

Fomulation And Physical Quality Testing Of A Scrub Gel With Ethanol (70%) Extract Of Pineapple Core (*Ananas comosus (L.) Merr.*)

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ABSTRACT

Scrubs are widely used in cosmetic care, primarily for exfoliation to remove dead skin cells. This study used a 70% ethanol extract from pineapple cores, which contains active compounds such as flavonoids and saponins. This research employed a true experimental method with a post-test only design to determine the best physical quality of scrub gel formulations containing pineapple core extract (*Ananas comosus (L.) Merr.*) at three extract concentrations: 3%, 5%, and 10%. However, the pH testing results for the three formulations did not meet the standards, as they were relatively neutral to slightly acidic (6.48-7.58). The hedonic test results indicated that Formulation 2 (5%) received the highest ratings in terms of aroma and texture, while Formulation 1 (3%) and Formulation 3 (10%) also showed a tendency to be preferred in certain aspects, particularly in terms of color and texture. The One-Way ANOVA test on spreadability showed a very significant difference in spreadability, with Formulation 3 and the negative control having the highest spreadability (p-value <0.005). The concentration of pineapple core extract affects the physical and sensory characteristics of the scrub gel, with the addition of active pineapple core extract at a concentration starting from 3% already showing good physical characteristics.

Keywords : Scrub Gel, Pineapple Core, Bromelain, Exfoliation, Physical Quality Testing.

INTRODUCTION

The skin is the outermost part of the human body. It has a complex structure and varies according to climate, age, gender, race, and its location on the body (Wibawa and Winaya, 2019). Generally, the process of skin cell regeneration occurs naturally and lasts for 21-24 days (Rahmawaty, 2020). However, this process can be hindered due to stress, hormonal changes, temperature fluctuations, seasonal changes, and aging (Ibrahim *et al.*, 2022). The accumulation of dead skin cells is a skin issue that can lead to a dull complexion, damage to the skin barrier, clogged pores, acne, and other poor skin conditions (National Skin Cancer Centres, 2021). Therefore, there is a need for cosmetic products that function to exfoliate or remove the accumulated dead skin cells (exfoliating, defoliating) (Rahmawaty, 2020). According to the National Agency of Drug and Food Control (BPOM) in 2015, several cosmetics circulating in the market contain chemical substances, including mercury, more than 2% hydroquinone, retinoic acid, diethylene glycol, colorants such as rhodamine B, and red K3, as well as chlorofluorocarbons. As a solution to this issue, using natural active ingredients is recommended, known as green product cosmetics.

Scrubs are one of the cosmetic treatments used to cleanse and renew skin cells (Rahmawaty, 2020). According to the study "Utilization of Natural Waste Potential as an Active Ingredient for Body Scrub Formulations" in 2022, various natural ingredients derived from plants can be used as active ingredients in scrubs due to their phytochemical content; plant enzymes like papain and

bromelain can also be used (Nisa *et al.*, 2022). Pineapple is one such plant whose enzyme content can be used for this purpose. Research by Arlian, 2021, and Fitri *et al.*, 2023, found that pineapple cores contain flavonoids, alkaloids, saponins, terpenoids, citric acid, malic acid, oxalic acid, and the enzyme bromelain. As noted by Fitri *et al.*, 2023, bromelain has antibacterial, anti-inflammatory, and antioxidant properties (Thomas *et al.*, 2023). The ananasic and citric acids contained in it can soften and refresh the skin, while bromelain not only helps prevent swelling and itching but also aids in exfoliating dead skin cells, making the skin appear smoother and brighter (Australian Government, 2008).

Bromelain helps with exfoliation by breaking down proteins on the skin surface, removing dead skin cells, and allowing for the regeneration of new skin cells. Additionally, bromelain has anti-inflammatory properties by reducing the production of prostaglandin E2 (PGE-2) and the enzyme cyclooxygenase-2 (COX-2), which accelerates skin healing and regeneration (Schulz *et al.*, 2018; Arshad *et al.*, 2014). Bromelain content in pineapples is most abundant in the pineapple core at 80%, compared to 10% in the pineapple fruit itself (Tara, 2019). The crude extract of bromelain enzyme from pineapple core has an activity unit of 5.373 U/mL (Sagita *et al.*, 2023). The aim of this study is to develop a conventional natural scrub gel cosmetic preparation from pineapple core extract (*Ananas comosus* (L.) Merr.) to help remove dead skin cells and to determine the best formulation of the scrub gel with the optimal concentration of pineapple core extract (*Ananas comosus* (L.) Merr.) based on physical quality tests (organoleptic properties, homogeneity, pH, viscosity, adhesion, and spreadability).

