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Article

The Formulation of a Body Scrub Combining Sweet Orange Peel (*Citrus x sinensis* L.) and Coconut Dregs (*Cocos nucifera* L.) as an Antioxidant

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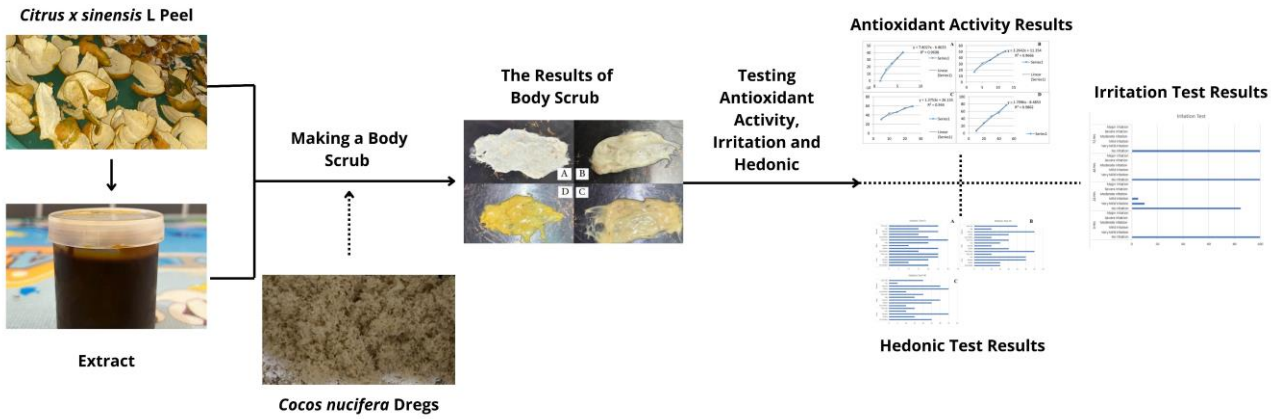
Abstract

The sweet orange peel (*Citrus x sinensis* L.) possesses antioxidant properties that can be harnessed for developing a pharmaceutical preparation based on natural ingredients, particularly in skin care products such as body scrubs. Meanwhile, coconut pulp (*Cocos nucifera* L.) has a textural quality that lends itself to use as a scrubbing agent, capable of removing dead skin cells. This study aimed to ascertain the antioxidant activity of body scrub formulations with varying concentrations of extract. The results of the phytochemical screening indicated the presence of alkaloids, flavonoids, tannins, and triterpenoids. Three formulations of body scrub were prepared, designated FI (1.25% extract), FII (2.5% extract), and FIII (5% extract). The results of the physical tests demonstrated that the organoleptic, pH, spreadability, and adhesion tests met the requisite standards. In the antioxidant activity test with the DPPH reagent, the IC50 value of each formula is as follows: The results demonstrated that the IC_{50} value of each formula was 7.28 ppm (very strong) for FI, 11.839 ppm (very strong) for FII, and 17.354 ppm (very strong) for FIII.

Keywords: *Antioxidant, body scrub, coconut pulp, sweet orange*

Graphical Abstract



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Introduction

The skin is the body's outermost layer, protecting against external influences, particularly on tissues and organs. Any damage to the skin will impact human aesthetics, health, and appearance [1]. The primary causes of skin damage are ultraviolet (UV) exposure and exposure to pollution containing free radicals [2]. Implementing particular skin care regimens is imperative to circumvent damage caused by free radical compounds from sun exposure and other factors [3]. Such processes may be facilitated through the use of cosmetic preparations.

Cosmetics are substances or mixtures applied to the external surfaces of the human body or the teeth and mucous membranes with the intention of cleansing, imparting a fragrant sensation, providing protection, or enhancing the appearance of specific body parts [4]. Based on research on literature, there has been a notable increase in awareness regarding the significance of skin health, which has led to a considerable rise in demand for cosmetic products, particularly those designed for skin care. One of the cosmetic dosage forms utilized in the domain of skin care is the body scrub. A body scrub is a liquid or semi-solid emulsion preparation that provides moisture, restores skin softness, and removes dead skin cell impurities caused by external exposure, such as free radicals from sunlight [5].

