{-3}\ AB$	0.0195	0.7032
IC50	$Y=1.35\ A - 7.53\ B + 1.18\ AB$	0.0268	0.4315

Note: ($P<0.05$ means significantly different)

Spreadability Test

The analysis results showed that PVA gave a synergistic effect (+0.40) while CMC Na was anatogonistic (-2.44) on the spreadability of the peel-off gel mask of kolang-kaling powder (Figure 1c). This is influenced by the adhesive properties of PVA which can form resistance so that it provides resistance to mass spread (Diah et al., 2022).

Adhesion Test

The synergistic effect is presented in the increase in the concentration of PVA (+1.23) and CMC Na (+3.56) on the adhesion power of the peel-off gel mask preparation of kolang-kaling powder. Figure 1d shows that the effect of PVA is lower than CMC Na because CMC Na is a hydrophilic polymer, which can expand with the presence of water so that the binding power between particles and viscosity increases (Kusuma et al., 2018). Meanwhile, PVA in high concentrations forms a thinner liquid so that the resulting synergistic effect is lower (Wibowo et al., 2022).

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

The results of the antioxidant activity test analysis showed that PVA gave a synergistic effect (+1.35) while CMC Na had an antagonistic effect (-7.53) on the IC50 value of the peel-off gel mask preparation of kolang-kaling powder as seen in the equation Table 3 and reinforced by Figure 1e. Increasing the concentration of CMC Na in the preparation can increase the dispersion of galactomanan the form of molecular dispersion. It can bind to DPPH free radicals so that increasing its antioxidant activity as indicated by a decrease in the IC50 value (Zahara et al., 2020).

