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Optimization of Formulation and Physical Evaluation of Peel-Off Gel Face Mask from Clove Leaf Essential Oil (*Syzygium aromaticum L.*)

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Abstract: Acne is an inflammatory infection triggered by *Propionibacterium acnes* bacteria. Clove leaf essential oil (*Syzygium aromaticum* L.) has attracted attention as a potential active ingredient in skincare products, such as peel-off gel face masks, due to its anti-inflammatory properties. This study aims to develop a natural, effective, and safe skincare product for treating acne using clove leaf essential oil as the active ingredient. This study is experimental, the active ingredient was 2% essential oil, the determination of the optimum formula is carried out by varying two factors, namely PVA (A; %) as forming agent and propylene glycol (B; %) as humectant using the SLD method, with 4 observed responses (pH, spreadability, drying time, dan viscosity). The optimum formulation of the peel-off gel face mask containing clove leaf essential oil was achieved with a PVA concentration of 1.163% and propylene glycol at 6.333%, with a desirability value of 1.000. The physical evaluation results of the optimum formula showed a pH value of 5.90, a spreadability of 6.60 cm, a drying time of 26.08 minutes, and a viscosity of 30,682.50 cps.

Keywords: Face Mask; Peel-Off Gel; Clove Leaf; Essential Oil

1. INTRODUCTION

The skin is the outermost organ of the human body that directly interacts with the environment. The skin has the function of protecting the body from negative influences originating from outside the human body. The skin must be cared for so that it is not easily attacked by disease or bacteria, especially on facial skin [1]. According to Febriana et. al., [2], the skin is the outermost part of the body that serves as both an internal body protector and an aesthetic factor. The skin acts as a barrier against ultraviolet (UV) radiation and microorganisms that can trigger skin problems such as acne, wrinkles, premature aging, and many others. Skin issues may arise due to free radicals or bacterial infections, leading to dryness, dullness, and acne formation [2].

Acne, also known as *acne vulgaris*, is an inflammatory infection of the pilosebaceous unit, characterized by blockage and accumulation of keratinous material, which can be triggered by *Propionibacterium acnes*, *Staphylococcus epidermidis*, and *Staphylococcus aureus*. However, the primary bacteria responsible for acne is *Propionibacterium acnes*. This gram-positive bacterium can infect the

skin and the gastrointestinal tract. *P. acnes* causes opportunistic infections such as acne, especially during puberty, due to increased androgen activity, which stimulates sebaceous gland growth and sebum production [3]. Acne occurs due to chronic inflammation of the pilosebaceous glands, leading to blackheads and lesions, which commonly appear in areas rich in sebaceous glands, such as the face, neck, chest, and back [2], [4].

The prevalence of acne in Indonesia is approximately 3% in individuals aged 35-44 years, 12% in women over 25 years, and 80-85% in teenagers, with peak incidence occurring between 15-18 years of age [2]. The number of acne cases in developing countries varies from 40% to 80%. Vasam et al., (2023) stated that acne vulgaris is the most common type, accounting for 99% of all acne cases [5]. In Indonesia, the prevalence of acne among teenagers is between 80% and 85%, and this number increases annually. A study conducted in 2019 on 66 acne vulgaris patients at Abdul Moeloek Hospital found that 69.7% of women suffered from acne compared to 30.3% of men [6]. Acne outbreaks often affect an individual's self-confidence. About 30-50% of people with acne tend to experience low self-esteem and psychological distress due to their perception that acne negatively impacts their appearance [7].

With the increasing consumer interest in natural skincare products, clove leaf essential oil (*Syzygium aromaticum* L.) has gained attention as a potential active ingredient in skincare formulations, such as peel-off gel face masks, due to its anti-inflammatory, antimicrobial, and antioxidant properties [8]. Benefit from peel off gel mask is a type of gel mask that will leave a thin film layer and can be peeled off [9]. Clove plants contain essential oils in large enough quantities, both in flowers, bud and leaves [10]. Clove leaf essential oil (*Syzygium aromaticum* L.) has long been used in traditional medicine for its various pharmacological properties, including anti-inflammatory and antimicrobial effects [11]. Clove possesses stimulant, antiseptic, and antispasmodic properties [12]. Clove oil has been reported to exhibit antimicrobial activity against acne-causing microorganisms such as *Propionibacterium acnes*, *Staphylococcus epidermidis*, *Staphylococcus aureus*, and *Candida albicans* [5]. Despite its therapeutic potential, studies indicate that eugenol, the main component of clove oil, has low bioavailability due to its limited water solubility [13].

