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The Evaluation of Physicochemical Properties of Corn Silk (*Zea mays* L.) Ethanolic Extract Face Scrub with Various of Corn's Particle Size

[1] Dian Eka Ermawati, [2] Nurul Eka Hidayati

[1, 2] Department of Pharmacy, Vocational School, Universitas Sebelas Maret, Surakarta, Indonesia

Corresponding Author: **Dian Eka Ermawati**

Abstract

Corn silk contains flavonoids as antioxidants so that it can be used as an active ingredient in topical preparations. Corn is used as a scrub to help remove dead skin cells, so the skin looks brighter. This study aims to determine the effect of scrub particle size by using sieve number variations on the physicochemical properties and stability of corn silk ethanolic extract face scrub. The corn scrub is sieved using sieve numbers 30, 40, and 60 mesh. The scrubs were dispersed into a cream formula of face scrub containing corn silk ethanolic extract of 1% (b/v). The formulas were tested for physicochemical properties, namely organoleptic, homogeneity, pH, sticking test, and dispersion test. Statistical analysis using Two Way ANOVA. The result showed that the difference in the number of particles causes differences in the physical properties of face scrub preparations in terms of viscosity and spreadability. The temperature variation in the stability test decreased the pH value but showed stable conditions in organoleptic, viscosity, and spreadability of the face scrub preparation. The particle size of the scrub of 40 mesh sieve produces the best physicochemical properties.

Keywords: Corn Silk Extract, Corn Scrub, Physicochemical Properties, Stability, Face Scrub

Introduction

Corn silk is a waste of corn plants but has yet to be used optimally. Previous research stated that corn silk contains secondary metabolites, one of which is flavonoids. Flavonoids are potent antioxidants and metal binders so that they can reduce skin damage [1]. Flavonoid compounds can protect cells from damage due to oxidation processes in the body triggered by free radicals [2]. Based on the results of the antioxidant activity test conducted by Armadany *et al* [4], the ethanol extract of corn silk has an IC50 value of 114.75 µg/mL, which is included in the moderate interpretation, so the addition of an extract of 0.0114% (w/v) has provided antioxidant effect. Corn silk ethanol extract also has antibacterial activity so that it can protect the skin from bacterial infections. Corn silk ethanol extract has a strong category of inhibition zone with an average diameter of 13.0 mm inhibition zone for *Staphylococcus aureus* and 19.30 mm for *Escherichia coli*, with a concentration of 100% w/v corn silk ethanol extract with DMSO as a diluent. The inhibition zone is strong if it has an average diameter of 11-20 mm [3].

Corn kernels contain thiamin, which can help disguise blemishes such as acne scars. Corn also contains pro-carotene, which can improve skin structure. In addition, corn's substances also help overcome acne's appearance by keeping the skin moist. The texture of corn kernels is easy to process and is abrasive (abrasive), so it can be used as a scrub to help remove dead skin cells, which can make the skin look brighter in face scrub preparation. Particle size determines the total surface area. In this case, the particle size of face scrub preparation components, such as scrub particles, can affect their effectiveness in removing dead skin cells and their nature in irritating the skin [4]. The varying sizes of corn scrub particles have never been made in face scrub preparations. Yuliati and Binarjo [4] state that the best particle size of scrub for removing dead skin cells in face scrub preparations is 30-40 mesh. The smaller the particle size, the greater the adhesion and spreading power produced. In Yulia and Binarjo's study [4], it was said that a 30-40 mesh size could remove 4.99 ± 0.57 mg of dead skin cells. Hassan *et al*. [5] have also researched formulating face scrub preparations using corn scrub, which removes dead skin cells with a particle size of 0.5 mm.

The face scrub is a cosmetic product with active and cream-based ingredients. The face scrub preparation in this study will use the active ingredient of 1% (w/v) corn silk ethanol extract. The greater the concentration of the extract used, the relatively more antioxidant content in the extract, so the effect of inhibiting free radicals is expected to be stronger [6]. Stearic acid and triethanolamine were chosen as emulsifying agents because they can improve the physicochemical properties of the preparation [7]. The presence of stearic acid can cause the cream to become softer so that the viscosity is lower. Stearic acid is

a stable base, especially for antioxidant preparations. Stearic acid in cream preparations is used in the concentration range of 1-20% [8]. In triethanolamine, the concentration usually used for emulsification is 0.5-2% of the weak acid [9]. Based on these considerations, this study was carried out on the effect of corn kernel scrub particle size on the physicochemical properties of face scrub preparations. The physicochemical tests included organoleptic, homogeneity, spreadability, adhesion, and pH during two weeks of storage.