METHODS

This research is an experimental study using a true experimental design with a post-test only experiment approach, aimed at obtaining the best physical quality test results for the scrub gel preparation made from pineapple core extract (*Ananas comosus* (L.) Merr). The research includes several stages, such as the extraction process, formulation development, and physical quality testing (organoleptic properties, homogeneity, pH, viscosity, adhesion, and spreadability).

A. Preparation of Thick Pineapple Core Extract

The pineapple core is separated from the fruit, chopped, and cleaned with running water. After being naturally dried, the core is further chopped into smaller pieces and weighed as fresh *simplicia*. The core is then air-dried for 3-5 days and subsequently dried in an oven at 40°C. The dried *simplicia* is sorted, weighed, ground, and stored in a tightly sealed container. For maceration, 787.3 grams of dried *simplicia* are prepared at a ratio of 1:10. The *simplicia* is placed in a clean container and soaked with ethanol until fully submerged. The container is tightly sealed and left for 3 days, with occasional stirring. After maceration, the mixture is filtered to separate the extract liquid from the residue, and then the liquid is processed with a rotary evaporator to reduce its volume. The extract is further concentrated in a water bath, and its weight is measured for further evaluation, including yield calculation and ethanol-free testing.

B. Phytochemical Screening

Phytochemical screening using the test tube method is conducted as a preliminary study to identify flavonoids, alkaloids, saponins, tannins, steroids, and terpenoids. For flavonoids, the extract is heated with ethanol, magnesium, and HCl, resulting in an orange-red color (Forestryana and Arnida, 2020). Alkaloids are identified using dragendorff, wagner, or bouchardat reagents, producing a reddish-brown or orange precipitate (Forestryana and Arnida, 2020; Sulasmi *et al.*, 2018). Saponins are tested by shaking the extract with distilled water, and stable foam indicates the presence of saponins. Tannins are tested with FeCl₃, which turns the color to blue-black or green. Steroids are identified with anhydrous

acetic acid and concentrated H_2SO_4 , producing a blue or green color. Terpenoids are tested with glacial CH_3COOH and concentrated H_2SO_4 , producing a red color (Novia, 2020).

Thin-layer chromatography (TLC) is used with different eluents depending on the type of active compound. The pineapple core extract is applied on a silica G60F254 plate, eluted with a specific eluent, and illuminated under UV light at 254 nm to observe the spots. Each group of compounds uses a different mobile phase mixture and is tested in a saturated chamber (Rahman *et al.*, 2017).

- Flavonoids: Ethanol and water (5:5), quercetin as a reference (Novia, 2020).
- Tannins: Chloroform, ethanol, and distilled water (20:1:5), catechin as a reference (Novia, 2020).
- Alkaloids: Ethyl acetate and n-hexane (5:5), piperine as a reference (Novia, 2020).
- Saponins: n-Butanol and water (5:5), rutin as a reference (Novia, 2020).
- Steroids/Terpenoids: n-Hexane and ethyl acetate (4:1), β -sitosterol as a reference (Novia, 2020).

C. Preparation of Pineapple Core Extract Scrub Gel

In the preparation of the pineapple core extract scrub gel, first, dissolve methyl paraben and propyl paraben in hot distilled water (40°C) until completely dissolved. Meanwhile, mix carbopol with distilled water until homogeneous. Then, add the preservative solution and propylene glycol to the gel base, followed by TEA until a clear and stable gel is formed. Finally, add the pineapple core extract and rice particles to the gel and stir until evenly mixed.

D. Formulations

Table 1. Formulation of Pineapple Core Extract Scrub Gel

E. Evaluation of Pineapple Core Scrub Gel

- Organoleptic Test

This test involves placing the gel on a glass slide and visually examining it to note the shape, aroma, and color (Firdausi, 2021).

