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Formulation body scrub from silica gel of palm oil shell (*Elaeis Guineensis* Jacq.)

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Abstract

***Background**: Oil palm shell is a waste of oil palm plants that can be used as a source of silica gel because it contains SiO_2. This study aims to determine the oil palm shell silica gel can be used as an active ingredient in the manufacture of body scrubs.*
***Method**: This study uses active ingredients with various concentrations of 2,5%, 5%, and 10%. The process of making silica gel starts with ashing, washing, making a sodium silicate solution, and adding NaOH. The preparation test includes an organoleptic test, homogeneity test, pH test, dispersibility test, adhesion test, irritation test, dead skin cell removal test, and stability test.*
***Results**: From the results of this study, the SiO2 content of palm oil shell silica gel is as much as 43% with XRF testing, and XRD results at an angle of 2Θ=22,93º. The FTIR results showed the presence of a Si-OH group at a wave number of 3439.08 cm -1, a Si-O-Si group at a wave number of 1020,34 cm -1, and a -OH group at a wave number of 806,25 cm -1. The preparations that have been made meet the physical evaluation of the preparations, namely in the form of semi-solid, white color, and smells of oleum citri. Each homogeneous preparation, pH with a value ranging from 4,7 to 6,5, does not irritate the skin, meets the requirements of the spreadability test and adhesion test, has poor stability because the pH value has increased and decreased, has a good keratin content value, and the stability test of the formula against temperature at pH showed significant results (p>0.05).*
***Conclusion**: This research concludes that palm oil shell silica gel can be formulated as an active ingredient in making body scrubs and has the effect of removing dead skin cells on the skin.*
***Keywords:** Body scrubs, formulation, palm oil shell (Elaeis guineensis Jacq), silica gel*

BACKGROUND

Jambi Province is a province that has a large enough role in producing superior commodities from plantations and agriculture. Batanghari Regency is one of the centers of oil palm plantations in Jambi Province. The area of oil palm plantations in 2020 amounted to 143.456,00 hectares with a production output of 13,735 tons per year (1). The process of developing oil palm plantations and harvesting areas is one of the factors for the emergence of several wastes that are not used properly, such as stem roots, leaves, shells, and empty bunches. A small part of the waste from palm shells is used for paving roads, animal feed, and fuel (2).

As for other sources of silica such as rice husk as much as 87-97% (3) and corn cobs contain as much silica as 67.41% (4) so the silica content in palm shells can be used as the main ingredient in body scrub formulation.

Silica is generally used as an exfoliating agent in a body scrub because it has an important role in removing dead cells from the surface of the skin (5). Silica is safe in use to maintain moisture such as in food, medicine, sensitive materials, electronics, and even films (3). A previous study conducted by (6) used palm oil shell charcoal as an active ingredient in the manufacture of body scrubs that are used for detoxification or can eliminate toxins that are not needed by the body. Based on the description above in the presence of industrial waste produced by palm shells (*Elaeis guineensis* Jacq.), researchers are interested in conducting research by utilizing pal shell waste as an active ingredient of silica gel as an active

ingredient for making body scrub. This study aims to determine whether palm oil shell silica gel (*Elaeis guineensis* Jacq.) can be used as an active ingredient for making body scrub and to find out how the formulation of body scrub from palm oil shell silica gel (*Elaeis guineensis* Jacq.) is produced.

METHOD

This research was conducted at the Pharmaceutical Technology Laboratory, Clinical Microbiology Laboratory, and the Research Laboratory of STIKES Harapan Ibu Jambi. The manufacture of silica gel ash from oil palm shells (Elaeis guineensis Jacq) is carried out by cleaning the oil palm shells and washing them with running water and then drying them in the sun to a constant weight. After the oil palm shells are cleaned, they are dried in the sun and then burned using a furnace at a temperature of 700°C for 4 hours. Then obtained ash is done by smoothing and sieving with mesh sieve no.100 (3).