Developing body scrub dosage formulations using natural ingredients containing antioxidant compounds can be an alternative to prevent skin damage. Sweet orange peel (*Citrus sinensis* L.) contains flavonoid compounds with antioxidant, anti-inflammatory, and antibacterial properties. These activities avoid skin damage, such as inflammation and premature ageing [6]. Based on research conducted on sweet orange peel *(Citrus sinensis* L.), flavonoid content and antioxidant activity were found in it. The IC_{50} test results on sweet orange peel extract showed an IC_{50} value of 18.792 ppm, which showed antioxidant activity in the strong category. Other studies have shown that sweet orange peels also contain vitamin C. Therefore, sweet orange peel extract has the potential to be developed into cosmetic preparations [7].

Another natural ingredient that can be utilized in the formulation of body scrub preparations is coconut pulp (*Cocos nucifera* L.). Coconut pulp is a source of gluten-free protein that is low in fat and can moisturize the skin. The nutritional composition of coconut pulp includes 5.78% protein, 38.24% fat, and 15.07% crude fibre. The coarse grains in coconut pulp can be used as a scrub to remove dead skin cells [8].

Based on literature research, researchers are interested in developing the potential of orange peel and coconut pulp for several reasons. First, orange peels have high antioxidant activity based on their IC_{50} value. Second, high antioxidant activity can ward off free radicals, thus preventing damage to the skin. Third, the texture of coconut pulp, which tends to be rough, makes this material suitable as a scrubbing agent rather than synthetic materials, which are quite expensive, and no research discusses the combination of sweet orange peels and coconut pulp as a body scrub. This research is expected to reduce and manage organic waste in other forms and emphasize using natural ingredients with antioxidant and exfoliating benefits, which are the main points of body scrubs and help develop the concept of natural ingredient-based skin care products currently trending.

Material and Methods

Materials and Instrumentations

Sweet orange peel (*Citrus x sinensis* L.) collected from Citrus Distributor in Cempaka Putih, Jambi City, Indonesia. This study's coconut pulp (*Cocos nucifera* L.) was taken from the Santan Squeeze Shop in Kasang Pudak, Muaro Jambi Regency, Indonesia. The chemicals used were distilled water, 96% ethanol and methanol p.a (PT. Kisbiokim), Triethanolamine (PETRONAS), Cethyl Alcohol (Emercol®), Methyl Paraben and Propyl Paraben (Ueno®), Propylene Glycol (Hi-Tech Spring), Stearic Acid (PT. Wilmar), Oleum citri (PT. Kimia Jaya Labora). For reagents used are DPPH (Sigma Aldrich), Mayer (PT. Kimia Jaya Labora), Dragendorff (PT. Kimia Jaya Labora), Sulfuric Acid (PT. Kimia Jaya Labora), Anhydrous Acetic Acid (Mahavero Chems), $FeCl_3$ 1% (PT. Kimia Jaya Labora), NaOH 10% (PT. Kimia Jaya Labora) and

Chloroform (PT. Kimia Jaya Labora). Tools were prepared, such as beaker glass (Iwaki®), stirring rod, drop pipette, Sudip, mortar, stamper, porcelain cup, analytical balance (Pioneer™), water bath (Memmert®), rotary evaporator (Büchi Rotavapor R-114®), adhesion test kit, petri dish, weights (50 gs, 100 g and 1000 g), UV-Vis spectrophotometry.

Methods

Preparation of Citrus x sinensis Peel Extract. Sweet orange peels are taken from orange ice vendors or businesses that use sweet orange fruit. The sorting process involves criteria like smooth, shiny, and firm orange peels. After sorting, the peels were washed with running water to remove impurities. The orange peel waste obtained is thinly sliced and dried at 60^{o}C for 24 hr. The dried orange peels were pulverized using a blender [9], [10].

Preparation of Cocos nucifera Dregs. The coconut dregs (*Cocos nucifera*) simplisia was prepared by cleaning the coconut dregs from visible impurities. Then, the coconut pulp was washed thoroughly to remove the remnants of invisible impurities and other residues. After that, the sample is then roasted in a pan to dry while maintaining the colour of the coconut pulp, which is still white and not brownish.