Methodology

Materials

Corn plant was harvested from Kawedanan, Magetan, Central Java, Indonesia; ethanol 70% (PanRec ApliChem ITW Reagents Production); quercetin standard (Sigma Aldrich; St. Louis, MO); acetic acid and triethanolamine (repackaged by PT. Brataco, Indonesia); glycerin (repackaged by PT. Brataco, Indonesia); aqua dest (repackaged by CV. Agung Jaya); phenoxyethanol (repackaged by Cipta Kimia). Instruments: Analytical balance (Precisa® EP 220A, S/N 4600349, Made in Switzerland); stove (Quantum® QGC 201 E, glassware (Pyrex® made in Germany); pen type pH meter (ATC neutron. tech ® PH-009-A, made in China); Memmert Beschikung-Loading oven Model 100-800 D-91107 (Schwalbach, Germany); viscometer VT-04F; Microscope Olympus-CX43 (Tokyo, Japan); sieve number 30, 40, and 60 mesh.

Methods

Sample Preparation and Extraction

Corn silk was washed and dried using indirect sunlight and then crushed. The dried corn silk obtained was 120 g, then the maceration process was carried out using 1.2 L of ethanol 70% solvent. The maceration process was carried out for 96 hours. The maceration solution was evaporated over a water bath to obtain a thick extract [17]. **Corn scrub**: Corn kernels were washed and dried using an oven at 40 °C for 24 hours. The dried corn was crushed in a blender and then sieved in stages until it passed sieve numbers 30, 40, and 60 mesh; the corn grains were used as a scrub.

Extract Tests

An organoleptic examination was done by observing the extract's color, odor, and consistency. The yield value was the ratio between the extract obtained and the initial simplicia weight expressed in percent (%). The moisture content of the extract was calculated using a Moisture Analyzer, where as much as 1.0 grams of the extract was placed in an empty aluminum pan. The tool was closed to obtain optimal heating of the extract until the tool showed a constant reading of the extract's moisture content. Qualitative test of the flavonoid content of the extract by standard quercetin using the Thin Layer Chromatography (TLC) method. TLC analysis of the extract was carried out by spotting the extract and quercetin standard on a silica gel GF254 plate and then eluting it with the mobile phase of n-butanol: acetic acid: water (4:1:5). The results were observed under UV light at 254 nm and 366 nm [10].

Corn Hair Ethanol Extract Face Scrub Formulation

The oil phases of cetyl alcohol and stearic acid are mixed and then melted over a water bath at 60 ºC. The aqueous phases of propylene glycol, glycerin, triethanolamine, aquadest, and phenoxyethanol were mixed and heated over a water bath at 60 ºC until homogeneous. The oil and water phases are then mixed with constant stirring to form a cream base, and the last is a perfume that is added and stirred until homogeneous. Corn silk extract and corn scrub were added to the scrub base and stirred until homogeneous [11] (table 1).

Physicochemical Properties and Stability Test of Corn Hair Ethanolic Extract Face Scrub

Face scrub preparations were tested for 14 days of storage at room temperature 23 ± 2 °C on day 0 and day 14. The stability test accelerated on face scrub preparations carried out by cycling test method at 23 ± 2°C and 40 ± 2°C for 24 hours, respectively. Face scrubs preparation test for physicochemical properties observations on day 0 and day 12 after six cycles. Organoleptic testing was a preparation test to describe color, smell, and consistency. Homogeneity testing was done by smearing the preparation between glass objects and then observing it with a microscope with 40 times magnification. The spreadability test was carried out by weighing 500 mg of the preparation, placing it on the test equipment, and giving a load of 0, 50, 100, and 150 grams. Viscosity test by weighing 150 g of the preparation and then measuring the viscosity value using a VT-04F Rion viscometer and spindle number 1. The pH test was carried out by dissolving 1.0 mL of the sample into distilled water up to 10 mL. The pH meter was previously calibrated using a solution of pH 4 and pH 7 [12]. The adhesion test was carried out by placing 0.5 g of the preparation on a glass slide covered with other objects and then giving a load of 1.0 Kg for 5 minutes. The slide was then attached to the test equipment, and the adhesion time measurement was carried out starting when the load on the test tool was released until the two slides were released [13].

