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Formulation of Gel Preparation Based on a Combination of Activated Palm Shell Charcoal and Tea Tree Oil as an Antibacterial for Cutibacterium Acnes

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ABSTRACT: Acne (acne vulgaris) is a common skin problem caused by clogged pores and the growth of Cutibacterium acnes. The use of chemicals such as benzoyl peroxide and azelaic acid often causes side effects such as irritation and dry skin, so a safer and more effective natural alternative is needed. This study aims to develop a peel-off gel mask preparation containing a combination of activated charcoal from palm kernel shells and tea tree oil as a natural antibacterial against Cutibacterium acnes. Activated charcoal was obtained through a carbonization process at 350°C for 2 hours and sieved through 200 mesh, while tea tree oil was analyzed using GC–MS to determine the main content of terpinen-4-ol. Gel formulations were made with varying concentrations of tea tree oil (0.1–0.4%) and tested for physical properties, stability, pH, homogeneity, absorption, and antibacterial activity using the disk diffusion method. The results showed that the formula with a concentration of 0.4% tea tree oil had a pH of 6.17, a viscosity of 10,000 cps, a drying time of 18.6 minutes, an iodine absorption capacity of 786.78 mg/g, and the largest inhibition zone of ±11 mm against Cutibacterium acnes without causing irritation. Based on these results, the combination of activated charcoal from palm shells and tea tree oil is effective as a natural active ingredient for a safe anti-acne peel-off gel mask preparation.

KEYWORDS: acne, activated charcoal, tea tree oil, antibacterial

I. INTRODUCTION

Acne (acne vulgaris) remains a major skin health concern, especially in adolescents and young adults. It is caused by excess sebum production and the growth of Cutibacterium acnes (Utari et al., 2024). While chemical agents such as benzoyl peroxide and azelaic acid have been shown to be effective, they often cause side effects such as irritation and dry skin (Otlewska et al., 2020), necessitating safer and more effective natural alternatives. Various natural ingredients, such as aloe vera, gotu kola (Centella asiatica), and honey, have been studied, but most have only anti-inflammatory effects without strong antibacterial activity.

Activated charcoal from palm kernel shells has potential as a natural ingredient due to its high surface area and porosity, capable of absorbing oil, dirt, and acne-causing microorganisms (Osman et al., 2016). Its effectiveness can be enhanced by the addition of tea tree oil, which contains the active compound terpinen-4-ol, which has antibacterial, antiseptic, and anti-inflammatory properties against Cutibacterium acnes. Although these two ingredients have been studied separately, no study has combined them in a stable and effective topical formulation (Nascimento et al., 2023).

This research gap prompted the development of a peel-off gel mask formulation containing a combination of activated charcoal from palm kernel shells and tea tree oil. This peel-off gel mask was chosen because it forms a thin layer on the skin, allowing the active ingredients to adhere longer and work more effectively (Aprianti et al., 2024). This study aimed to develop and evaluate the physical characteristics, stability, and antibacterial activity of the peel-off gel mask against Cutibacterium acnes as an effective, safe, and natural-based alternative acne treatment.

II. METHOD

Time and Place of Research

The research was conducted at the Teaching Factory Laboratory, Vocational School, Diponegoro University, over a four-month period.

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Research Materials

The research materials used were palm kernel shells as a source of activated charcoal, tea tree oil, and additional materials such as polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP), propylene glycol, methylparaben, propylparaben, 96% ethanol, distilled water, iodine solution, potassium iodide (KI), sodium thiosulfate, methylene blue solution, Cutibacterium acnes, nutrient agar, and paper disks.

Research Tools

The tools used in this study included a furnace, oven, desiccator, grinder, 200-mesh sieve, pH meter, viscometer, measuring cylinder, dropper, cursive cup, magnetic stirrer, volumetric flask, UV-vis spectrophotometer, cuvette, burette, Erlenmeyer flask, glass stirrer, analytical balance, watch glass, beaker, measuring cylinder, and hotplate.