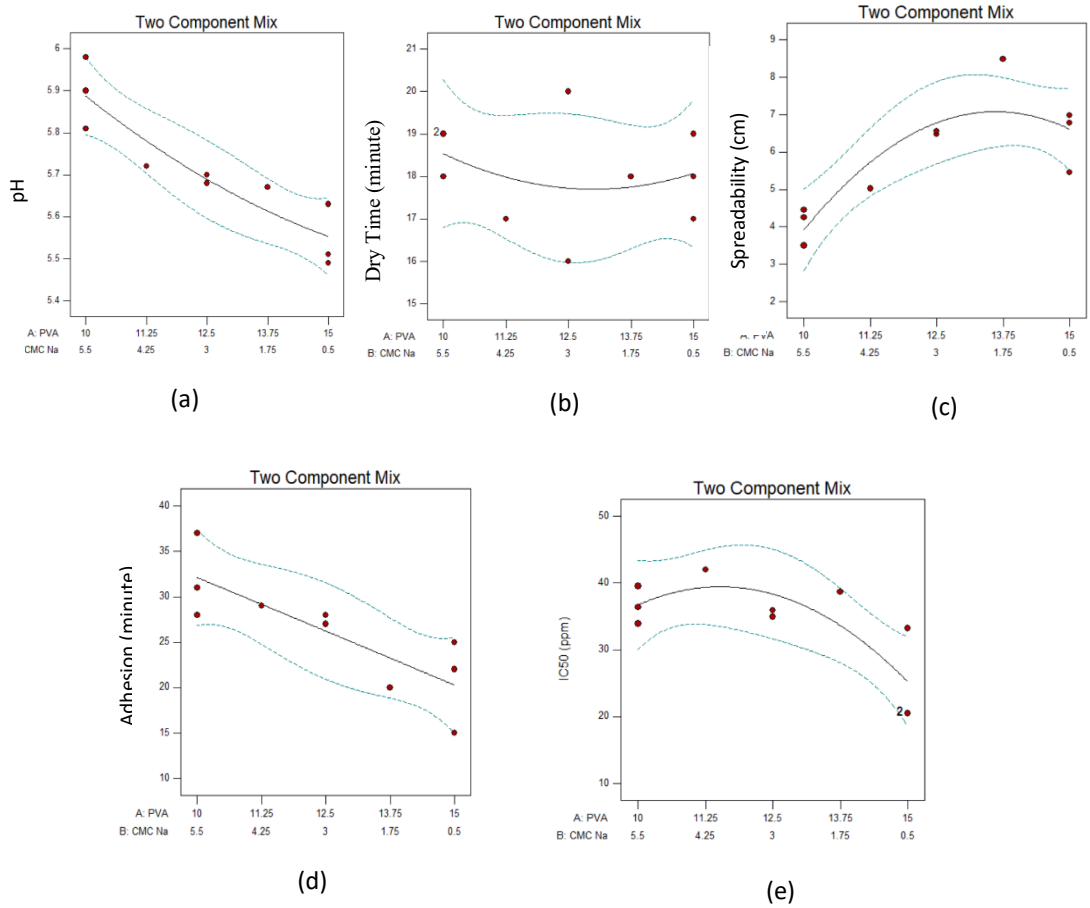

Figure 1. Contour Plot Effect Concentration of PVA and CMC Na on (a) pH; (b) Dry Time; (c) Spreadability; (d) Adhesion; and (e) IC50

Optimization

The optimum formula of peel-off gel mask preparation of kolang-kaling powder was obtained based on the target regression model of all response, namely pH, adhesion, spreading, drying time and IC50.

Table 4. Comparative Analysis of Prediction and Observed Data

Response	Prediction	Observation ±SD	p-value

pH	5.55	5.60 ± 0.03	0.06
Drying Time	18.06	18 ± 0.00	0.09
Spreadability	6.63	6.54 ± 0.03	0.09
Adhesion	20.34	20.28 ± 0.07	0.20
IC50	25.5	25.55 ± 0.73	0.91

Note: ($P<0.05$ means significantly different)

The selected formula is expected to produce peel-off gel mask preparation of kolang-kaling powder with optimal results for each parameter by determining the priority scale at a weight of 0.1-1. Furthermore, the optimization analysis will produce a desire function in which this study each parameter response is expected as an objective function and the desire function is developed to obtain optimum conditions (Mesa et al., 2017). The results of the model analysis show that the optimum formula is obtained at PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spreadability of 20.34 cm; drying time of 18 minutes and IC50 of 25.5 ppm. The formula has a desirability value of 0.954. The high desirability value indicates that the system will produce predicted values that are close to ideal (Amdoun et al., 2018) To ensure the predicted value, a one-sample t-test analysis was performed with the observation results. The data results show that the observation results are not significantly different from the predicted values ($P>0.05$) for all optimization parameters as shown in Table 4.

CONCLUSIONS

The optimal formula was achieved with a PVA concentration of 14.97% and CMC Na of 0.53%, resulting a predicted pH 5.55, adhesion 6.63 seconds, spreadability 20.34 cm, drying time 18 minutes, and IC50 25.5 ppm (desirability of 0.954). These values showed no significant differences from the observation results, indicating the validity of the optimization equations used for each parameter.

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OPTIMIZATION FORMULA OF KOLANG-KALING (ARENGA PINNATA.) PEEL-OFF GEL MASK WITH COMBINATION PVA - CMC NA AS GELLING AGENT ON SIMPLEX LATTICE DESIGN

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Abstract

Kolang-kaling fruit in topical preparations increase its effectiveness as an antioxidant. The peel-off gel mask is as an effective topical preparation for skin health and user comfort. Its formulation is determined by quality parameters, particularly forming a plastic film of Polyvinyl Alcohol (PVA) and Carboxymethylcellulose Sodium (CMC Na) as polymers. This study aims to determine the effect of these combination and the ratio of both concentrations based on the physical characteristics and IC_{50} value. The optimization on the simplex lattice design was obtained in 8 runs with PVA concentrations (10% -15%) and CMC Na (0.5% - 5.5%). The pH value, spreadability, adhesion, drying time, and IC50 were used as parameters to determine optimal formula. The results showed that the difference in PVA and CMC Na concentrations had a significant effect on all optimization parameters (p-value <0.05) and the lack of fit for each replication was not significant (p-value> 0.05). The optimal formula obtained PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spread power of 20.34 cm; drying time of 18 minutes, and IC50 of 25.5 ppm (desirability of 0.954), which is not significantly different from the observation results indicating the optimization is valid.

