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Effectiveness of silica gel from palm kernel shell ash as a moisture absorber of bottle packaging medicine

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Abstract. Silica gel is widely used in industrial and pharmaceuticals, especially as a moisture absorber. Silica gel can be synthesized from a material containing high SiO₂. One of natural material SiO₂ resource is palm kernel shell ash (PKSA). PKSA contains 55.2% SiO₂. The purpose of this study was to determine the effectiveness silica gel from PKSA as a moisture content absorber for bottle packaging medicine. This study used experimental method with laboratory analysis. The steps in this study were initiated with the preparation of PKSA, synthesis of silica gel from PKSA, characterization of the synthesized silica with X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD) instruments, water content analysis to determine the optimum absorption time of silica gel as a moisture absorber for bottle packaging medicine followed by testing the effectiveness silica gel at the optimum absorption time. The results showed that obtained silica gel from PKSA with a yield of 34%. XRF characterization shows content of SiO₂ by 45.13 %. XRD characterization shows the nature of the solid silica gel in the form of crystalline phase. The optimum absorption time is 2 weeks for silica gel from PKSA. The effectiveness of the silica gel from the PKSA is 85.65 % as bottle packaging medicine moisture content absorber. Further analysis is needed on how to minimize the level of impurities in silica gel of PKSA to increase the effectiveness as a bottle packaging medicine moisture absorber.

1. Introduction

Palm Kernel Shell (PKS) is one of the palm oil processing wastes, which is quite large, which reaches 60% of oil production. Combustion of PKS generated PKSA containing high silica (SiO₂). Zarina [1], studied characterization of PKSA and obtained levels of SiO₂ 51.18%, Al₂O₃ 4.61%, Fe₂O₃ 3.42%, CaO 6.93% and MgO 4.02%. High silica content in PKSA indicated that this material can be used as adsorbent and as SiO₂ source to produce natural silica gel. SiO₂ is a porous solid compound consisting of the elements Silicon and Oxygen bonded covalently. SiO₂ has a large surface area. In addition to composition and polarity, pore structure is also an important factor to consider. The pore structure is related to the surface area, the smaller the pores, the larger the surface area [2].

Silica gel as an adsorbent has been widely known because of its amorphous (unordered) polymer structure, the combination of Si-OH aggregates that form Si-O-Si directed polymers, making this compound has a fairly good stability. Si atoms in silica gel are covalently bonded to four O atoms in a tetrahedral arrangement. Each O atom is covalently bonded to another Si atom to form siloxane (-Si-O-Si-) and silanol (-Si-OH) functional groups. In general, the Si-O bond length is ±0.16nm and the Si-O-Si bond angle is around 1480 [3]. Heating does not change the structure of the silica gel, even when the silica gel has been saturated, heating is often carried out to remove or break the H₂O bonds from the surface of the silica gel.



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Silica gel has wide surface and high adsorption rate to water, so it is widely used in the manufacture of diapers and moisture absorbing agents in the packaging of various shoe products, medicines, and things to prevent mold and damage. Silica gel is a highly porous form of glass-like granule, silica gel is made synthetically from sodium silicate. Silica gel is a natural mineral that is purified and processed into one form of granules or beads. Silica gel has an average pore size of 2.4 nm and has a strong affinity for water molecules [4]. Silica gel can be synthesized from natural material that contains high SiO₂. A study by [5], on the analysis of the absorption of silica gel based on rice husk ash, showed that silica gel from rice husk ash had a greater absorption of water content than synthetic silica gel with a difference in absorption of 0.285%.

Research by Kalapathy [6], on a simple method for the manufacture of silica gel from rice husk ash. The silica gel was then characterized by XRF (X-Ray Fluorescence) to determine the type and amount of SiO₂ and FTIR (Fourier Transform Infra-Red) to determine the functional groups in the silica gel. The silica gel produced has a purity of 93% SiO₂ and 2.6% water. Research by Novita [7], regarding the use of PKSA to improve the quality of used cooking oil, it was found that the SiO₂ content in PKSA was 55.2% from the XRF characterization. Silica gel is widely used, including in the pharmaceutical industry as a moisture content absorber. Therefore, this study aims to utilize PKSA for synthesis silica gel and analysis its absorption effectiveness on bottle packaging medicine.