Conclusion of Research Results

This study produced the best gel formula based on activated charcoal from palm kernel shells and tea tree oil through in vitro and in vivo tests according to SNI 01-2346-2006 and SNl 16-4399-1996 standards. Data were analyzed using descriptive statistics and one-way ANOVA ($\alpha = 0.05$) through stability, antibacterial, homogeneity, organoleptic, skin irritation, pH, viscosity, spreadability, drying time, and adhesion tests to assess differences between formulas. The results showed one formula was the most effective, stable, and safe to use.

Sample Preparation

Palm oil shell samples were obtained from PT. Bakrie Pasaman Plantations (Sungai Aur Estate), West Sumatra. The shells were separated from kernels and other impurities, and after cleaning, they were dried in the sun to reduce their water content.

Activated Charcoal Production

The dried palm shells were then carbonized in a furnace at 350°C for 2 hours to form charcoal. The charcoal was then cooled, ground using a grinder into charcoal powder, and then sieved using a 200-mesh sieve to homogenize the activated charcoal particles. X-ray diffraction (XRD) analysis was performed to identify particle uniformity. The charcoal yield was then calculated (Limbong et al., 2021).

Yield formula:

$$\text{Yield} = \frac{\text{Extra weight of activated charcoal}}{\text{Amount of activated charcoal}} \times 100\%$$

Production of Activated Charcoal by Physical Activation

The charcoal powder was physically activated by heating in a furnace at a temperature of 750 ˚C for 3 hours, then the yield was calculated from the ratio of the final charcoal weight to the initial weight (Limbong et al., 2021).

Qualitative Adsorption Test of Activated Charcoal with Methylene Blue

The adsorption capacity test was conducted qualitatively and quantitatively to assess the adsorption capacity of activated charcoal. Qualitatively, 0.1 gram of activated charcoal was mixed with 20 ml of 100 ppm methylene blue solution, stirred for 15 minutes at 100 rpm, allowed to stand for 30 minutes, and then filtered to observe color changes (Limbong et al., 2021). Quantitatively, the absorbance value of the activated charcoal was measured using a UV-Vis spectrophotometer.

Quantitative Iodine Absorption Test of Activated Charcoal

A total of 0.25 grams of activated charcoal powder was reacted with a 0.1 N iodine solution. Then stirred using a magnetic stirrer for 15 minutes and then filtered. 10 ml of the filtrate was titrated with a 0.1 N Sodium Thiosulfate ($Na_2S_2O_3$) solution until it turned light yellow, then added 2 drops of a 1% starch solution and titrated until the color disappeared. A good iodine absorption value according to SNI 06-3730-1995 is at least 750 mg/g (Limbong et al., 2021).

Peel-Off Gel Mask Formulation from Activated Charcoal from Palm Kernel Shells

a. Gas Chromatography–Mass Spectrometry (GC–MS) Analysis of Tea Tree Oil

This test aims to ensure the purity of the material and identify the main active compound, namely terpinen-4-ol in tea tree oil according to ISO 4730:2017 standards before being used in the formulation process.

b. Preparation of Peel-Off Gel Mask Preparations Using Activated Charcoal from Palm Kernel Shell

The composition of the ingredients used in each formula is shown in Table 1.

Table 1 Peel-Off Gel Mask Preparation Formulation

Ingredients	Concentration (%)	Function
Activated charcoal	1.5	Cleaning agent from particles or residue
PVA	15	Gelling agent
PVP	5	Softener or plasticizer
Propylene glycol	10	Moisturizing agent
Methyl Paraben	0.2	Preservative
Propyl Paraben	0.1	Preservative
Ethanol	15	Solvent for active ingredients
Aquadest	Ad 100	Solvent for active ingredients

Variations in tea tree oil concentration as the independent variable are presented in Table 2.