Keyword: Kolang-kaling, Optimization, Peel-off gel mask, PVA, CMC Na.

INTRODUCTION

Kolang-kaling is a type of *Aren* fruit with a chewy texture and a fresh taste, it is widely used as an ingredient in drinks or traditional foods. Several studies have stated that kolang-kaling has numerous health benefits because of the content of compounds, including 9.1% calcium (bones) (Holly et al., 2018) flavonoids (joint pain) (Yolanda & Febriyanti, 2021) crude fiber 10.52% (digestion), and galactomannan, which is widely used as an emulsifier and thickener in ice cream (Ihsan et al., 2022).

Galactomannan, is a natural hydrocolloid (Hasna, 2020) the content of kolang-kaling is 10μg/mL with antioxidant activity >50% higher than vitamin C (Yanti & Ali, 2017). In another study, it was stated that the antioxidant activity of galactomannan was included in the very strong category with IC_{50} (20.45 ppm) (Tarigan, 2012). The process of processing and drying Kolang-kalinginto powder with several heating methods can affect the antioxidant

activity of glucomannan. The drying method that can maintain antioxidant activity in the very strong category is a microwave at a temperature of 40°C for 22 hours with an IC_{50} value of 25.30 ppm (Hanhadyanaputri et al., 2023) showing the potential to be developed as a peel-off gel mask preparation to increase its effectiveness and comfort.

Peel-off gel mask is the right dosage form for use on facial skin with an elastic membrane and is easy to remove after use (Sulastri & Dewi, 2018) in its formulation, gelling agent plays an important role for this purpose. PVA can function as a gelling agent because it is a hydrophilic polymer with adhesive properties that will help provide a film layer effect called the peel-off effect and can be easily peeled off after drying (Anung Anindhita et al., 2023). Not only does PVA have advantages as a gelling agent, but there are also disadvantages that must be considered in its use, namely the formation of a rigid and easily broken film layer (Sinambela & Telaumbanua, 2022). CMC Na is a hydrophilic polymer with a more stable viscosity over a long period of time (Eryani et al., 2023) thus it can increase the elasticity of the PVA layer formed. This study aims to determine the effect of the combination of PVA and CMC Na and the ratio of these concentrations to obtain the optimal peel-off gel mask formula for kolang-kaling.

MATERIALS AND METHODS

a) Materials and Tools

The materials needed are kolang-kaling taken from Gunung Pati Semarang PVA, CMC Na, propilenglycol, methylparaben, 2.2-diphenyl-1-picrylhydrazyl (DPPH), and Vitamin C. In this research process, tools are needed, including a blender, microwave, pH meter, and UV-Vis spectrophotometer.

b) Methods

Preparation of Kolang-Kaling Powder

Soak the kolang-kaling in clean water for 1 x 24 hours, wash it clean, drain, thinly sliced, and dried in a microwave at 40ºC for 22 hours, blended and sieved through mesh No. 44.

Preparation of Kolang-Kaling Peel-Off Gel Mask

PVA, CMC Na, and palm sugar powder were each developed with 80ºC aqua destillata, then mixed and homogenized. The mixture was added with methylparaben that had been dissolved in propylene glycol, and stirred until homogeneous. The proportion of PVA and CMC Na was determined according to the optimization design with the simplex lattice design method according to the formula for each run as seen in Table 1.