Table 1: Corn Hair Ethanol Extract Face Scrub Formula with variations in the size of corn kernel scrub particles based on passing sieve numbers 30, 40 and 60

Ingredients	Function of ingredients	Weight (gram)			
		Base Formula	Formula 1 (Sieve no 30 with particle size of 0.6 mm)	Formula 2 (Sieve no 40 with particle size of 0.4 mm)	Formula 3 (Sieve no 60 with particle size of 0.25 mm)
Extract	Active substance	-	1.00	1.00	1.00
Corn Scrub	-	5.00	5.00	5.00	5.00
Cetyl alcohol	emulgator	0.78	0.78	0.78	0.78
Stearic acid	emulgator	7.80	7.80	7.80	7.80
Triethanolamine	emulgator	0.88	0.88	0.88	0.88
Glycerin	Humectant	4.90	4.90	4.90	4.90
Propylene glycole	Enhancer	4.00	4.00	4.00	4.00
Phenoxyethanol	Preservative	0.10	0.10	0.10	0.10
Water	Solvent	75.54	75.54	75.54	75.54

Data Analysis

The test results are discussed based on literature such as journals and books that include standard parameters of test procedures and test results. The data obtained were analyzed statistically using the Two-Way ANOVA test with a 95% confidence interval. Statistical analysis to determine whether there were significant differences in the three formulas in each test of the physical properties of the corn silk ethanol extract face scrub preparation.

Results and Discussion

Plant Determination

The results obtained from the determination show that the sample used was corn plant with document number 232/UN27.9.6.4/Lab/2019. Determination aims to prove that the plants used from the corn plant family by the morphological characteristics. In this study, the raw material used was corn plant with the species name *Zea mays* L.

Extractraction Process

The time of the maceration process affects the yield of the extract, where the maceration time that was too short causes not all the phytochemical compounds to dissolve in the solvent used, and the extraction time that is too long results in the damaged phytochemical compounds extracted [14]. Extraction time in the extraction of flavonoids that was too long can result in the degradation of flavonoid compounds by light and oxygen [3, 15]. In this research, corn hair maceration was carried out for 96 hours, and it was hoped that the extraction process of the active compounds would be optimal. Macerated viscous corn hair extract has a reddish-brown color and a characteristic herbal odor. Corn silk contains secondary metabolites, one of which was a flavonoid that has antioxidant activity [1].

Yield and Water Content of the Extract

This study's percentage yield value of corn silk extract was 6.175%. The longer extraction time causes the process of penetration of the solvent into the cell. The longer the time used, the better the penetration of the solvent into the cell, and the more compounds that diffuse out of the cell so that the extract yield value is relatively high. The percentage of yield, which was quite high, indicates the number of compounds that were successfully extracted [16, 17]. Previous research yielded a yield of 4% [2]. Determination of the water content in the ethanol extract of corn silk was carried out using a moisture analyzer and obtained a value of 8%. The maximum permissible water content requirement was 10% [18].

Qualitative Test Results for the Active Content of the Extract

Qualitative test of the content of flavonoids in corn silk using the TLC method. The principle of TLC was to separate samples based on differences in polarity with the solvent, where the analyte moves across the stationary phase under the mobile phase [19]. The eluent has a function as a solvent for a mixture of substances, carrying the active components to be separated through the stationary phase so that the spot extract has an Rf value within the required range and to provide selectivity based on polarity for the compound mixture to be separated [20]. The eluent requirements include having sufficient purity, being stable, having low viscosity, having a linear isothermal partition, not having too low or high vapor pressure, and having a low toxicity. The eluent for the mobile phase used in this study was a mixture of n-butanol: acetic acid: water (4: 1: 5) because the composition of the three eluents has very polar properties, so it can separate quercetin compounds, which are also polar in the stationary phase on Silica GF254 plate. Quercetin was a compound belonging to the flavonoid compound, one of the largest natural phenol groups. Because it has several hydroxyl groups or sugar, flavonoids are polar compounds. Every 1.0 gram of dry corn silk contains a Total Flavonoid Content (TFC) of 17.12 mg Rutin equivalent (RE) [15]. Flavonoids are potent antioxidants [1], so they can protect cells from damage due to oxidation processes in the body triggered by free radicals [2]. The Rf value is a characteristic parameter of Thin Layer Chromatography (TLC). The Rf value was also a measure of the migration speed of a compound on the chromatogram. The Rf value was the ratio between the compound's and solvent's distance eluted. In this study, the Rf (Retrogradation Factor) measurements of corn silk ethanolic extract and standard quercetin observed using UV light at 254 and 366 nm were 0.913 and 0.938, respectively. The results in Figure 1 show that the ethanol extract of corn silk and the standard quercetin have no different Rf values, so the Rf value still indicates the presence of total flavonoid compounds in the ethanol extract of corn silk.