- Homogeneity Test

The gel is spread between two glass slides and observed under a microscope to check for any unmixed particles or clumping (Firdausi, 2021).

- pH Test

This test is conducted by calibrating the pH meter using standard pH solutions, then immersing the electrode in the gel solution and recording the displayed pH value. The pH value for the scrub gel is 4.5 (Firdausi, 2021).

- Viscosity Test

Using a Brookfield viscometer, the preparation is placed in a beaker, and its viscosity is measured at a speed of 60 rpm. The viscosity value is determined based on the dial reading multiplied by the correction factor (Lestari *et al.*, 2022).

- Adhesion Test

A sample is placed between two glass slides and weighed down, then the time required for the slides to separate after applying a certain force is measured (Yuniarsih and Sari, 2021).

- Spreadability Test

The gel is measured and placed on a glass surface, then a weight is applied. After 1 minute, the spread diameter is recorded. A quality gel has a spreadability of 5-7 cm (Yuniarsih and Meilinda Sari, 2021).

- Hedonic Test

This test involves 20 panelists to evaluate their preference for the gel using a five-point hedonic scale (Qamariah *et al.*, 2022).

F. Data Analysis

Statistical data analysis was conducted by measuring the physical quality of the three gel formulations, using the Shapiro-Wilk normality test to determine if the data were normally distributed ($p > 0.05$). If the data were normal, a One-Way ANOVA was performed to compare the mean data between the three gel formulations, the negative control, and the positive control that met the homogeneity requirement based on the Levene's statistic, followed by a Post Hoc Tukey HSD for multiple comparisons. If the ANOVA assumptions were not met and the results showed a significant difference ($p < 0.05$), the analysis proceeded with the non-parametric Kruskal-Wallis test and post hoc Mann-Whitney test.

RESULTS

A. Extraction of Pineapple Core

The extraction of the simplicia was carried out using the maceration method with 70% ethanol in a ratio of 1:10 for 3 days, with stirring twice a day. The extract was then evaporated using a rotary evaporator at 45°C and concentrated using a water bath at 50°C, yielding a thick extract weighing 317.2 grams with a yield of 40.28%, which is considered good as it is more than 10% (Ministry of Health of the Republic of Indonesia, 2017).

Table 2. Pineapple Core Extraction Results

Type of Extract	Weight of Dry Simplicia	Weight of Extract	% Yield
Thick Extract	787,3 gr	317,2 gr	40,28%

An ethanol-free test was conducted by adding 1 mL of acetic acid and 1 mL of concentrated sulfuric acid to the test solution, followed by stirring and heating over a bunsen burner flame. No ester smell was detected during heating, indicating that the extract is ethanol-free (Klau *et al.*, 2021).

B. Phytochemical Screening

The results of the phytochemical screening using test tubes in table 3 show the phytochemical screening results for six types of compounds: flavonoids, saponins, tannins, alkaloids, steroids, and terpenoids. each compound was tested using specific reagents and has specific color or shape observation requirements. observations that match the requirements indicate the presence of the compound in the sample, with positive (+) or negative (-) conclusions listed in table 3. Phytochemical screening also serves as an initial validation that guides and confirms the TLC results, allowing for optimization of TLC conditions such as selecting appropriate mobile and stationary phases, thereby enhancing the resolution and efficiency of compound separation.






C. of Pineapple Core Extract Scrub Gel

• Organoleptic Test

gel formulations and the negative control. Differences in the results of this test were due to the varying concentrations of pineapple core extract used in each formulation. Formulation 1 produced a pale yellow, slightly clear color, with a mild pineapple scent and a thick gel texture. Formulation 2 showed a more intense yellow and clear color, a more noticeable pineapple smell, and a slightly runny gel texture. Formulation 3 resulted in a dark yellow color approaching brown, a stronger pineapple smell, and a more fluid gel texture. The negative control had a clear color, a characteristic TEA and carbopol odor, and a thick texture. The higher the concentration of pineapple core extract, the more intense the color and smell produced, and the gel texture becomes more fluid. Pineapple core extract contains active compounds such as ananasic acid, bromelain enzyme, and flavonoids that can affect the physical characteristics of the gel (Varilla *et al.*, 2021). The acids in the extract can lower the mixture's pH, which affects carbopol's ability to form a thick gel, so at higher extract concentrations, the gel becomes more fluid (Fissore *et al.*, 2023). Additionally, bromelain in the extract can break down proteins or polymers in the gel, altering its viscosity (Firdausi, 2021).