Table 1. Design Optimization of Kolang-Kaling Peel-Off Gel Mask

Formula	**RUN**									
	I	**II**	**III**	**IV**	**V**	**VI**	**VII**	**VIII**	**IX**	**X**
Kolang-Kaling Powder (%)	2	2	2	2	2	2	2	2	2	2
PVA (%)	**10**	**12.5**	**15**	**10**	**13.75**	**11.25**	**10**	**12.5**	**15**	**15**
CMC Na (%)	**5.5**	**3**	**0.5**	**5.5**	**1.75**	**4.25**	**5.5**	**3**	**0.5**	**0.5**
Propylene Glycol (%)	10	10	10	10	10	10	10	10	10	10
Methyl Paraben (%)	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
Aquadest (%)	Ad 100									

Physical Characteristics Test of Kolang-Kaling Peel-Off Gel Mask

pH Test

This was done by dipping the pH meter electrode into the preparation, leaving it until showing a constant pH value (Andini et al., 2017). A good peel-off gel mask has a pH that matches the skin's pH, namely in the range of 4.5-6.5 so that when the peel-off gel mask is applied, it does not cause irritation or cause dry and scaly skin (Silvia et al., 2021).

Drying Time Test

A peel-off gel mask of 0.1 grams was applied to an area of 2.5x2.5 cm on the arm and the time required for the preparation to dry is observed (Andini et al., 2017). The drying time for a good peel-off gel mask spent 15-30 minutes (Silvia et al., 2021).

Spreadability Test

Of 0.5 grams peel-off gel mask was placed in the center of the spreadability tester, covered with a glass plate that had been previously weighed and its weight recorded, left for one minute, then the four sides of the spread diameter were measured and a load of 50 grams was added, and left again for one minute. The spread diameter of the peel-off gel mask was then recorded as before. The addition of the load was stopped when the spread diameter of the peel-off gel mask is constant (Cahyani & Putri, 2017). The spread of a good peel off gel mask is in the range of 5-7 cm.(Silvia et al., 2021)

Adhesion Test

It was carried out by placing 0.25 grams of the peel-off gel mask on the object glass, given pressure with the addition of a load of 100 grams for 5 minutes and mounted on the adhesion tester. The time for the two object glasses to be released was then recorded. The adhesion test of a good peel-off gel mask was more than 1 second.(Syam et al., 2021)

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

Vitamin C was produced in a series with concentrations of 2,4,6,8,10 ppm. Gel mask samples were made in concentrations of 20,40,60,70,80 ppm. A 2.0 ml sample was added with 2.0 ml of DPPH solution, placed in a test tube that had been coated with aluminum foil. Samples were incubated at room temperature for 23 minutes. Measure the absorption using a UV-Vis spectrophotometer at the maximum wavelength (516,60 nm). Perform testing on all runs and determine the IC50 value of each run (Diah et al., 2022).

RESULTS AND DISCUSSION

The peel-off gel mask preparation of kolang-kaling powder obtained was then tested for physical characteristics, including pH, drying time, spreadability, adhesion test and antioxidant activity (IC_{50}) with the results of each run can be seen in Table 2.

Table 2. Optimization Response to Characterization of Kolang-Kaling Peel-Off Gel Mask

Response	RUN									
	I	II	III	IV	V	VI	VII	VIII	IX	X
pH	5.98	5.70	5.63	5.81	5.69	5.72	5.90	5.68	5.51	5.49
Drying Time (minute)	19	16	17	18	18	17	19	20	18	19
Spreadability (cm)	4.25	6.48	5.45	3.50	8.48	5.02	4.45	6.56	6.78	6.98
Adhesion (second)	37	27	22	28	20	29	31	28	15	25
IC50 (ppm)	33.94	34.97	33.24	39.52	38.69	42.00	36.38	35.95	20.51	20.43

pH Test

Based on the optimization equation of the pH test parameters, it shows that PVA and CMC Na both have an effect on increasing the pH value with coefficients of +0.36 and +0.47 respectively (Table 3), but the effect of the increase was dominated by CMC Na because CMC Na has a higher pH (7.0 - 8.5) (Wijayanti et al., 2005) compared to PVA pH (5.0 – 8.0) (Arijani & Ariani, 2019). The results are in accordance with the pH test profile of the kolang-kaling peel-off gel mask preparation shown in Figure 1a. where there is an increase in the curve that is proportional to the increase in CMC Na concentration.