After that, it continued with the manufacture of oil palm shell ash (Elaeis guineensis Jacq) by purifying a 3% HCl solution with a ratio of ash: HCl composition of 1: 10 for 2 hours. Then stirred using a magnetic stirrer for 1 hour, and allowed to stand for 24 hours, then filtered to form white gels. The resulting residue was rinsed using distilled water until it reached a pH of 7 or neutral. Then the ash was evaporated to dryness in an electric oven at a temperature of 100ºC (7).

Extract oil palm shell ash (Elaeis guineensis Jacq.) and 1 N NaOH solution, with a ratio of 1: 6 stirred using a magnetic stirrer in an Erlenmeyer heated at 80°C for 60 minutes. Then let stand one night, filtered and the residue is washed with some hot water. The resulting sodium silicate solution will be transparent white (3).

A concentrated CH_3COOH solution was added to the sodium silicate. Before the addition of acid, the pH was measured as the initial pH and an acid solution was added to pH 7. After the addition of acid, the resulting white hydrogel-like glass was left for 18 hours and dried at 80°C (3). Then sieved according to the degree of fineness of the powder using a 40 mesh sieve (8). Furthermore, silica gel is formulated into body scrub preparations.

a. Design Formula

Table 1. Design Formula

Ingredient	Formula		
	F1 (%)	F2 (%)	F3 (%)
Silica gel	2,5	5	10
Stearic acid	15	15	15
Propylene glycol	5	5	5
Triethanolamine	1,2	1,2	1,2
Glycerin	5	5	5
Cetyl alcohol	2	2	2
Perfume	qs	qs	qs
Aquadest	ad 100	ad 100	ad 100

b. Preparation of Body Scrub

Oil phases such as stearic acid and cetyl alcohol were melted in a water bath until they became liquid at a temperature of 70°C. Then the aqueous phase such as triethanolamine, glycerin, and propylene glycol was dissolved in distilled water. Then the oil phase and water phase were mixed while stirring slowly to form an o/w-type base. Then add silica and perfume and stir until homogeneous. Let it cool and put it in a container (6).

c. Evaluation of Body Scrub

1. Organoleptic Test
   Tests are carried out by observing changes in color, odor, and changes in shape (9).
2. Homogeneity Test
   The test was carried out by observing visually using two glass objects, one of which was smeared with a body scrub thinly and evenly, then observed under ultraviolet light (10).
3. pH test
   Testing is done by observing using a pH meter. A total of 1 g of the preparation was put in a beaker glass and diluted in 10 ml of aqua dest (9).
4. Spreadability Test
   The test was carried out by weighing 0.5 g of body scrub preparation placed in the middle between 2 glass plates, on it was given a load of 150 grams,

left for 1 minute and the diameter of the spread was measured.

5. Adhesion Test
   This test is carried out by weighing 0.5 grams of body scrub preparation and then placing it on a slide, then another object of glass is placed on it. Then put a load of 100 grams for 5 minutes, then release the load weighing 80 grams. Record the time it takes for the two sides to come off. Recorded time describes adhesion (11).
6. Stability Test
   The test was carried out for 6 cycles by storing the preparation at 4°C for 24 hours and then removed and placed at 40°C, this process was counted as 1 cycle. Tests carried out during the storage process are organoleptic, pH, and homogeneity (12).
7. Irritation Test
   This test requires 3 volunteers by applying a body scrub on each forearm and then observe for 5 minutes the symptoms that occur. Do it 2 times a day for 2 consecutive days, and observed if there are symptoms of redness and edema on the skin (13).
8. Scrubbing Test
   a) Application Scrubbing Pharmacist
      This test was carried out or applied to 8 volunteers who were grouped into 4 groups, namely group 1 F1, group 2 F2, group 3 F3, and a comparison group, each of which was applied to 2 different volunteers. The test was carried out by applying 2 grams of each test F1, F2, F3, and the comparison formula to the volunteers' arms that had been marked with a certain size for 4 treatments. Then let stand and rubbed for 15 minutes. The preparations that have been applied are collected and weighed, then extracted using 10 ml of 4% sodium hydroxide solution. Then the solution was stirred with a stirrer for 10 minutes and centrifuged for 30 minutes. The clear layer was taken for qualitative and quantitative testing.
   b) Qualitative test of dead skin cell protein
      According to (14) in a qualitative test, a solution that can be dripped with biuret reagent (made by dissolving 45 grams of sodium potassium tartrate, 15 grams of CuSO4.52O, and 5 grams of potassium iodide in 0.2 N NaOH to 1000 ml). If the solution turns purple it indicates the presence of protein.
   c) A quantitative test of dead skin cell protein
      The quantitative test is done by:
      (a) Make albumin liquor with a concentration of 1000 ppm by weighing 100 mg dissolved in 100 ml of water in a measuring flask.
      (b) Dilution of the standard solution, then add Biuret reagent, one concentration of which is measured at maximum wavelength by UV-Vis spectrophotometry in the range of 400-800 nm.
      (c) The extraction result of the applied preparation solution was taken as much as 200 l which was diluted with 4% NaOH to 10 ml in a volumetric flask, then re-measured the maximum wavelength absorbance using a UV-Vis spectrophotometer. The blank used was a body scrub preparation that was not applied to the volunteer's arm and extracted with the same treatment on the sample. Then the value of the levels is calculated which aims to determine the dead skin cells that are lifted.