- *Homogeneity Test*

Table 6. Homogeneity Test Results

F I	F II	F III
		
(-) Control		(+) Control
		

The homogeneity observations of the scrub gel preparations from the three formulations and the negative control showed the presence of exfoliating particles in the form of rice, which had a uniform shape, as did the positive control, which had uniform particle shapes, as indicated in Table 6. A gel preparation is considered homogeneous if there are no distinct particles and there is uniformity in color (Sayuti, 2015). Based on the observations, it can be concluded that the prepared scrub gel is suitable in terms of homogeneity evaluation.

- *pH Test*

The pH testing of the three scrub gel formulations with varying concentrations of pineapple core extract showed different results, but in general, they did not meet the standard pH for scrubs because the pH of the preparations tended to be acidic, around 4.5 (Yuniarsih and Sari, 2021). Formulation 1 had an average neutral pH of 7.58, and the average pH of Formulation 2 was also near neutral at 7.05. Meanwhile, Formulation 3 had an average slightly acidic pH of 6.48. The neutral pH of Formulation 1 was due to the balance between acidic and basic components in the ingredients used (Chu *et al.*, 2021). The negative control, which did not contain active ingredients, showed an alkaline nature with a pH of 8.46, while the positive control had a very acidic pH of 2.38. Pineapple core extract contains active components that can impart acidity, but at low concentrations, this acidic effect is not dominant. Additionally, the use of 2% TEA (triethanolamine) functions as a pH balancer, as TEA is a basic compound that can neutralize acids in the formulation. This pH variation is caused by the different concentrations of pineapple core extract used in each formulation (Fissore *et al.*, 2023).

- *Viscosity Test*

The viscosity test results showed that the negative control had an average viscosity of 9905.3 cP, while Formulation 1 had an average viscosity of 9804.73 cP. Formulation 2 had a lower average viscosity of 9390.67 cP, and Formulation 3 showed the lowest average viscosity at 8762.73 cP. According to SNI 16-4380-1996, the required viscosity value for skin cleansing gel preparations is between 3,000 and 50,000 centipoise, indicating that the pineapple core extract scrub gel meets this standard (Yuniarsih and Sari, 2021). This data shows that the viscosity of the scrub gel decreases as the concentration of pineapple core extract increases from Formulation 1 to Formulation 3. This difference in viscosity is due to several factors, including the concentration of pineapple core extract used. Pineapple core extract

contains the enzyme bromelain, which can affect the structure and interactions between molecules in the gel. Increasing the extract concentration leads to higher enzymatic activity, which reduces the gel's thickness by breaking down bonds within the gel matrix (Varilla *et al.*, 2021). Additionally, acids in the pineapple core extract, such as ananasic acid, can lower the gel's pH, affecting the structure of carbopol, the gel-forming polymer. Carbopol swells optimally at neutral to slightly alkaline pH but becomes less effective at acidic pH, causing a decrease in viscosity (Indrawati and Zissakina, 2011; Agustiani *et al.*, 2022).