Sweet Orange Peel Extraction and Phytochemical Screening. The extraction process was carried out using the maceration method and ethanol solvent, and dry simplisia orange peels that had been mashed were weighed as much as 500 g. The maceration process was carried out with the ratio between simplisia powder and solvent at 1:10 (%w/v). Simplisia was macerated for 4 x 24 hrs and routinely shaken once a day. After that, the results of maceration, called the macerate, are then filtered and evaporated with a rotary evaporator until a thick extract is obtained and phytochemical screening is carried out, which includes alkaloids, flavonoids, tannins, saponins, and triterpenoids [10].

Table 1. Formulas Body Scrub

Formulas	**Quantity % (w/w)**				**Functionality**
	K-	FI	FII	FIII	
Ethanol Extract of Orange Peel Sweet	-	1.25	2.5	5	Active ingredient
Coconut Dregs	4	4	4	4	Scrubbing agent
Rice Starch	6	6	6	6	Scrubbing agent
Stearic Acid	15	15	15	15	Emulsifier
Cethyl Alcohol	5	5	5	5	Base
Propilen Glikol	10	10	10	10	Humectant
Trietanolamin	3	3	3	3	Emulsifier
Gliserin	5	5	5	5	Humectant
Methyl Paraben	0.20	0.20	0.20	0.20	Preservative
Prophyl Paraben	0.02	0.02	0.02	0.02	Preservative
Oleum citri	1 mL	1 mL	1 mL	1 mL	Fragrance
Aquadest	Add 100	Add 100	Add 100	Add 100	Solvent

Preparation of the Body Scrub. The preparation of the body scrub entails the mixing of the oil phase and water phase in a heated mortar. The ingredients of the oil phase (stearic acid and cetyl alcohol) and the water phase (propylene glycol, propylparaben, methylparaben, triethanolamine,

and glycerin) were weighed. Subsequently, previously weighed, the oil and water phases are placed in separate porcelain dishes and heated with a water bath at a temperature of ±75°C until complete melting occurs. Once the oil phase has melted, it should be placed in a hot mortar, and the water phase should be added gradually until a homogeneous mixture is formed. Subsequently, the hot water should be added gradually until a cream phase is formed. Once the cream has been formed, it should be cooled and combined with rice starch and coconut pulp until a homogeneous mixture is achieved. Subsequently, the ethanol extract of orange peel should be added gradually until a homogeneous mixture is formed. Once the mixture has reached a homogeneous consistency, the *oleum citri* can be added [11].

Physical Quality Test of Body Scrub Preparation. The purpose of conducting physical quality testing of body scrub preparations is to ensure that the body scrub formula meets the specified requirements and is suitable for application to the skin. Physical quality tests were conducted, including organoleptic analysis, spreadability assessment, adhesion evaluation, and pH measurement. Organoleptic testing is carried out by looking at the body scrub in terms of colour, odour and texture and comparing it with other formulas. Spreadability testing is carried out by placing the preparation on a petri dish and covering it with a petri dish, then giving it a weight and measuring the spreading diameter. Adhesion testing is carried out by placing the preparation on an adhesion test tool and then calculating the time until the glass plate on the tool separates. pH measurements are carried out using a pH meter, where the body scrub is first diluted with distilled water, then the electrode is dipped to determine the pH of the body scrub made [12].

Determination of Antioxidant Activity. DPPH solution is prepared by weighing DPPH crystals and dissolved with methanol p.a in a measuring flask. In the determination, as much as 5 mg of DPPH was weighed with a solvent volume of 100 mL to obtain a DPPH solution with a concentration of 50 ppm. DPPH solution at a concentration of 50 ppm was read at a wavelength of 500 - 550 nm. The test sample used is the extract weighed as much as 100 mg, added methanol p.a as much as 100 mL, and then vortexed. From the stock solution, 10, 20, 30, 40, and 50 ppm concentrations were taken, and methanol was added until the limit mark. DPPH solution was pipetted as much as 4 mL and put into a volumetric flask. Then, the test solution in various concentration series that have been prepared is added to as much as 1 mL. The mixture was then allowed to stand for 30 min at 37°C, and the absorbance was read using UV-Vis Spectrophotometry with the maximum wavelength [13].