Therefore, research on the formulation and physical evaluation of peel-off gel face masks containing clove leaf essential oil as an anti-acne agent is essential for further exploration. This study aims to develop an effective and safe natural skincare product for acne treatment by utilizing clove leaf essential oil as the main active ingredient. Consequently, this research not only offers a more natural and safer alternative for skincare but also enhances the understanding of clove leaf essential oil's potential in treating skin and acne-related issues.

2. MATERIALS AND METHODS

2.1. Materials

The equipment used in this study includes a stirring rod Iwaki (Iwaki, Indonesia), 50 mL and 100 mL beakers Pyrex (Pyrex, Indonesia), a 10 mL graduated cylinder Pyrex (Pyrex, Indonesia), a porcelain dish Pyrex (Pyrex, Indonesia), a dropper pipette OneMed (OneMed, Indonesia), a volumetric pipette Iwaki (Iwaki, Indonesia), a mortar and pestle OneMed (OneMed, Indonesia), a

watch glass Duran (Duran, Indonesia), a hotplate Ohaus (Ohaus, Indonesia), a pH meter Ohaus (Ohaus, Indonesia), a Brookfield viscometer Ametek (Ametek, Indonesia), a spreadability test apparatus, and an analytical balance Ohaus (Ohaus, Indonesia). The primary ingredients used in this study include clove leaf essential oil (*Syzygium aromaticum* L.) (NuAroma, Indonesia), PVA (Kuraray, Indonesia), propylene glycol, HPMC, methyl paraben, propyl paraben, and distilled water.

2.2. Methods

2.2.1 Optimization of the Peel-Off Gel Face Mask Formulation

Optimization was performed using Design Expert 13 software to optimize PVA as a film-forming agent and propylene glycol as a humectant in the formulation of a peel-off gel face mask containing clove leaf essential oil (*Syzygium aromaticum* L.). PVA concentration is the most important factor affecting the film formation performance [14]. According to the *Handbook of Pharmaceutical Excipients*, the concentration ranges for the ingredients used were PVA (1–2.5%) and propylene glycol (5–6.5%). Eight formulations were developed for optimization, with response parameters used to determine physical properties, including pH value, spreadability, drying time, and viscosity. The results of the eight formulations are presented in Table 1.

Table 1. Peel-Off Gel Face Mask Formulation

Material	Concentration (%)								Function
	F1	**F2**	**F3**	**F4**	**F5**	**F6**	**F7**	**F8**	
Clove Leaf Essential Oil	2	2	2	2	2	2	2	2	Active Substance
PVA*	1.75	1	2.5	1.75	2.5	1	2.13	1.38	Film-Forming Agent
HPMC	2	2	2	2	2	2	2	2	Gelling Agent
Propylene Glycol*	5.17	6.5	5	5.75	5	6.5	5.38	6.13	Humectant
Metyl Paraben	0.18	0.18	0.18	0.18	0.18	0.18	0.18	0.18	Preservative
Propyl Paraben	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	Preservative
Aquadest	Ad 100	Ad 100	Ad 100	Ad 100	Ad 100	Ad 100	Ad 100	Ad 100	Solvent

** based on the results of running the formula using SLD method*

2.2.2. Preparation of Face Mask Gel Peel-Off

The face mask gel peel-off was prepared according to the formula in Table 1. HPMC was weighed and poured into a mortar, then dissolved in hot distilled water. PVA was weighed and placed in a beaker, then dissolved in hot distilled water on a hotplate. Methylparaben and propylparaben were dissolved in propylene glycol in a porcelain dish. The PVA solution was mixed with HPMC in a mortar and stirred until homogeneous. The methylparaben, propylparaben, and propylene glycol solution was then added to the mortar and stirred until homogeneous. Two milliliters of essential oil were added to the mortar and stirred until homogeneous. The remaining distilled water was added up to 100 ml, stirred until homogeneous, and formed a gel.