Table 2 Tea Tree Oil Concentration

Ingredients	Formulation 1	Formulation 2	Formulation 3	Formulation 4
Tea Tree Oil	0,1 %	0,2 %	0,3 %	0,4 %

The mask formulation begins by dissolving PVA in warm distilled water at 80˚C (Mass A), PVP dissolved in cold distilled water (Mass B), Methyl paraben and propyl paraben in propylene glycol (Mass C). After that, Mass C and Mass B were added to Mass A and stirred until homogeneous, then added activated charcoal powder, 96% ethanol, and tea tree oil with varying concentrations (0.1%, 0.2%, 0.3%, and 0.4%). The preparation was stirred until it formed a stable peel-off gel (Limbong et al., 2021).

In-vitro Test of the Effectiveness of Peel-Off Gel Masks as Antibacterials and Acne Treatment

a. **Antibacterial Testing with Agar Media**

Antibacterial testing was conducted using the disk diffusion method to assess the mask's ability to inhibit Cutibacterium acnes. After 24 hours of incubation at 35-37°C, the zone of inhibition around the disk was observed to determine the most effective concentration of tea tree oil before in vivo testing (Putri & Muflihah, 2024).

b. **Formula Stability Test**

The gel samples were stored at 4°C for 24 hours, then placed in an oven at 40°C for 24 hours as one cycle. Six cycles of testing were performed to observe physical changes and assess the stability of the preparation (Navarro-Pérez et al., 2021).

Physical Characteristics Test

a. **Homogeneity Test**

This is done by applying 0.1 gram of the preparation to transparent glass, then observing whether there are any parts that are not mixed well (Muflihunna et al., 2019).

b. **pH Test**

pH testing is performed by dipping a pH meter into the sample. According to SNI 16-4399-1996, the pH of leather products ranges from 4.5–8.0.

c. **Viscosity Testing**

The viscosity of the samples was measured using a Brookfield viscometer. According to SNI 16-4399-1996, a good gel viscosity ranges from 2,000–50,000 cps.

d. **Spreadability Test**

A total of 0.5 g of gel was placed on glass-coated graph paper, left for 15 seconds, then given a load of up to 150 g. The ideal distribution diameter for a peel-off gel mask is 3-5 cm (Limbong et al., 2021).

e. **Rapid Drying Time Test**

One gram of gel was applied to a glass (7 x 7 cm) and the drying time was recorded. The ideal peel-off gel mask dries in 15–30 minutes. (Saputra et al., 2019).

f. Adhesion Testing

A 1 gram of gel is pressed between two glass slides for 5 minutes with a 1 kg load. After the load is removed, the time until the slides separate is recorded. The ideal adhesion strength should not be less than 0.07 minutes or preferably more than 1 second (Limbong et al., 2021).

Organoleptic Testing

An examination of the smell, shape and color is carried out which involves observing the human senses regarding the physical properties of the preparation based on SNI 01-2346-2006.

In vivo Effectiveness Test of Peel-Off Gel Masks as Antibacterial and Acne Care

a. Skin Irritation Test

The preparation was applied to the skin for 24 hours. After removal, reactions such as heat, itching, or stinging were observed at 0, 24, 48, and 72 hours (Laras et al., 2014).

III. RESULTS AND DISCUSSION

Activated Charcoal Production

The carbonization process yields a dense, black charcoal with a dense texture. A yield of 34.54% indicates that the activated charcoal has optimal porosity without excessive mass loss. The charcoal is then cooled, ground, and sieved using a 200-mesh sieve to obtain uniform particle size. The following image shows the results of the X-Ray Diffraction (XRD) test on the activated charcoal particles used in this study.