The percentage of antioxidant activity and value is calculated based on the ratio between the sample solution and its interaction with the DPPH reagent using equation 1.

$$\% \text{ Inhibition} = \frac{A_{Blanko} - A_{Sample}}{A_{Blanko}} \times 100\% \quad \text{.....(1)}$$

Notes:
A_{Blanko}= Absorbance Blanko
A_{Sample}= Sample Absorbance

Concurrently, the IC_{50} value is calculated through a linear regression analysis of the sample concentration concerning the percentage of inhibition. The following equation was derived:

$$y = ax + b \quad \text{.....(2)}$$

Antioxidant Activity of Body Scrub. In testing the antioxidant activity of body scrub, a 100 mg sample was weighed and then diluted with 100 mL methanol p.a. Then, the concentration value of the extract contained in the body scrub was calculated. Based on the calculation results, FI has a concentration variation of 1.25, 2.5, 3.75, 5, and 6.25 ppm. In FII, the concentration variations used in the test are 2.5, 5, 7.5, 10, and 12.5 ppm. For FIII, the concentration variations used were 5, 10, 15, 20, and 25 ppm. All concentrations were measured, and the per cent inhibition was calculated to determine the IC_{50} value.

Hedonic and Irritation Test. Hedonic testing uses panellists to assess the body scrub preparations based on aroma, colour, and texture suitability. The number of respondents who will be the

panellists in the hedonic test is 20 people. Panelists will give a score of 1-5, with the number 1 (one) being very disliked and 5 (five) meaning very like [14]. [14]. Meanwhile, the irritation test was carried out to determine the level of irritation caused when the preparation was applied to the skin. Tests were carried out for 0, 24, 48, and 72 hr, and the level of irritation was seen on a scale of 1-5, where number 1 (one) indicates severe irritation and number 5 (five) indicates no irritation [15].

Results and Discussions

Secondary Metabolites of Extracts

The extract obtained has a percentage of 27.114%. Based on the Indonesian Herbal Pharmacopoeia, a good extract has a percentage above 7.5%, which is declared to meet the requirements [16]. Phytochemical testing was carried out on the ethanol extract of sweet orange peel, and the results were obtained in the following Table 2.

Table 2. Phytochemical Screening Result of Ethanol Extract of Sweet Orange Peel

Secondary Metabolites	Reagent	Results
Alkaloids	Mayer	Positive
	Dragendorf	Positive
Flavonoids	H_2SO_4	Positive
	$FeCl_3$ 1%	Positive
Saponins	Aquadest	Negative
Tannin	$FeCl_3$ 1%	Negative
Triterpenoid	$CHCl_3 + C_4H_6O_3 + H_2SO_4$	Positive

Organoleptical of Body Scrub.

The parameters observed in the organoleptic test of body scrub preparations include the preparation's colour, aroma, and texture [14]. Based on colour, only FI is ivory white, while FII and FIII are yellow. This can occur due to different extract concentrations in the formula where the extract in FI is only 1.25% while FII and FIII use extracts as much as 2.5% and 5%. (Figure 1) Meanwhile, the negative control only has a white colour, and this is because there is no added extract to K-.

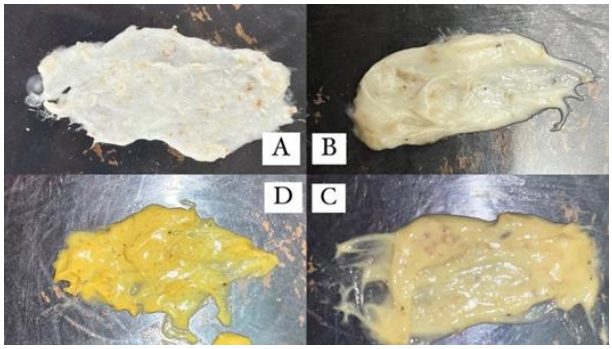

Figure 1. Results of Body Scrub Formulation: (a) K- body scrub (0%); (b) FI body scrub (1.25% Extract); (c) FII body scrub (2.5% Extract); (d) FIII body scrub (5% extract).