2.2.3. Physical Evaluation of Face Mask Gel Peel Off

a. Organoleptic Test

The organoleptic test was conducted to observe the physical texture of the formulation by direct visual assessment of the appearance, color, and odor of the clove leaf (*Syzygium aromaticum* L.) essential oil face mask gel peel-off [15], [16], [17].

b. Homogeneity Test

This test was conducted by applying the face mask gel peel-off to two glass slides and examining whether there were any clumps or particles in the clove leaf essential oil face mask gel peel-off formulation [15].

c. Viscosity Test

This test was conducted to determine the viscosity of the formulation. A Brookfield viscometer with spindle no. 4 at a speed of 10 rpm was used [18].

d. Spreadability Test

This test was performed by placing 1 gram of the formulation at the center of a glass plate with a millimeter block sheet attached. The glass plate was then subjected to weights of 0 grams, 50 grams, 100 grams, and 150 grams. The spread diameter was measured for each added weight over a duration of 1 minute [14], [15].

e. pH Test

The pH measurement was conducted using a pH meter. One gram of the face mask gel peel-off was dissolved in 10 ml of distilled water, and the pH of the solution was measured using a standardized pH meter [15], [19].

f. Drying Time Test

This test was conducted to determine the time required for the formulation to form a dried film layer. The formulation was applied to the back of the hand, and the drying time was observed from the moment of application until a dry layer formed [15].

g. Data Analysis

The clove leaf essential oil face mask gel peel-off was optimized using Simplex Lattice Design (SLD) with the Design Expert 13 application. The response parameters observed included pH, spreadability, drying time, and viscosity [20]. The evaluation results were used to determine the optimal formulation. If the optimal formulation was obtained, data analysis was performed using the Shapiro-Wilk normality test. If the data were normally distributed, a One Sample T-Test was conducted. If the results were not normally distributed and homogeneous, the Kruskal-Wallis and Mann-Whitney tests were used.

3. RESULTS AND DISCUSSION

3.1. Results

3.1.1. Optimization of Face Mask Gel Peel Off Formulation form Clove Leaf Essential Oil

The physical evaluation results of eight run formulations were analyzed using the Design Expert 13 application to determine the optimal formula using the SLD method. The optimal formula was determined based on the results of pH testing, spreadability, drying time, and viscosity. The results of each formulation are presented in Table 2.

Figure 1. Formulation of Face Mask Gel Peel Off Clove Leaf Essential Oil

Table 2. Result of Each Formulation Test

Formula	Concentration (%)			Test Results		
	PVA	Propylene Glycol	pH	Spreadability (cm)	Drying Time (minute)	Viscosity (cps)
1	1.75	5.75	5.79	6.1	26.50	31980
2	1	6.5	5.45	6.2	26.20	38000
3	2.5	5	5.85	6.3	26.58	24400
4	1.75	5.75	5.54	6.2	26.60	37350
5	2.5	5	6.47	6.5	26.60	35400
6	1	6.5	5.43	6.1	26.45	39000
7	2.13	5.38	5.51	6.3	26.45	32940
8	1.38	6.13	5.39	6.2	26.33	36500

The test results were analyzed using ANOVA in the Design Expert 13 software to determine the significance of the responses and identify the model recommended by the software. The following are the ANOVA analysis results for each response using Design Expert 13.

Table 3. ANOVA Analysis Results for Each Respons Using Design Expert 13

Parameter	ANOVA Results			
	pH	Spreadability	Drying Time	Viscosity
Model : linier mixture	0.0358	0.0313	0.0343	0.0458
Residual : Lack of fit	0.5527	0.6042	0.6062	0.9984
R-square	0.5476	0.5661	0.5534	0.5125
Adeq.precision	5.0815	5.2755	5.1413	4.7357

The results of each parameter test were analyzed using ANOVA with Design Expert 13 to determine the significance of the obtained values and identify the recommended model in the application, as shown in Table 3. Based on the physical quality test data from the eight formulations, one optimum formulation was obtained. The results from the *Simple Lattice Design* (SLD) recommendations can be seen in Table 4.