2. Methodology

2.1. Preparation of PKSA

PKSA is obtained from palm oil processing plant in Siak Regency. PKSA burned in a furnace at temperature of 600 C for 8 hours to remove organic material. After cooling, PKSA then soaked in 1.2 M HCl for 24 hours to dissolve the metal content. After that PKSA then neutralized with NaOH, washed with distilled water, dried at 120°C for 5 hours until a constant weight was obtained [8].

2.2. Synthesis of Silica Gel from PKSA

10 g of PKSA was adjusted to pH 5, stirred for 2 hours and filtered with Whatman 41 filter paper. The residue was washed with distilled water and 60 ml of NaOH was added. Then boil with stir for 1 hour and filter with Whatman 41 paper. Wash the residue. All the filtrates were combined, 1N HCl was added to pH 7. Allow the filtrate to form to form a gel. Centrifuge at 2500 rpm for 15 minutes. Separate the supernatant and wash again. The gel was dried at 80 C.

2.3. Silica Gel Packaging

Silica gel is packed with a weight of 1 g into semi-permeable paper and then tested for its effectiveness in absorbing the moisture content of bottle packaging medicine.

2.4. Silica Gel Characterization

XRD characterization is a crystal structure determination of a solid with X-ray radiation. X-ray scattering is produced when a metal electrode is fired with electrons at high speed in a vacuum tube. A crystal can be used to diffract X-ray beams because the order of the X-ray wavelength is almost the same as or smaller than the order of the distances between atoms in a crystal [9]. XRF (X-Ray Fluorescence) or X-ray fluorescence is an analytical technique that can analyze the elements that build up a material. This technique can also be used to determine elemental concentrations based on the wavelength and amount of X-rays that are re-emitted after a material is bombarded with high-energy X-rays [10].

2.5. Moisture Content Analysis to Determine Optimum Absorption Time of Silica Gel

Silica gel is weighed before packaging. The packaged silica gel was put into a brown bottled medicine with a weight of 150 g medicine. The absorption process is left for 1 week, 2 weeks, 3 weeks and 4

weeks. Silica gel after the absorption process is weighed again. The moisture content of absorption of silica gel in bottle packaging medicine is determined as follows:

$$\text{Moisture Content} = \frac{(\text{g silica gel after absorption}) - (\text{g silica gel before absorption})}{\text{g silica gel before absorption}} \times 100$$

2.6. Determination of Silica Gel Effectiveness at Optimum Absorption Time

The silica gel obtained with the optimum absorption time was heated to a constant weight. Then the process of absorbing the moisture content of the bottle packaging medicine was repeated until 2 times repetition.

$$\%R = \frac{(\text{moisture content after repetition})}{(\text{moisture content before repetition})} \times 100$$

%R = Recovery Percentage

3. Result and Discussion

The results and discussion are based on the methods of this study.

3.1. Synthesis of Silica Gel from PKSA

The first step PKSA is heated in furnace at 600 °C for 8 hours to remove the organic fraction so that only the inorganic fraction remains and to increase the quantity of SiO₂ in the PKSA. The ash produced in this process has a light gray color. PKSA then washing with Hydrochloric Acid (HCl) 1.2 M solution, this process is to eliminate the levels of impurities such as metal oxides. The impurities will form salts and water molecules when reacted with HCl. The resulting salt has a high solubility in water, so the impurities in the form of salt will be lost during the washing process [6].