In the odour of body scrub, it was found that both K-, FI, FII, and FIII preparations had a distinctive smell, namely the fragrance of citrus. This is due to the addition of oleum citri, which gives the sensation of citrus fragrance and helps cover rancid odours due to the addition of coconut pulp. The texture of the three formulas is rough but not irritating to the skin. Indeed, body scrub preparations must have a rough texture because they remove dead skin cells. The bumpy texture is obtained from adding coconut pulp and rice starch

Table 3. Organoleptic Test Results of Body Scrub Preparations

Results	**Formulas**			
	K-	**FI**	**FII**	**FIII**
Colour	White	Ivory white	Yellow	Yellow
Odor	Unique	Unique	Unique	Unique
Texture	Rough	Rough	Rough	Rough

Table 4. Results of the Spreadability Test

Samples	Spreadability (cm)	Spreadability Standard (cm)
K-	5.00 ± 0.334	5.0-7.0 cm
FI	5.01 ± 0.311	
FII	4.96 ± 0.408	
FIII	5.01 ± 0.503	

Body Scrub Spreadability Test

The spreadability test on the preparation is intended to determine the ability of a preparation to spread on the skin's surface so that the active substances contained in the body scrub preparation can spread evenly. In this test, the value of good spreadability in body scrubs is 5.0-7.0 cm [17]. Based on the test results, it was found that FII did not meet the requirements with a spreadability value of 4.96 ± 0.408 cm. In contrast to FI, FIII, and K- which have a spreadability of 5.01 ± 0.311 cm, 5.01 ± 0.503 cm and 5.00 ± 0,334 cm. This is due to the consistency of the body scrub preparation, which is too dense, so more pressure is required to apply it.

Body Scrub Adhesion Test

The adhesion test of the preparation is carried out to determine the contact ability of a preparation, especially body scrub, to the skin, which is the place of application. Good body scrub adhesion is not less than 4 sec [5]. Based on the test results, the stickiness of FI scrub was obtained at 5.39 ± 0.342 sec. While FII was at 5.91 ± 0.095 sec, FIII was at 6.20 ± 0.121 sec and K- was at 5.51 ± 0.464 sec. The stickiness of the preparation was measured with the initial formula and its replication. Based on the test results, all preparations appear to meet the requirements above 4 sec.

Table 5. Adhesion Test Results

Samples	Adhesion Value (sec)	Standard Adhesion Value (sec)
K-	5.51 ± 0.464	> 4
FI	5.39 ± 0.342	
FII	5.91 ± 0.095	
FIII	6.20 ± 0.121	

Table 6. Results of pH Test of Preparations

Samples	pH ± SD	Body Scrub pH (Standard)
K-	7.07 ± 0.153	4.5-8.0
FI	6.98 ± 0.745	
FII	7.26 ± 0.276	
FIII	7.77 ± 0.186	

Body Scrub pH Test.

The pH test of the preparation is carried out to determine the level of acidity or basicity of a preparation that will affect the skin. Based on the standard set, SNI 16-4399-1996, the pH of a good body scrub preparation ranges from 4.5 to 8.0 [17]. Based on the pH test results of all formulas, the pH value of FI was 6.98 ± 0.745; FII was 7.26 ± 0.276; FIII was 7.77 ± 0.186 and K- was 7.07 ± 0.153. All formulas and their replications were measured for pH value, with formula replication done 3 (three) times [18].

Antioxidant Activity of Extract and Body Scrub

Initially, measurement was carried out towards blank absorbance. Absorbance measurements were carried out on DPPH blanks, which were repeated three times, known as triplo (Table 7). The average absorbance value of the DPPH blank was 0.852. This average value will be used to calculate each sample's inhibition percentage to be tested.

Table 7. Antioxidant Activity Test Results

Samples	Cons. (ppm)	Average Absorbance	IC_{50} (ppm)
FI	1.25	0.856	7.280
	2.5	0.717	
	3.75	0.642	
	5.0	0.577	
	6.25	0.511	
FII	2.5	0.710	11.839
	5.0	0.589	
	7.5	0.543	
	10.0	0.468	
	12.5	0.423	

Samples	Cons. (ppm)	Average Absorbance	IC_{50} (ppm)
FIII	5.0	0.595	17.354
	10.0	0.481	
	15.0	0.458	
	20.0	0.380	
	25.0	0.353	
Extract of *Citrus sp.*	10	0.801	34.210
	20	0.614	
	30	0.454	
	40	0.373	
	50	0.194	
Vitamin C (Compare)	10	0.587	33.410
	20	0.475	
	30	0.454	
	40	0.375	
	50	0.340	

Antioxidant activity determines a sample's ability to protect against free radicals. On the skin, free radicals will cause dullness, hyperpigmentation and premature ageing [19]. Antioxidant activity testing was carried out on two types of samples, namely extracts and body scrubs. In this case, vitamin C was used as a comparator [13]. The sample's test results were then calculated as the percentage value of the inhibition to determine the IC_{50} value of the sample used. The IC_{50} value determines the ability to inhibit free radicals by 50% against the sample calculated by linear regression (Figure 2) [20].