Table 4. Optimum Formulation and Predicted Test Result form Design Expert 13

Composition (%)		Test Results				*Desirability*
PVA	**Propylene glykol**	**pH**	**Spreadability**	**Drying Time**	**Viscosity**	
1.167	6.333	5.90	6.60	26.08	30682.50	1.000

3.1.2. Evaluation of the Optimum Peel-Off Gel Mask Formulation from Clove Leaf Essential Oil

The optimum formulation obtained was then prepared and subjected to physical evaluation testing, repeated three times. The physical evaluation results of the optimum formulation are presented in Table 5.

Table 5. Physical evaluation results of the Optimum Face Mask Formulation

Parameter	Replication			Mean	Acceptance Criteria	Information
	1	**2**	**3**			
Form	Thick	Thick	Thick	Thick	Thick	Qualify
Color	White	White	White	White	White	Qualify
Smell	Typical of clove leaves	Typical of clove leaves	Typical of clove leaves	Typical of clove leaves	Typical of clove leaves	Qualify
Homogeneity	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Homogeneous	Qualify
pH	5.98	5.88	5.89	5.91	4.5 – 6.5	Qualify
Spreadability	5.8	6.4	6.7	6.3	5 – 7	Qualify
Drying Time	25.55	26.30	26.43	26.09	>30	Qualify
Viscosity	31200	30120	29760	30360	2000 - 50000	Qualify

3.1.3. Analysis of Results

The analysis of the optimization results for the peel-off gel face mask formulation containing clove leaf essential oil (*Syzygium aromaticum* L.) was conducted by processing prediction and experimental data using SPSS. The results are presented in Table 6.

Table 6. Normality Test (*Shapiro-Wilk*)

Normality Test (*Shapiro-Wilk*)	Sig	Conclusion
pH	0.174	Significant
Spreadability	0.637	Significant
Drying Time	0.262	Significant
Viscosity	0.463	Significant

The Shapiro-Wilk normality test results indicate that the data are normally distributed ($p > 0.05$). The analysis was then continued with a one-sample T-test. The obtained data are presented in Table 7.

Table 7. One-Sample T-Test

T-Test	**Predictions**	**Experiment**	**Sig**	**conclusion**
pH	5.994	5.91	0.652	Not Different
Spreadability	6.600	6.30	0.374	Not Different
Drying Time	26.085	26.09	0.966	Not Different
Viscosity	30682.500	30360	0.534	Not Different

The test results show a significant value ($p > 0.05$), indicating no significant difference between the predicted and experimental formulations. This suggests that the predicted and experimental results are consistent and demonstrate similar outcomes.

3.2. Discussion

3.2.1. Optimization of Peel-Off Gel Face Mask Formulation with Clove Leaf Essential Oil

Optimization was conducted using Design Expert 13 software to obtain the optimum concentration of PVA as a binder and propylene glycol as a humectant in the peel-off gel face mask formulation containing clove leaf essential oil (*Syzygium aromaticum* L.). Design Expert 13 generated eight formulations for physical evaluation testing. The results of these tests were analyzed using the Simple Lattice Design (SLD) method, and one optimum formulation was identified that met the physical evaluation criteria.

Each test parameter result was analyzed using ANOVA in Design Expert 13 to determine the significance of the obtained values and to identify the recommended model. A good model is indicated by a linear mixture model with a *p*-value of less than 0.05, meaning that the value is significant. Additionally, a *p*-value greater than 0.05 for the lack-of-fit test indicates that the value is not significant. A non-significant lack-of-fit value suggests that the desired model is appropriate and has minimal noise. An R-Square (R^2) value close to 1 indicates that the model is recommended. Furthermore, an adequate precision value greater than 4 suggests that the model is acceptable. Based on these criteria, the obtained results meet the necessary requirements, confirming that each conducted test satisfies the established standards[21].