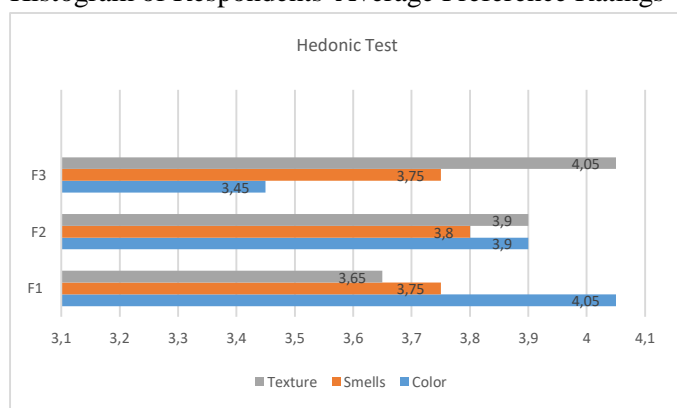
From the adhesion test results in Table 9, it can be seen that the higher the concentration of pineapple core extract in the formulation, the lower the gel's adhesion. The negative control, which did not use extract, had the highest adhesion with an average of 15.05 seconds. Formulation 1 showed the second highest adhesion with an average of 11.67 seconds, Formulation 2 had an adhesion of 7.18 seconds, while Formulation 3 had the lowest adhesion with an average of 5.15 seconds. It can be concluded that the adhesion of the three formulations meets the requirements, as good adhesion is considered to be not less than 4 seconds (Nurlily *et al.*, 2021). This is related to viscosity; the lower the viscosity, the greater the spreadability produced, as the gel is expected to spread well without requiring significant pressure, making it easy to apply (Silvia and Dewi, 2022).

• Spreadability Test

The spreadability test aims to measure how far the gel can spread when a certain amount of pressure is applied. Table 10 shows the spreadability measurement results from three repetitions for each different formulation. The negative control had the lowest spreadability with a test result of 3.5 cm. Formulation 1 had an average spreadability of 4.03 cm. Formulation 2 showed an increased spreadability with an average of 5.20 cm. Formulation 3 showed the highest spreadability with an average of 6.03 cm, the same as the positive control, which also had an average spreadability of 6.03 cm. This data indicates a positive correlation between viscosity, the increasing concentration of pineapple core extract, and the increase in gel spreadability. The spreadability of Formulations 2 and 3 meets the requirements for good spreadability, which is between 5-7 cm (Sayuti, 2015).

• Hedonic Test

Histogram of Respondents' Average Preference Ratings



Legend:

- 1: Dislike
- 2: Slightly dislike
- 3: Neutral
- 4: Like

5: Strongly like **DISCUSSION**

Respondents' evaluation of the three scrub gel formulations showed variations in aspects of color, aroma, and texture. Formulation 1 was generally liked, with an average rating of 4.2 for color, 4.3 for aroma, and 4.0 for texture. Formulation 2 also received positive ratings, with an average of 4.1 for color, 4.2 for aroma, and 4.3 for texture, making it the most preferred overall. Formulation 3 (FIII) had greater variation in ratings, with an average of 3.7 for color, but was still liked with an average of 4.0 for aroma and 4.2 for texture. Overall, Formulation 1 was considered the most favored by respondents based on the consistency of ratings across all parameters.

Data Analysis

The analysis of the data shows that formulation 1 consistently performed well across various tests. The post hoc Mann-Whitney tests for pH, viscosity, and adhesion indicated that formulation 1 and the positive control had the highest mean ranks, suggesting optimal characteristics in these parameters (p value $0,46-0,05 \leq 0,05$). The One-Way ANOVA test for spreadability showed significant differences among the groups (p value $<0,05$), with formulation 1 and the positive control demonstrating the lowest spreadability, while formulation 3 and the negative control had the highest. The Kruskal-Wallis test results for hedonic test between color, aroma, and texture revealed no significant differences among the groups, as all Asymp. Sig. values were above the 0.05 threshold. Overall, Formulation 1 exhibited optimal properties comparable to the positive control in most tests.

CONCLUSION

The study shows that the extraction of pineapple core yields a high percentage and contains various phytochemical compounds such as flavonoids, saponins, tannins, alkaloids, steroids, and terpenoids. The concentration variations of the extract affect the physical and sensory characteristics of the scrub gel, such as color, smell, texture, pH, viscosity, spreadability, and adhesion. The concentration of pineapple core extract affects the physical and sensory characteristics of the scrub gel, with the addition of active pineapple core extract at a concentration starting from 3% already showing good physical characteristics. Formulation II was the most preferred by respondents, although there were no significant differences in the hedonic test for color, aroma, and texture.

Further research is needed to optimize the formulation's pH, ensure long-term safety and stability, and evaluate the pharmacological effects of the pineapple core extract. Clinical and microbial testing is also recommended to assess the effectiveness, safety, and quality of the product. Additionally, the potential use of the extract in various other skincare products should be further explored.

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