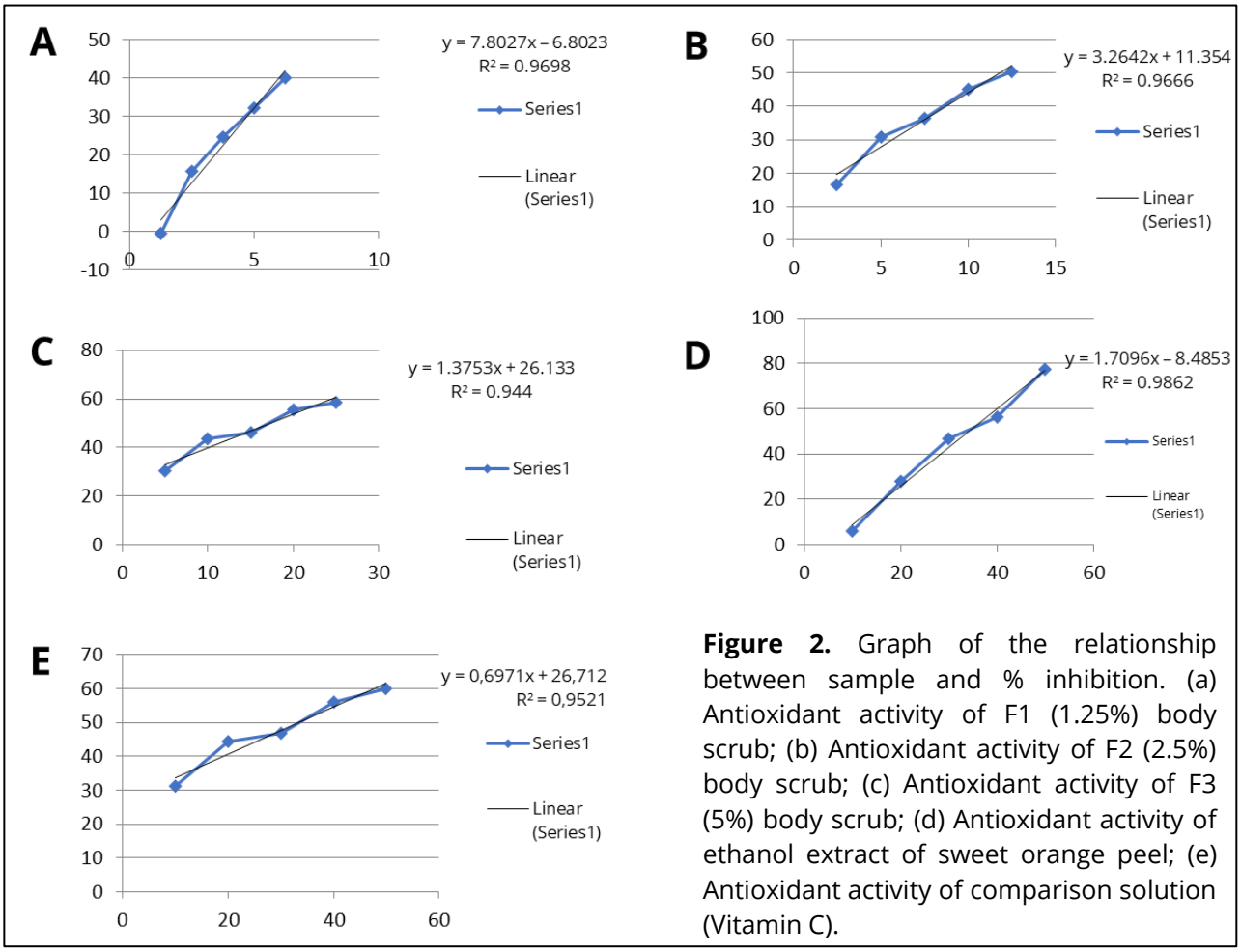

Figure 2. Graph of the relationship between sample and % inhibition. (a) Antioxidant activity of F1 (1.25%) body scrub; (b) Antioxidant activity of F2 (2.5%) body scrub; (c) Antioxidant activity of F3 (5%) body scrub; (d) Antioxidant activity of ethanol extract of sweet orange peel; (e) Antioxidant activity of comparison solution (Vitamin C).