RESULT

1. Silica Gel Manufacturing
   The steps of making silica begin with the process of taking samples, curing, and ashing, until the formation of silica gel. The yield of silica gel manufacture is 5.56%.

2. Analysis XRD (X-Ray Diffraction)

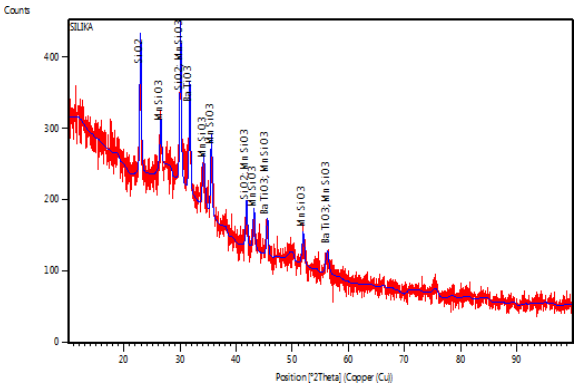

Figure 1. Silica Gel Diffractogram Palm Shell

3. Analysis XRF *(X-Ray Fluorescence)*
   The results of the XRF analysis of SiO_2 showed that the percentage of silicon oxide content in oil palm shell ash was 61.67% while in oil palm shell ash silica it was 43.93%.
4. Analysis FTIR *(Fourier Transform Infrared)*
   The results of the FTIR analysis of SiO_2 obtained a typical functional group in the form of a silanol group (Si-OH) with several 3439.08 cm^{-1} and a siloxane group (Si-O-Si) with several 1020.34 cm^{-1}.

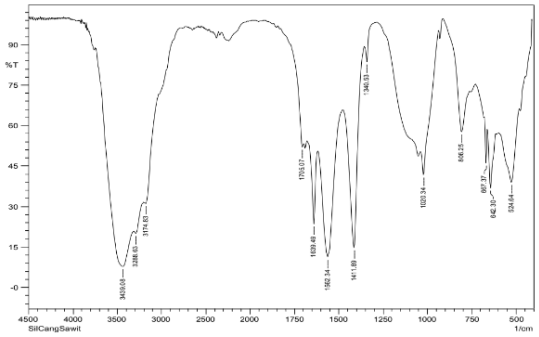

Figure 2. FTIR Ash Silica Palm Shell

5. Preparation of Body Scrub
   Body scrub preparations were made using formula 1, formula 2, and formula 3 with different concentrations of each formula on silica gel, namely 2.5%, 5%, and 10%. Then the body scrub preparation was evaluated which included organoleptic testing, homogeneity testing, pH testing, dispersion testing, stability testing, irritation testing, and scrubbing testing on volunteers. The preparation obtained was in the form of body scrub in the form of a semi-solid, white color, and a characteristic odor of oleum citri.