Dry Time Test

Table 3 shows the optimization equation showing that the increase in drying time is significantly influenced by the increase in PVA and CMC Na concentrations (+1.18 and

+2.15) with the increase in the curve being dominated by the influence of CMC Na (Figure 1b), while the combination of both has the opposite effect (-0.09). These results indicate that increasing the concentration of PVA can accelerate the drying time due to its adhesive properties (Septiani et al., 2021) so that it forms a dry layer more quickly, as report (Diah et al., 2022).

Table 3. Optimization Equation of Kolang-Kaling Peel-Off Gel Mask

Response	Equation	Model (p-value)	Lack of fit (p-value)
pH	$Y=0.36\ A + 0.47\ B – 4.97.10^{-3}\ AB$	0.0015	0.5356
Drying Time	$Y=1.18\ A + 2.15\ B – 0.09\ AB$	0.0268	0.7580
Spreadability	$Y=0.40\ A – 2.44\ B + 0.24\ AB$	0.0054	0.1025
Adhesion	$Y=1.23\ A + 3.56\ B + 4.21.10^{-3}\ AB$	0.0195	0.7032
IC50	$Y=1.35\ A – 7.53\ B + 1.18\ AB$	0.0268	0.4315

Note: ($P<0.05$ means significantly different)

Spreadability Test

The analysis results showed that PVA gave a synergistic effect (+0.40) while CMC Na was anatogonistic (-2.44) on the spreadability of the peel-off gel mask of kolang-kaling powder (Figure 1c). This is influenced by the adhesive properties of PVA which can form resistance so that it provides resistance to mass spread (Diah et al., 2022).

Adhesion Test

The synergistic effect is presented in the increase in the concentration of PVA (+1.23) and CMC Na (+3.56) on the adhesion power of the peel-off gel mask preparation of kolang-kaling powder. Figure 1d shows that the effect of PVA is lower than CMC Na because CMC Na is a hydrophilic polymer, which can expand with the presence of water so that the binding power between particles and viscosity increases (Kusuma et al., 2018). Meanwhile, PVA in high concentrations forms a thinner liquid so that the resulting synergistic effect is lower (Wibowo et al., 2022).

Antioxidant Activity Test by DPPH (2,2-diphenyl-1-picrylhydrazyl) Method

The results of the antioxidant activity test analysis showed that PVA gave a synergistic effect (+1.35) while CMC Na had an antagonistic effect (-7.53) on the IC50 value of the peel-off gel mask preparation of kolang-kaling powder as seen in the equation Table 3 and reinforced by Figure 1e. Increasing the concentration of CMC Na in the preparation can increase the dispersion of galactomanan the form of molecular dispersion. It can bind to

DPPH free radicals so that increasing its antioxidant activity as indicated by a decrease in the IC50 value (Zahara et al., 2020).