Based on the test results of the extract, the IC_{50} value is 34.209 ppm, which is included in the category of powerful antioxidants. After testing each formula, the IC_{50} value for FI was 7.28 ppm, FII was 11.839 ppm, and FIII was 17.354 ppm. The three body scrub formulas have antioxidant activity in the robust category. These results are compared to the IC_{50} of Vitamin C, which is 33.410

ppm, which is also categorized as very strong. Based on the test results and comparing the results with the comparison sample, the body scrub preparation made can be said to be proven to have antioxidant activity [21].

Hedonic Testing Results of Body Scrub. Hedonic testing was carried out to determine the panellist's preference level based on aroma, colour, and texture. Data analysis was performed using SPSS with one-way ANOVA and Duncan methods. Based on the test results, the significance value of colour, aroma, and texture is above 0.05 (p> 0.05) with a significance value of 0.512, 0.414, and 0.780, respectively, which means that there is no significant difference between each preparation formula in colour, aroma and texture [22].

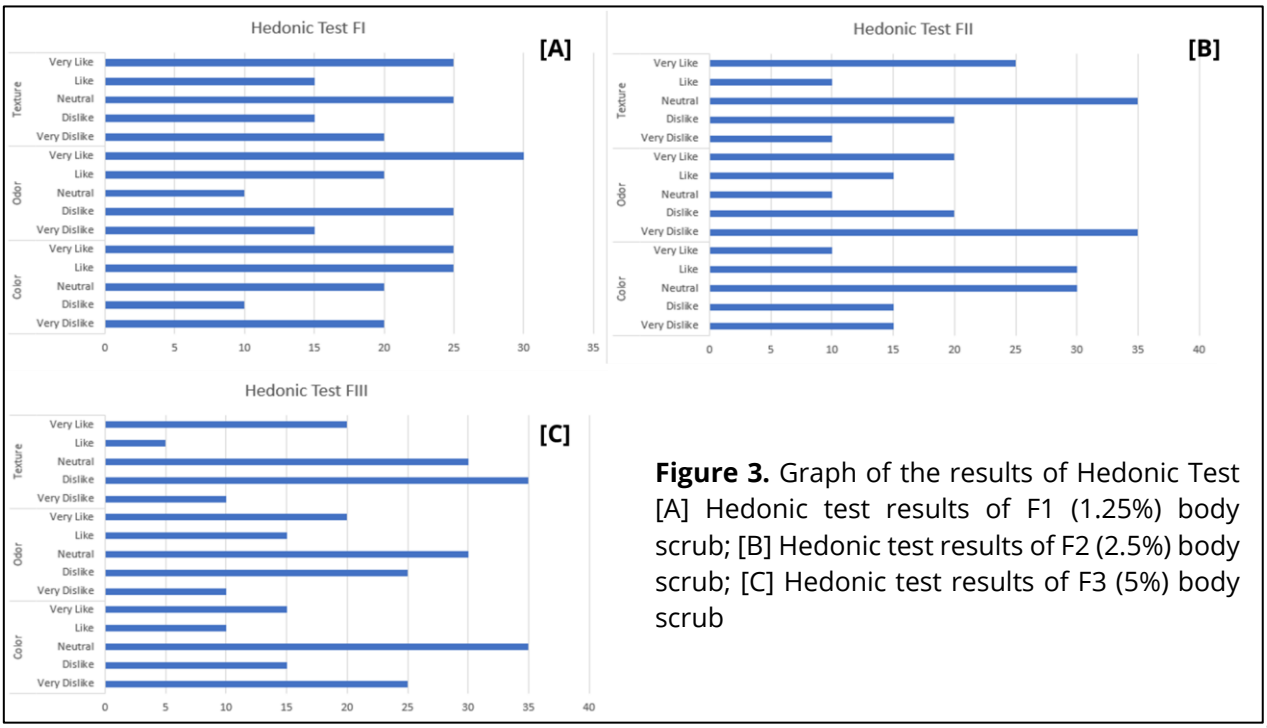

Figure 3. Graph of the results of Hedonic Test [A] Hedonic test results of F1 (1.25%) body scrub; [B] Hedonic test results of F2 (2.5%) body scrub; [C] Hedonic test results of F3 (5%) body scrub

Duncan's test was also carried out, a process that can determine a good preparation group. The Duncan test was carried out in hedonic testing to determine the panellists' liking of the 3 (three) preparation formulas. The highest subset value indicates the preferred formula. The results showed that FI excelled in assessing aroma, colour, and texture. This may occur because FII and FIII provide sharp colour and aroma when compared to FI, where FII and FIII have a greater concentration of extracts, emitting a sharper characteristic odour than FI [23].