6. Evaluation of Physical Properties of Body Scrub
   a) Organoleptic Test
      The results of the organoleptic test of body scrub preparations that have been observed are changes in color, shape, and odor (Figure 3).

Figure 3. Organoleptic Test

   b) Homogeneity Test
      The results of the homogeneity test of body scrub preparations that have been carried out show that all formulas are homogeneous, and no granules are obtained.
   c) pH Test
      The results of the pH test of body scrub preparations showed that the pH of the body scrub met the pH requirements of topical preparations.
   d) Spreadability Test
      The results of the dispersion test carried out three times showed that each formula had good dispersion.
   e) Adhesion Test
      The results of the adhesion test that has been carried out show that the body scrub preparation formula has good adhesion.
   f) Stability Test
      The results of the stability of the body scrub preparation were carried out for 12 days at a temperature of 4ºC and 40ºC, these results showed that there was no change in color, aroma/odor, shape, homogeneity, and pH.

g) Irritation Test
The results of the irritation test carried out on 3 volunteers on each body scrub preparation formula (Figure 4)

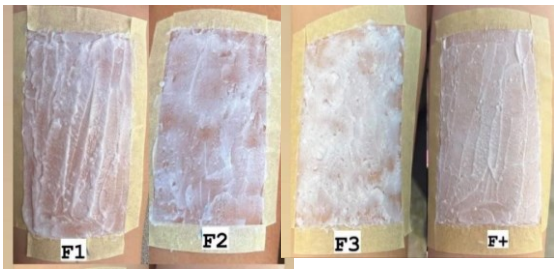

Figure 4. Irritation Test

7. Scrubbing Test
a) Qualitative Test
The results of the qualitative test of the presence of dead skin cell protein in each body scrub preparation formula (Figure 5)

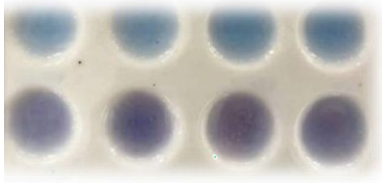

Figure 5. Qualitative Scrubbing Test

b) Quantitative Test
The results of the qualitative test for the presence of dead skin cell protein in each body scrub preparation formula were indicated by the value of creatinine levels.
Test for Keratin Levels
The results of the keratin test in each formula were carried out on 2 people for each formula with four repetitions (Table 2)

Table 2. Keratin Level

Formula	Keratin Level (mg/ml)
F1	40,5±0,7
F2	49,9±5,2
F3	62,8±5,0
FP	68±0,9

DISCUSSION

The sample used in this study was oil palm shells in Bungku Village, Batanghari Regency, Jambi Province. The sample is then identified which aims to prove that the sample used obtains a clear identity from the plant being studied and avoids errors in using the materials in the study (15). From the results of the identification, it was stated that the plant was an oil palm shell (Elaeis guineensis Jacq.).

The manufacture of silica gel from oil palm shells by burning until it becomes black charcoal obtained a yield of 40%. Then ashing is carried out to remove impurities contained in the ash using high temperatures in the process carried out, and the ash yield is 3.15%, in the previous study, oil palm shell ash was obtained by 31.45% (16). The yield of silica gel from oil palm shells was 5.56%, the results obtained were different from previous studies where the results obtained were 58.02% due to impurities other than SiO2 remaining or sticking (Karimullah et al., 2018). Silica curing time affects the silica yield value obtained, so the longer the curing time for silica, the silica yield value decreases (17).

XRF analysis of the ash of oil palm shells was carried out to determine the silica content of the purification process. The results of the analysis showed that the silica contained in oil palm shell ash had a SiO2 compound content of 61.67%. Then the palm oil shell ash silica was analyzed by XRF with the results of the analysis showing that the silica content of the SiO2 compound was 43.93%.