The next step is to extract the silica contained in the PKSA using 1N sodium hydroxide (NaOH) solution. The extraction process was carried out using a hot plate with constant stirring with a magnetic stirrer for 1 hour at a temperature of ±120 °C. The sodium silicate resulting from the extraction process cooled and filtered to separate the insoluble residue or precipitate from the filtrate in the form of a sodium silicate solution. Then residue was washed with 100 mL of distilled water to optimize the dissolution of sodium silicate. The resulting sodium silicate solution is cloudy white. The mechanism formed during the formation of the sodium silicate is shown in Figure 1 below :

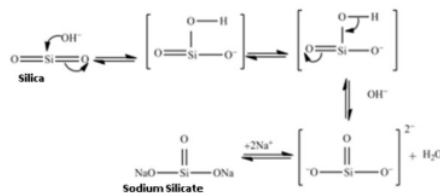


Figure 1. Reaction Mechanism of Sodium Silicate Formation [11]

Based on the reaction, NaOH dissociates to form sodium ions (Na⁺) and hydroxide ions (OH⁻). The hydroxide ion is able to attack the Si atom in SiO₂ to form a silicate. The Si atom in the presence of an empty d orbital is able to accept an electron pair from O to OH⁻. In addition to SiO₂, the high electronegativity of O atoms causes Si to be more electropositive and an unstable intermediate [SiO₂OH]⁻ is formed, resulting in dehydrogenation and the second hydroxyl ion will bind to hydrogen to form a water molecule. Sodium ions will balance the negative charge formed and bind to SiO₃²⁻ ions to form sodium silicate [12].

Al	0,98	%	Al ₂ O ₃	1,67	%
Si	23,56	%	SiO ₂	45,13	%
P	1,83	%	P ₂ O ₅	3,49	%
Cl	69,99	%	NaCl	45,55	%
K	1,27	%	K ₂ O	1,11	%
Ca	1,03	%	CaO	1,03	%
Fe	0,02	%	Fe ₂ O ₃	0,02	%
Zn	0,01	%	Zn	0,00	%

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From table 1 can be seen that the content of SiO₂ in the silica gel from PKSA is 45,13 %. Other compounds which also dominates is NaCl at 45,55 %, because in the process of synthesis of silica gel from PKSA, use of NaOH and HCl as a reagent to form a byproduct in the form of NaCl. The presence of oxides and other elements in silica gel indicates that the process of immersing PKSA using 1.2M HCl is not optimal as well as when washing the soaking PKSA with distilled water. PKSA naturally contains various elements and oxides. Novita [7] characterized PKSA with XRF obtained SiO₂ as 55,2 %, meaning that there is an impurity compound, or another oxide contained in the PKSA.

3.3. XRD Characterization of Silica Gel from PKSA

The results of the characterization of silica gel from PKSA are shown in Figure 5 below:

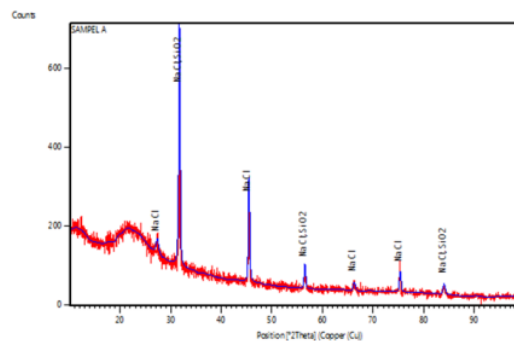


Figure 5. Diffractogram of Silica Gel from PKSA

Figure 5 shows that silica gel solids from PKSA are crystalline solids with the mineral name Cristobalite. In addition, other minerals with solid properties which are also crystalline are also detected, namely solid NaCl. The presence of NaCl in silica gel is a by-product of the silica gel synthesis process using NaOH and HCl as reagents. The purity of the silica gel produced has not been optimized, there are the presence of peaks from other compounds, allegedly caused by a less than optimal ash washing process so that there are other mineral residues left behind [11][13].