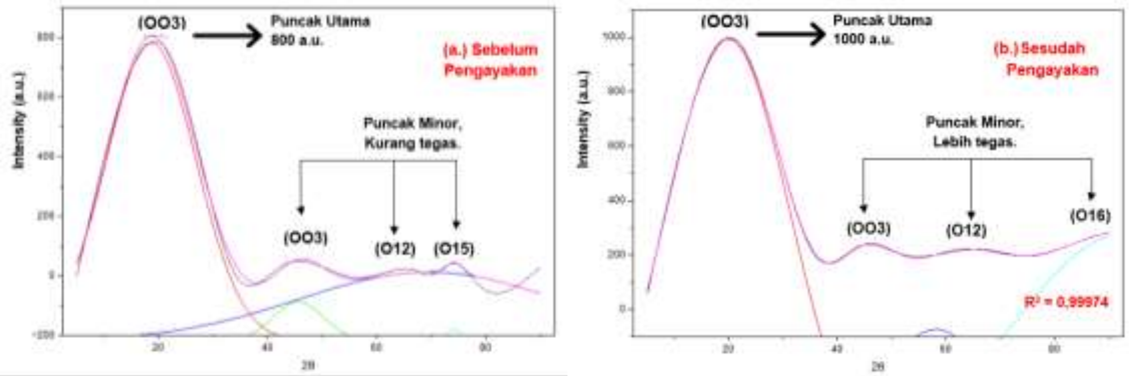

Figure 1 X-ray diffraction (XRD) graph of palm kernel shell charcoal: (a) before sieving and (b) after sieving.

The XRD results show that the sieving process improves the quality and sharpness of the diffraction pattern. Before sieving (Figure 1(a)), the intensity of the main peak (003) was only 800 a.u. with less clear minor peaks, indicating that the particles were not yet uniform. After sieving (Figure 1(b)), the peak intensity increased to 1000 a.u. and the R^2 value reached 0.99974, indicating a more regular crystal structure as well as better particle uniformity and clearer minor phases (Luo, 2022).

Activated Charcoal Production by Physical Activation

The activation process increases the surface area and porosity of the charcoal by breaking down hydrocarbon bonds, making it lighter and better able to absorb impurities. The resulting activated charcoal yield was 50.8%, indicating a conversion efficiency of approximately half the initial mass of palm kernel shells (Rangari, 2017).

Qualitative Test of Activated Charcoal Adsorption with Methylene Blue

The test results showed that activated charcoal was able to effectively absorb methylene blue. Before filtration, the solution was deep blue, while after filtration the color became paler, indicating the adsorption process by activated charcoal (Limbong et al., 2021). The absorbance value of 0.0902 from the UV-Vis spectrophotometer test showed a significant decrease in methylene blue concentration, indicating that activated charcoal has good adsorption capacity due to its optimal surface area and pores.

Quantitative Iodine Adsorption Test of Activated Charcoal

The iodine absorption test aims to determine the ability of activated charcoal to adsorb dissolved substances (Limbong et al., 2021). The iodine absorption value obtained was 786.78 mg/g, which has met the quality standards for activated charcoal based on SNI 06-3730-1995, which is a minimum of 750 mg/g. This proves that activated charcoal has a good surface area and porosity, so it is able to adsorb iodine molecules effectively and demonstrates optimal activated charcoal quality.

Formulation of Gel Preparation Based on a Combination of Activated Palm Shell Charcoal and Tea Tree Oil as an Antibacterial for Cutibacterium Acnes

Palm Shell Charcoal Peel-Off Gel Mask Formulation with Tea Tree Oil

a. Gas Chromatography–Mass Spectrometry (GC–MS) Analysis of Tea Tree Oil

The results of this test are intended to ensure the purity and primary content of the active compounds in the material before it is used in the formulation process. The results of this analysis are presented in the following figure.