According to Ittiqo *et.al.,* (2019), the normal probability plot of residuals for each evaluation demonstrates data distribution along a linear equation or optimum formula equation that spreads around and follows the direction of the diagonal line [22]. This indicates that the data are normally distributed and can be further analyzed [21].

The optimization results for the peel-off gel face mask formulation containing clove leaf essential oil (*Syzygium aromaticum* L.) yielded an optimum concentration of 1.167% PVA and 6.333% propylene glycol, with a desirability value of 1.000. A study conducted by Ittiqo et al. (2019) stated that a desirability value close to 1 indicates a well-optimized formula, meaning the obtained formulation closely aligns with the desired optimum formula [22]. In contrast, research conducted by Widynyana *et. al.,* (2021) suggests that a desirability value approaching 0 indicates an undesirable formulation [21].

3.2.2. Evaluation of the Optimum Peel-Off Gel Face Mask Formulation with Clove Leaf essential Oil

The optimum formulation obtained was reformulated and re-evaluated for its physical properties through three replications. A good peel-off gel face mask formulation can be determined through evaluation tests such as organoleptic testing, pH testing, viscosity testing, spreadability testing, and drying time testing [23].

The organoleptic test was conducted to observe the appearance, color, and odor of the prepared formulation. The results showed that the formulation had a white color, a characteristic clove-like aroma, and a thick consistency. This aligns with the study by Kurniasari *et. al.,* [6], which stated that the formulation should have a mild clove scent and a homogeneous gel texture that is smooth upon application to the skin.

The homogeneity test was performed by placing the sample between two glass slides and observing whether any coarse particles were present and whether the formulation had a uniform color [24]. The homogeneity test results showed that the formulation had a consistent color and contained no coarse particles, indicating that it was homogeneous.

The pH test was conducted using a pH meter to determine whether the formulation met the required pH standards. The acceptable pH range for topical formulations is 4.5 to 6.5, which corresponds to normal skin pH. If the pH is too alkaline, the formulation may feel slippery and cause skin dryness, whereas if it is too acidic, it may cause skin irritation [25]. The pH test result was 5.91, which falls within the acceptable range for topical formulations.

The spreadability test was performed by weighing 1 gram of the formulation and placing it in the center of a 20×20 cm glass plate, which was then covered with another glass plate. Weights of 0, 50, 100, and 150 grams were applied sequentially for 1 minute each [18]. The acceptable spreadability range is 5–7 cm [25]. The spreadability test result was 6.3 cm, indicating that the formulation met the required criteria.

The drying time test involved weighing 0.7 grams of the formulation and applying a thin layer onto the skin while timing how long it took to dry completely. The acceptable drying time should not exceed 30 minutes [25]. The drying time test result was 26.09 minutes, indicating that the formulation met the required standards.

The viscosity test was conducted using a Brookfield viscometer with spindle No. 4 at a speed of 10 rpm. The viscosity test result was 30,360 cps. According to Samsul *et. al.,* (2022), a good peel-off gel face mask should have a viscosity between 2,000 and 50,000 cps, meaning that the prepared formulation met the required criteria [24].

3.2.3. Result Analysis

This analysis aims to determine whether there are differences between the predicted formulation and the experimental formulation. After conducting the analysis, the significance results for the physical evaluation tests—pH, spreadability, drying time, and viscosity—were found to be 0.652, 0.374, 0.966, and 0.534, respectively ($p > 0.05$). These results indicate that there is no significant difference between the predicted and experimental formulations, confirming that the optimum peel-off gel face mask formulation with clove leaf essential oil (*Syzygium aromaticum* L.) meets the required standards.

4. CONCLUSION

This analysis aims to determine whether there is a difference between the predicted formulation and the experimental formulation. After conducting the analysis, the significance results for the physical evaluation tests—pH, spreadability, drying time, and viscosity—were 0.652, 0.374, 0.966, and 0.534, respectively ($p > 0.05$). These results indicate that there is no significant difference between the predicted and experimental formulations, confirming that the optimum peel-off gel face mask formulation with clove leaf essential oil (*Syzygium aromaticum* L.) meets the required standards.

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