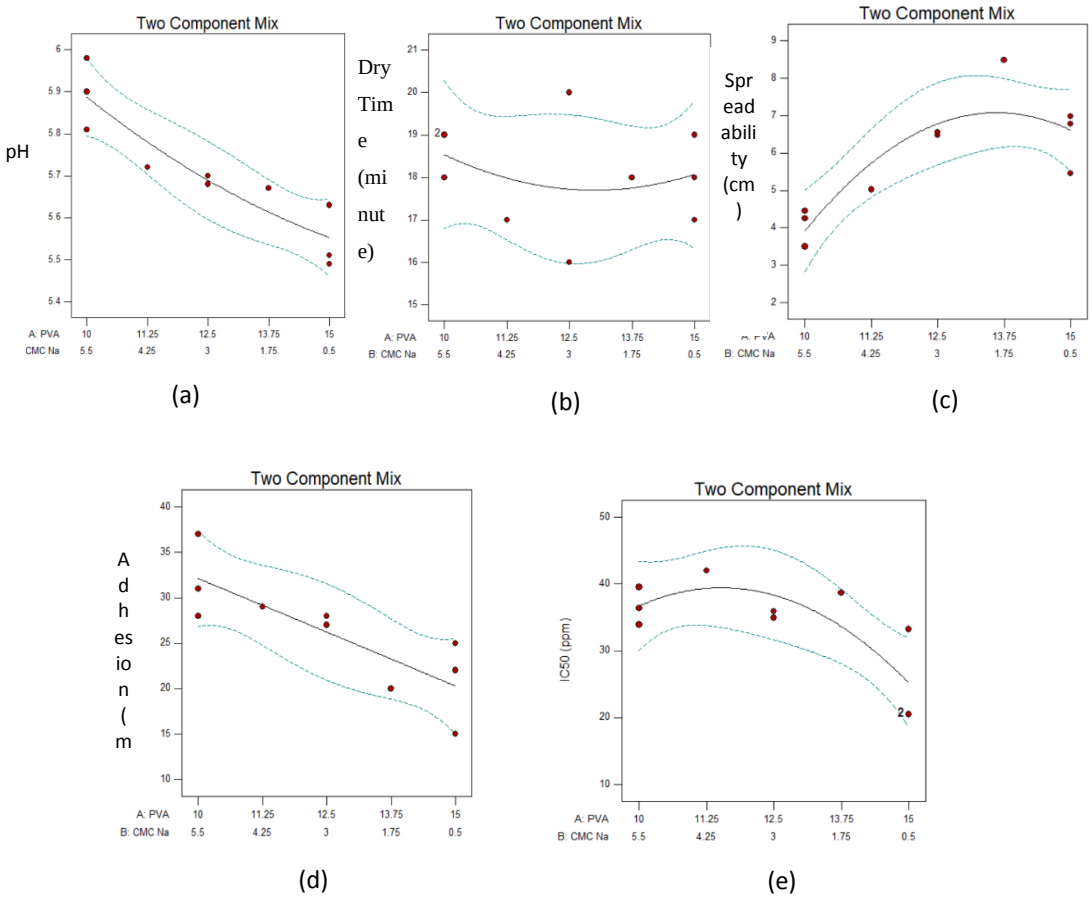

Figure 1. Contour Plot Effect Concentration of PVA and CMC Na on (a) pH; (b) Dry Time; (c) Spreadability; (d) Adhesion; and (e) IC50

Optimization

The optimum formula of peel-off gel mask preparation of kolang-kaling powder was obtained based on the target regression model of all response, namely pH, adhesion, spreading, drying time and IC50.

Table 4. Comparative Analysis of Prediction and Observed Data

Response	Prediction	Observation ±SD	p-value
pH	5.55	5.60 ± 0.03	0.06
Drying Time	18.06	18 ± 0.00	0.09
Spreadability	6.63	6.54 ± 0.03	0.09
Adhesion	20.34	20.28 ± 0.07	0.20
IC50	25.5	25.55 ± 0.73	0.91

Note: ($P<0.05$ means significantly different)

The selected formula is expected to produce peel-off gel mask preparation of kolang-kaling powder with optimal results for each parameter by determining the priority scale at a weight of 0.1-1. Furthermore, the optimization analysis will produce a desire function in which this study each parameter response is expected as an objective function and the desire function is developed to obtain optimum conditions (Mesa et al., 2017). The results of the model analysis show that the optimum formula is obtained at PVA concentration of 14.97% and CMC Na 0.53% with a predicted pH of 5.55; adhesion of 6.63 seconds; spreadability of 20.34 cm; drying time of 18 minutes and IC50 of 25.5 ppm. The formula has a desirability value of 0.954. The high desirability value indicates that the system will produce predicted values that are close to ideal (Amdoun et al., 2018) To ensure the predicted value, a one-sample t-test analysis was performed with the observation results. The data results show that the observation results are not significantly different from the predicted values (P>0.05) for all optimization parameters as shown in Table 4.

CONCLUSIONS

The optimal formula was achieved with a PVA concentration of 14.97% and CMC Na of 0.53%, resulting a predicted pH 5.55, adhesion 6.63 seconds, spreadability 20.34 cm, drying time 18 minutes, and IC50 25.5 ppm (desirability of 0.954). These values showed no significant differences from the observation results, indicating the validity of the optimization equations used for each parameter.

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