This shows a decrease in SiO_2 compounds in each XRF analysis because the washing of the sample is not so clear that the Cl element is left behind, if washing is done more often then the results of the analysis are achieved until a higher purity is achieved. In addition to the Cl element contained in these compounds, there are other compounds such as Fe and Ca (18). In addition to the above elements, there is also an element of Mn which is formed because at the time of combustion it uses high temperatures which causes the silica gel of the ash of palm shell ash to be blackish gray (19).

XRD analysis of the ash of oil palm shells was performed to determine the degree of crystallization produced. From the

XRD analysis of oil palm shell silica gel, it shows the formation of SiO2 compounds in the diffractogram results with high intensity at an angle of 2Θ: 22.93º; 30.05º; and 41.79º which is by the SiO2 provisions in the ICSD number standard 01-075-3163. Silica ash from oil palm shells is formed in the area of 2Θ = 22.93, this shows the same results as research (20) where the silica produced is amorphous. Meanwhile, in the 2Θ = 30.05º and 41.79º regions, it shows that the silica is in crystalline form. The formation of amorphous silica is due to the presence of a silanol group (Si-OH) on its surface. Meanwhile, crystalline silica is formed due to the presence of inorganic impurities that are still attached or do not dissolve during acidification (21).

Silica ash from oil palm shells was analyzed by FTIR or infrared spectrum which aims to identify the typical functional groups of a compound (22), namely the silanol group (Si-OH) and the siloxane group (Si-OH). O-Si). Based on the FTIR analysis that has been carried out, the results of the wave absorption number of Si-OH in the silanol group (Si-OH) are 3439.08 cm-1. Then the value of the wave absorption number on Si-H is the number 806.25 cm-1. Then the value of the absorption number of Si-O-Si waves in the siloxane group (Si-O-Si) is the number 1020.34 cm-1. The absorption spectra pattern of palm oil shell ash silica shows a broad band with a peak at a wave number of 3439.08 cm-1 which can be interpreted as a vibrational absorption band in the -OH group, namely silanol. The absorption spectra pattern is 1020.34 cm-1 which is the asymmetric stretching vibration of the Si-O group in siloxane (23).

In this study, oil palm shells were synthesized into silica gel in the manufacture of body scrub preparations for skin care. The use of silica gel in oil palm shells as a scrub acts as an active ingredient in maintaining skin care. Body scrubs have benefits for the skin, including removing dead skin cells, softening the skin, and moisturizing the skin.
In body scrub preparations using palm oil shell silica gel formula 1, formula 2, and formula 3 with a concentration of 2.5%, 5%, and 10% respectively, the body scrub is obtained in the form of a semi-solid body scrub, white and characteristically oleum citrus odor. and each of these formulas has complied with the cosmetic standards in group B, namely the cosmetics industry that can make certain forms and types of cosmetic preparations using simple technology.

Evaluation of Body Scrub Preparations

1. Organoleptic Test

   In this study, an evaluation of body scrub preparations from palm oil shell silica gel (Elaeis guineensis Jacq.) was carried out against organoleptic tests carried out by visually observing the shape, color, and aroma (9). Organoleptic examination of formula F0 has a semi-solid form, yellowish white, and has a characteristic odor of oleum citri, in formulas F1, F2, and F3 that the observed results have a semi-solid or semi-solid form, white color, and a characteristic aroma of oleum citri.
2. Homogeneity Test

   The results of the homogeneity examination showed that all body scrub preparations were homogeneous with an even distribution and there were no coarse grains. This is to the requirements of the preparation which must show a homogeneous arrangement and no coarse grains are seen (24).
3. pH Test

   In this study, an evaluation of body scrub preparations made from palm oil shell silica gel (Elaeis guineensis Jacq.) was carried out on pH examination to determine the pH value. Based on the value of the pH range of the scrub preparations that have been made, namely by using a pH meter on the formula F0 6.0; on the formula F1 5.2; on the formula F2 5.3; and the F3 5.5 formula. While the value of the pH range using universal indicator paper in all formulas with a pH value of 5. This shows the pH value is very important because it aims to determine acidity so that the pH of the preparation is the same as the

physiological pH of the skin. The results of pH testing on body scrub preparations are in the pH range of topical preparations, namely 4.5-6.5 (9) and no irritation occurs, if the pH is too alkaline it causes scaly skin, while the pH is too acidic triggering irritation skin (25).