The results of the phytochemical detection test by Karima [10] showed the presence of flavonoids because the Rf value of the compounds contained was close to the Rf value of the flavonoid compounds. Observation of 366 nm UV produces spots that glow against a dark background, so spots that can fluoresce can be seen visually. This was caused by the interaction between UV light and the chromophore groups, which were bound by auxochromes in the stain spots. Visible fluorescence results from the emission of light emitted by these components when electrons were excited from the ground level to a higher energy level, then return to their original state by releasing energy.

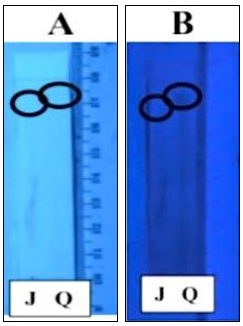

Fig 1: Results of TLC analysis where the stationary phase used is silica gel GF254, and the mobile phase is n-butanol: acetic acid: water (4: 1: 5) [10]. The TLC plate was observed under 366 nm UV light for fluorescent spots on the ethanol extract of corn silk (J) and quercetin (Q)

Corn Silk Ethanolic Extract Face Scrub Formulation

A scrub was the main component of face scrub preparations, which gives a soft and fresh impression on the face and removes dead skin cells to make a face look brighter [21]. Variation in scrub particle size was obtained by sifting corn dust using graded sieve numbers, namely 30, 40, and 60 mesh with the same weight (gram) in the formula. The particle size of corn scrub for face scrub preparation was determined by testing the physicochemical properties of face

scrub by comparing each formula's test results to determine the best physicochemical properties. The research shows that the face scrub preparation with the scrub particle size using the most significant sieve number (30 mesh) has a much thicker consistency, almost like a paste preparation, than the other two preparations. This was because this preparation has a much larger size of corn scrub particles, so the number of particles can fill the cavities between the particles in the face scrub preparation and make the preparation appear thicker. Based on calculations, it was known that in 5.0 grams of corn scrub, there were 4,535,660 particles for a 30-mesh sieve, 12,895,660 for a 40-mesh sieve, and 61,146,495 for the 60-mesh sieve respectively. It can be concluded that with the same weight of corn scrub in the formula, the number of scrub particles added to it differs. The difference in the number of particles causes differences in the physical properties of face scrub preparations in terms of viscosity and spreadability.

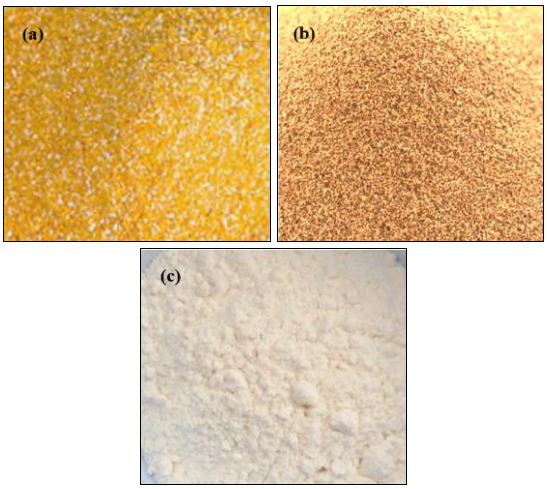

Fig 2: The corns scrub particles with variations of sieve where the 30-mesh sieve (a), 40 mesh sieve (b), and 60 mesh sieves (c)

Test Results of the Physicochemical Properties of Face Scrub

During storage, it was noted that the three formulas changed in consistency, smell, and color, which became darker on the 14th day. Budiman [22] stated that temperature causes discoloration of preparations during storage. Temperature was one of the factors that can affect the stability of the preparation. High temperatures can speed up chemical reactions because every 10°C temperature increase can increase chemical reactions two or three times. In addition, the color change occurs due to the reaction between Triethanolamine and the active ingredient (flavonoid). TEA was an amine with a strong base, and the flavonoids in corn silk extract were phenolic compounds, so the color would change when it reacted with a base [23]. The homogeneity test of the three formulas showed the presence of brownish corn scrubs. This was due to the difference in the size of the scrub dispersed in the preparation. The distribution of all scrub particles in the face scrub preparation was even when applied to the surface of a glass object. The results prove that the active substance was evenly dispersed in the preparation so that the three-face scrub formula of ethanolic extract of corn silk with corn scrub particles can be homogeneous. The homogeneity test aims to determine the homogeneity of extracts and scrubs in preparation and detect changes in preparation that may occur during storage. This relates to the active substance dispersed with the base and filler components in the face scrub preparation.