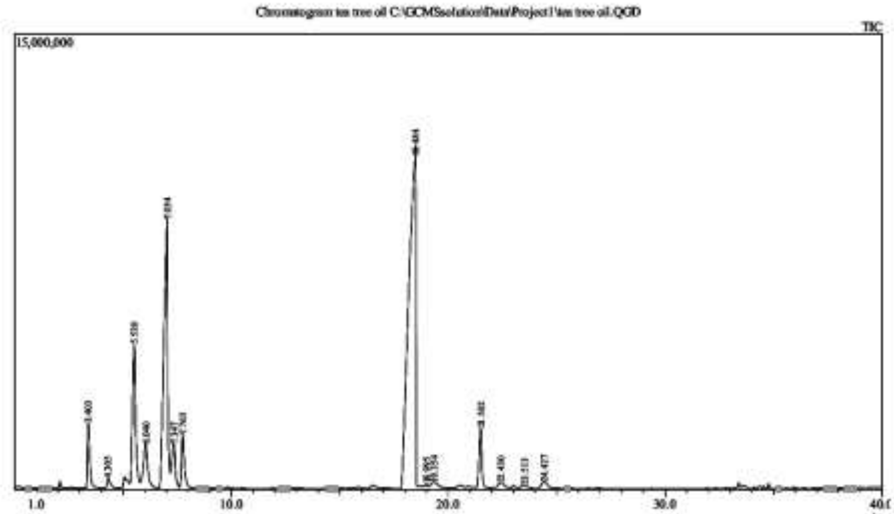

Figure 2 Chromatogram of the GC–MS analysis results of the sample

Based on the GC-MS analysis results in the image above, the main compounds in tea tree oil consist of 4-Terpineol (49.44%), γ-Terpinene (21.31%), α-Terpinene (9.21%), 1,8-Cineole (3.62%), and α-Pinene (3.03%). It can be concluded that the tea tree oil sample meets the ISO 4730:2017 standard because it contains high 4-Terpineol (>35%) as the main antimicrobial component and low 1,8-Cineole (<15%) which minimizes the potential for skin irritation.

b. Preparation of Peel-Off Gel Mask Preparation Using Activated Charcoal from Palm Kernel Shell

The gel mask was formulated by gradually mixing Masses C, B, and A to prevent clumping. Activated charcoal and 96% ethanol were added as solvents, while tea tree oil was added last as an antibacterial agent. The result was a homogeneous and stable gel for testing.

Evaluation of Peel-Off Gel Mask Preparation

In vitro Test of Peel-Off Gel Mask Effectiveness as an Antibacterial and Acne Treatment

a. Antibacterial Testing with Agar Media

Antibacterial activity testing was conducted using the disk diffusion method on blood agar plates suitable for the growth of Cutibacterium acnes. This test aimed to determine the ability of each mask formula to inhibit bacterial growth. The results are presented in Table 3 below. The results show that increasing the concentration of tea tree oil increases bacterial inhibition. Formulas 1 and 2 had low activity, Formula 3 showed an increase, and Formula 4 produced the largest inhibition zone (±11 mm) with the cleanest area, indicating the strongest antibacterial activity (Putri & Muflihah, 2024).

Table 3 Mask Test with Agar Media

Formula	Inhibitory Power (mm)	Visual Description
F1	± 7 mm	Colonies are still dense, the clear zone is barely visible, slightly dim around the disk.
F2	± 8 mm	There is a thin zone of inhibition, but there are still quite a lot of colonies at the edge of the zone.
F3	± 9 mm	The zone of inhibition is more visible than F1-F2, with fewer colonies.
F4	± 11 mm	The zone of inhibition is clear, almost clear around the disc, with significantly fewer colonies.

b. Formula Stability Test

Stability testing using the cycling test method at temperatures of 4°C and 40°C for six cycles showed that up to the fourth cycle there were no physical changes or homogeneity, so that the preparation was declared stable against temperature fluctuations and met cosmetic quality standards (Navarro-Pérez et al., 2021).

Physical Characteristics Test

a. Homogeneity Test

The homogeneity test aims to ensure that the peel-off gel mask mixture is evenly dispersed without lumps or coarse grains, indicating the uniformity of the preparation (Muflihunna et al., 2019). The results of the homogeneity test for each formula are presented in Table 4 below.