4. Spreadability Test

   In this study, an evaluation of body scrub preparations made from palm oil shell silica gel (Elaeis guineensis Jacq.) was carried out against the spreadability test to determine the speed of spread of the scrub preparation on the skin. The dispersion test of the scrub preparations that have been carried out is by using a load of 100 grams and 50 grams, by measuring the caliper, ruler, and millimeter paper required. The value of dispersion in the formula F0 is 4.0 cm2; in the formula F1 5.1 cm2; in the formula F2 5.1 cm2; and in the formula F3 5 cm2. This shows that the dispersion value obtained is by the range of good dispersion values with a range of 5-7 cm2 (26). Factors that affect dispersion are the concentration of the active substance added, temperature, stirring method, and pH (27). The wider the spread, the wider the contact of the active substance in the scrub preparation with the skin (28).

5. Adhesion Test

   In this study, an evaluation of body scrub preparations made from palm oil shell silica gel (Elaeis guineensis Jacq.) was carried out against the adhesion test to know how far the scrub preparations can stick to the skin. Based on the requirements for the adhesive power of the scrub preparations that have been made, namely with a load of 100 grams and 80 grams. The time obtained from the adhesion that has been carried out on the formula F0> 8.91 seconds; in the formula, F1> 5.27 seconds; in the formula, F2>4.11 seconds; and on the formula, F3> 4.25 seconds. This shows that the results of the dispersion test by the requirements for good adhesion for topical preparations are more than 4 seconds (26). One factor that affects adhesion is the addition of the concentration of the active substance. That is, the higher the concentration and the denser the body scrub dosage form, the longer the adhesion time will be (29). Light and temperature can affect the adhesion value of preparation (30).

6. Stability Test

   In this study, an evaluation of body scrub preparations made from palm oil shell silica gel (Elaeis guineensis Jacq.) was carried out for stability tests to know and see whether the preparations were stable at storage temperatures for a certain time. The time used for the stability test is 12 days. The stability test was accelerated by using a cold temperature of 4ºC and a temperature of 40ºC as a comparison of the results obtained. Observations were made on the storage stability test for 12 days at that temperature there was no change in the preparation, dosage form, color, and odor. Tests on pH decreased and increased pH values, due to the influence of temperature (27), but these results were still within the range of topical dosage values of 4.5-6.5 (9). This indicates that the preparation made is stable.

7. Irritation Test

   In this study, an evaluation of body scrub preparations made from palm oil shell silica gel (Elaeis guineensis Jacq.) was evaluated against the irritation test to see whether the preparation caused an irritating reaction to the skin. Reactions that occur such as the appearance of redness and edema on the skin (13). From the test results, there was no irritation effect in the form of itching, redness, and swelling of the skin caused by body scrub preparations that were applied to the skin.

8. Scrubbing Test

   a. Qualitative Test

      From the results of research that has been done, the formulas F0; F1; F2; and F3 experience a color change to purple. The biuret reagent reacts with the protein peptide bonds in the sample. The presence of protein in the

sample is indicated by a change in the color of the sample to purple. The color formation is due to the presence of substances containing two or more peptide bonds which can form a purple complex with Cu salts in an alkaline solution (31).

b. Quantitative Test
From the results of the research that has been done, the formula that removes the most dead skin cells lies in the F3 formula. This is because the concentration used is greater than the formulas F1 and F2 so it can affect the lifting power of dead skin cells. The keratin levels obtained were obtained from the application of body scrub preparations on the volunteers' arms. This indicates that the addition of silica with a certain concentration can affect the lifting capacity of dead skin cells (11).

CONCLUSION

Based on the results of the study, it can be concluded that the shell of oil palm (Elaeis guineensis Jacq.) can be formulated in the form of body scrub preparations and the difference in silica concentration is thought to have the effect of removing dead skin cells.

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