Irritation tests were carried out on FI, FII, and FIII test samples for 0, 24, 48, and 72 hr. Based on the results of reports from panellists, three panellists experienced irritation with details of the mild irritation category of two people and very mild irritation of one person. Irritation occurred 24 hr after use. Irritation occurs due to contact time, penetration rate, area, and the ingredients' toxicity level [24].

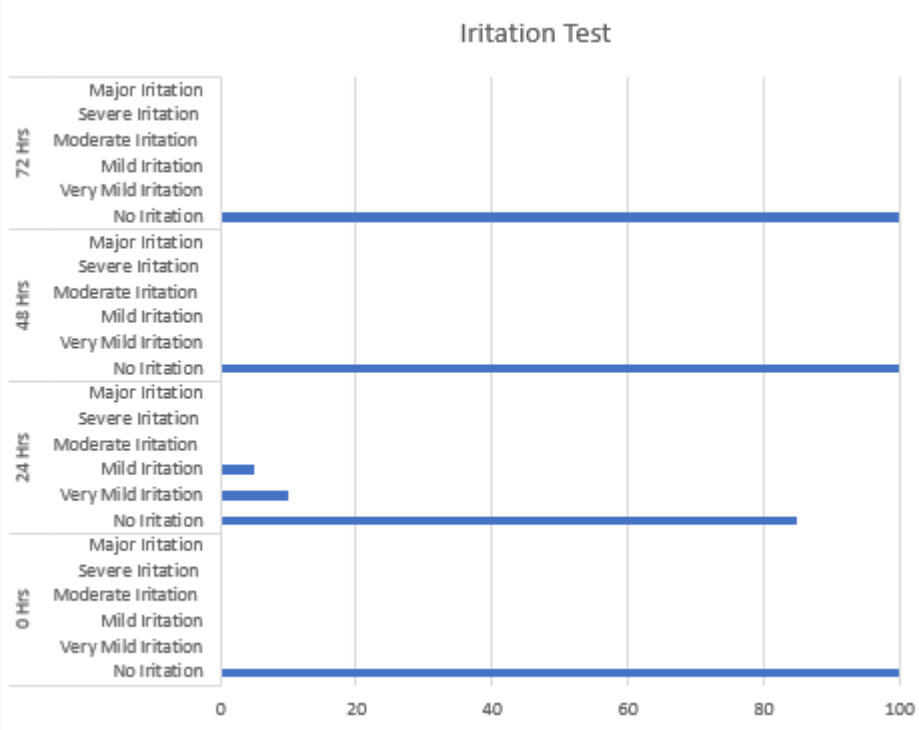

Figure 4. Graph of the results Irritation test of Body Scrub FI, FII, and FIII

Conclusions

Based on the study's results, it was found that the physical quality of the preparation showed promising results and met the standard values set. The results of antioxidant activity testing found that the three body scrub formulas have antioxidant activity values in the robust category (<50 ppm) with FI. FII. and FIII values of 7.28 ppm, 11.839 ppm, and 17.354 ppm, respectively. So, it is concluded that the body scrub is proven to have antioxidant activity

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Author Contibutions

Conceptualization, H.R., I.M. and A.S.; Methodology, I.M., M.R.E. and H.R.; Software, H.R.; Validation, I.M., M.R.E., F. and P.D.P; Formal Analysis, I.M.; Investigation, I.M., H.R. and G.A.; Resources, H.R., G.A and A.S; Data Curation, Z.; Writing – Original Draft Preparation, H.R.; Writing – Review & Editing, H.R., G.A. and R.H.; Visualization, R.H.; Supervision, I.M.; Project Administration, H.R.; Funding Acquisition, H.R.

Conflict of Interest

There are no significant conflicts

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