Table 4 Homogeneity Test Results

Formula	Average	Test Results
F1	2.0	Not Homogeneous
F2	2.6	Slightly Homogeneous
F3	3.2	Homogeneous
F4	3.2	Homogeneous

Test results showed that F3 and F4 were homogeneous, indicating an even distribution of the active ingredients. Homogeneity is essential to ensure consistent performance and quality of the preparation.

b. pH Testing

A pH test was conducted to ensure the peel-off gel mask had the right acidity level for skin (4.5–6.5) for safe use. A pH below 4.5 can cause irritation, while a pH above 6.5 can cause flaking. The pH measurement results for each formula are presented in Table 5 below.

Table 5 Results of Replication of pH Tests for Mask Preparations

Formula	pH Test Results			Mean ±SD
	R1	R2	R3	
F1	5,86	5,82	5,88	5,85±0,03
F2	6,84	6,4	6,8	6,79±0,05
F3	5,92	5,88	6,12	5,9±0,02
F4	6,18	6,12	6,2	6,17±0,05

The data obtained showed that the pH of all formulations ranged from 5.86 to 6.84. The one-way ANOVA test produced an F count of 325.3897 > F crit 4.066 and a P-value of $1.07\times10^{-8} < 0.05$, indicating a significant difference between the formulas. Formulation variations significantly affected pH, with formula 2 having the highest pH and formula 3 the lowest.

c. Viscosity Testing

Based on the SNI 16-4399-1996 standard, the appropriate viscosity for gel preparations is in the range of 2000–50000 cps. The viscosity measurement results for each formula can be seen in Table 6 below.

Table 6 Results of the Viscosity Test Replication of Mask Preparations

Formula	Viscosity Test Results			Mean ± SD
	R1	R2	R3	
F1	16200	15800	16000	16.000±163,2
F2	14100	13900	14000	14000±81,64
F3	12100	12000	11900	12000±81,64
F4	10100	9900	10000	10000±81,64

Based on the test results, all formulas have viscosity within the SNI standard range (10000–16000 cps), indicating good gel consistency. One-way ANOVA analysis showed Fcount (1142.86) > Fcrit (4.066) and P-value (7.2×10^{-11}) < 0.05, so there are significant differences between formulas. Viscosity decreases with increasing tea tree oil concentration due to its solvent effect, but all formulas still meet the ideal criteria with a gel texture that is easy to spread. Formula 1 is the thickest (16000cps), while formula 4 is the thinnest (10000cps).

d. Spread Power Test

A spreadability test was conducted to determine the peel-off gel mask's ability to spread across the skin's surface, which affects comfort during use. The test results are shown in Table 7.

Table 7 Results of the Replication of the Mask Spreadability Test

Formula	Spread Power Test Results (cm)			Mean ± SD
	R1	R2	R3	
F1	0,3	0,29	0,32	0,3±0,01
F2	0,5	0,48	0,52	0,5±0,02
F3	0,38	0,4	0,42	0,4±0,02
F4	0,9	0,89	0,91	0,9±0,01

One-way ANOVA analysis obtained F count 727.0882 > F crit 4.066 and P-value $4.38 \times 10^{-10} < 0.05$, there is a significant difference between the formulas. The spreadability test shows that Formulas 1–4 range from 0.3–0.9 cm, still below the ideal standard of 3–5 cm, so the preparation does not have optimal spreadability (Limbong et al., 2021) Formula 4 has the best spreadability.

e. Quick Drying Time Test

A drying time test was conducted to assess how quickly the peel-off gel mask formed a film after application. The test results are presented in Table 8.