The pH test was a parameter of chemical properties related to the stability and effectiveness of the active substance as safety and comfort when used because it was applied to facial skin. The pH value of the preparation was appropriate for pH skin, namely 4.5-6.5. Preparations that were too alkaline can cause dry skin, while too acidic preparations can irritate [24]. Based on the research results, the pH value was relatively stable during storage. The pH value must comply with SNI 16-4399-1996 as a condition for the normal pH of the skin, namely 4.5-6.5. The pH changes in the three formulas still met the skin's standard pH requirements on the 14th day after storage. The pH value of the face scrub preparation on day 0 for formula 1 was 7.2, and on day 14 was 7.0. That was not following the normal pH of facial skin, caused by the combination of stearic acid and TEA, which increases the pH value because TEA has a pH of 10.5. The interaction between acids and bases makes the preparation's pH neutral, so the longer the preparation was stored, the more the pH value decreases [25]. The formulas showed a significant difference ($p<0.05$) in the pH test value. The storage time factor showed that there was a significant difference ($p<0.05$) in the pH test value, and there was a significant interaction ($p<0.05$) between formulas at storage temperature (table 2).

Table 2: Test results for the physicochemical properties of the corn silk ethanolic extract face scrub formula with variations in the size of the corn scrub particles for two weeks of storage at room temperature

Formula	Physicochemical Properties					
	Adhesion time (second)		pH		Speadibility area (cm)	
	Week-1	Week- 2	Week- 1	Week- 2	Week- 1	Week- 2
1 (Sieve no 30)	3.18±0.05	5.52±0.01	7.20±0.10	7.00±0.10	4.60±0.10	3.50±0.10
2 (Sieve no 40)	4.66±0.01	6.10±0.01	7.20±0.10	5.50±0.10	5.10±0.10	4.00±0.10
3 (Sieve no 60)	5.45±0.01	7.51±0.01	7.20±0.10	6.00±0.10	5.60±0.10	4.40±0.10

*Mean±SD

The adhesion test aims to determine the ability of the face scrub preparation to adhere to the skin surface when used so that the absorption of the active ingredients was optimal. The longer the preparation was attached to the skin, the more optimal the active substance absorption [25]. Priawanto and Handining [24] stated that adhesion is related to viscosity. The thick consistency of the preparation causes a higher viscosity, so the adhesion time becomes longer. The particle size of the scrub affects the adhesion of the face scrub preparation, where the smaller the particle size of the scrub, the more excellent the adhesion produced. Ermawati *et al.* [10] explained that this was due to changes in space between particles at smaller particle sizes so that the adhesion became greater, whereas, in the face scrub formula with larger particle sizes, there were quite a lot of air spaces between them. Changes in the consistency of the preparation

during storage at different temperatures are due to the properties of the oil phase of the face scrub preparation, one of which is cetyl alcohol, whose consistency is easily affected by changes in temperature [8]. Particle size showed a significant difference ($p<0.05$) in the adhesion test value. The length of storage time also showed a significant difference ($p<0.05$) in the adhesion test value, and there was no significant interaction ($p>0.05$) between the formula and the storage temperature on the adhesion test value.

A spreadability test was conducted to determine the ability to spread the preparation when applied to the skin. The value of the area of spreadability is directly proportional to the area of contact of the active substance in the preparation. The results of the spreadability test in this study were between 5.0- 7.0 cm. These results follow the literature, which states that good spreadability was between 5.0- 7.0 cm. The smaller the particle size, the greater the resulting scattering power due to the high resulting density and reducing the space between particles, making it difficult for movement between particles [25]. Indrawati and Sari [25] stated that the decrease in spreadability was thought to be due to the less stability of the face scrub preparation emulsion. This will affect the coalescence of the dispersed phase particles, which causes the particle size to increase so that the spreading power of the face scrub decreases. Spreadability increases during storage due to temperature, causing cetyl alcohol to become softer in consistency. The formula shows a significant difference ($p <0.05$) in the spreadability test value. The storage time factor showed a significant difference ($p<0.05$) in the spreadability test values. There was no significant interaction ($p>0.05$) between each formula at the storage temperature on the spreadability test values.

Conclusion

The effect of variations in the corn scrub sieve number produces different particle sizes, affecting the physicochemical properties of the corn hair ethanol extract (*Zea mays* L.) face scrub preparation. Variations in particle size affect the spreadability and adhesion of preparation. The smaller scrub particle size caused an increase in the spreadability and adhesion values but didn't affect the pH of the face scrub preparation. The difference in the number of particles causes differences in the physical properties of face scrub preparations in terms of viscosity and spreadability. The temperature variation in the stability test decreased the pH value but showed stable conditions in organoleptic, viscosity, and spreadability of the face scrub preparation. The corn scrub sieve number that produced the best physicochemical properties in the corn silk ethanolic extract face scrub preparation was 40 mesh sieve number.

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