Table 8 Drying Time Replication Results

Formula	Quick test results of drilling time (minutes)			Mean ± SD
	R1	R2	R3	
F1	28,5	27,8	29,0	28,4±0,6
F2	24,3	25,0	24,8	24,7 ±0,36
F3	19,6	20,1	19,8	19,83 ±0,25
F4	16,2	15,8	16,0	18,6 ±0,98

Based on the one-way ANOVA analysis, the calculated F was 159.96 > Fcrit 4.066 and the P-value was $1.8 \times 10^{-7} < 0.05$, indicating a significant difference between the formulas. The drying time ranged from 18.6 to 28.4 minutes, still within the ideal range of 15–30 minutes, with Formula 4 showing the fastest drying time (Saputra et al., 2019).

f. Adhesion Test

The adhesion test was conducted to assess the mask's ability to adhere to the skin, which affects its effectiveness and comfort. The results for each formula are presented in Table 9. Based on the one-way ANOVA (F count 29916.5 > F crit 4.066; P-value $1.6 \times 10^{-16} < 0.05$), there were significant differences between the formulas. Formula 1 had the highest adhesion, while formula 4 had the lowest. However, all formulas met the minimum standard of ≥4 seconds (Limbong et al., 2021).

Table 9 Results of Replication of Mask Adhesion Test

Formula	Adhesion Test Results (seconds)			Mean ± SD
	R1	R2	R3	
F1	8,09	8,1	4,32	8,09 ±0,008
F2	6,82	6,8	6,85	6,82 ±0,02
F3	4,32	4,3	4,35	4,32 ±0,02
F4	3,23	3,2	3,25	3,22 ±0,02

Organoleptic Testing

Organoleptic testing was conducted to assess the physical appearance of the peel-off gel mask preparations, including color, odor, and texture. The results of observations for each formula can be seen in Table 10 below.

Table 10 Organoleptic Test Results

Formula	Color	Odor	Texture
F1	Thick Black	Distinctive	Soft
F2	Thick Black	Soft	Thick
F3	Grayish Black	Strong	Sticky
F4	Grayish Black	Strong	Soft

Organoleptic test results showed that all formulas had good physical characteristics and were easy to apply. Formulas 1 and 2 were jet black with a distinctive aroma and a soft to thick texture, while Formulas 3 and 4 were grayish with a stronger aroma and a stickier texture due to the higher concentration of tea tree oil (Wulansari et al., 2024).

In vivo Test of the Effectiveness of Peel-Off Gel Masks as Antibacterial and Acne Treatment

a. Skin Irritation Test

Testing of peel-off gel masks was carried out to observe redness (erythema) and swelling (edema) to assess irritation. The results of the irritation test on volunteers can be seen in Table 11. The irritation test results indicate that all peel-off gel mask formulas are safe to use. Testing on eight panelists only observed mild redness (erythema) at 24 hours, no swelling (edema), and no symptoms of irritation at 48–72 hours (Laras et al., 2014). Therefore, formulations 1–4 are not irritating and prove that the concentration of tea tree oil used is within safe limits.

Table 11 Irritation Test Results

Volunteers	24 Hours		48 Hours		72 Hours	
	Erythema	Edema	Erythema	Edema	Erythema	Edema
1	1	0	0	0	0	0
2	1	0	0	0	0	0
3	1	0	0	0	0	0
4	2	0	0	0	0	0
5	0	0	0	0	0	0
6	1	0	0	0	0	0
7	0	0	0	0	0	0
8	0	0	0	0	0	0

IV. CONCLUSION

Based on the test results, it can be concluded that formula 4, with a tea tree oil concentration of 0.4%, performed best compared to the other formulas. This formula had the largest inhibition zone of 11 mm, indicating the strongest antibacterial activity. Its pH remained within the safe skin range of 6.17, and its drying time was fast, at approximately 18 minutes. Its homogeneity and viscosity also met the ideal characteristics of a peel-off gel preparation. However, its spreadability was still relatively low, requiring optimization of the gelling agent composition to achieve optimal results.

Overall, this research successfully produced a peel-off gel mask combining palm shell activated charcoal and tea tree oil that is stable, safe, and has effective antibacterial activity against the causes of acne.

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