Based on its molecular structure, silica is divided into two, namely amorphous and crystalline silica. Amorphous silica is an irregular molecular arrangement silica, in other words, it does not have an arrangement pattern. Crystalline silica is a certain pattern molecular arrangement silica. Crystalline silica is also called polymorph [14,15]. Silica gel with amorphous solid properties has a higher surface area than silica gel with crystalline solid properties. From the XRD analysis, silica gel from PKSA is crystalline so it has a lower surface area. Dipowardhani [16] synthesized crystalline silica using sodium silicate and TEOS as silica sources. The result of silica characterization using XRD analysis method is that the synthesized crystalline silica material is a type of impure stishovit, quartz and cristobalite.

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3.4. Moisture Content Analysis for Determination of Optimum Absorption Time of Silica Gel

The optimum absorption time of silica gel from PKSA can be seen in Figure 6 below :

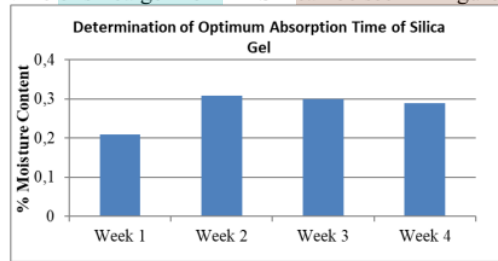


Figure 6. Determination of Optimum Absorption Time of Silica Gel from PKSA

Figure 6 shows the absorption of moisture content in the bottle packaging medicine by silica gel increased from the first week to the second week. Furthermore, there was a decrease in the absorption of silica gel in the third and fourth weeks. In the first week, the moisture content absorbed by silica gel was only 0.2096%. Silica gel had more capacity to absorb moisture content because not all siloxane groups (Si-O-Si) bonded with water molecules to form silanol groups (Si-OH). The optimum absorption of silica gel occurred in the second week of 0.3094%.

The tendency of absorption of silica gel from PKSA after 2 weeks, decreased with increasing absorption time. Based on the XRF characterization data, silica gel from PKSA has impurities in the form of other oxides. The absorption of silica gel PKSA in the fourth week tends to decrease, presumably due to the presence of impurities in the silica gel so that the absorption of silica gel decreases overtime. The mechanism of absorption of water (H₂O) by SiO₂ in silica gel is the formation of hydrogen bonds between water molecules and the oxygen of the silica gel [11].

3.5 Silica Gel Effectiveness As a Moisture Content Absorber At Optimum Absorption Time

The effectiveness of silica gel from PKSA, can be seen in Figure 8 below :

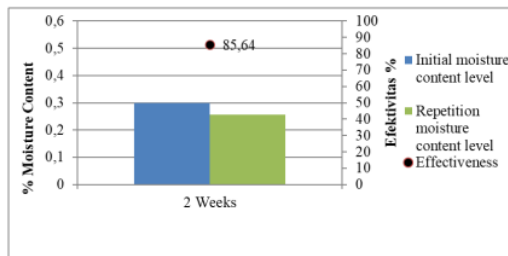


Figure 7. Silica Gel Effectiveness as a Moisture Content Absorber at Optimum Absorption Time

From Figure 7, the effectiveness of silica gel from PKSA is 85.64%. If the silica gel has more silica content, then the silica will bind more -OH and O which comes from the water vapor that absorbs the silica gel. This causes an increase in the absorption of silica gel [5]. From XRD analysis, the solid nature of silica gel of PKSA is crystalline. Amorphous solids have a higher porosity which makes the surface area larger, and the absorption capacity of H₂O also increases. Another advantage of this silica gel is that it comes from natural ingredients, namely PKSA, so it is safer to use. Silica gel in daily applications is used as an adsorbent in food, because it has the ability to absorb moisture so as to prevent food spoilage during storage [17]. Silica gel is also needed to store laboratory equipment and electronic equipment so that they are not damp. Silica gel, which is known as a drying agent, is in great demand by consumers [18].

4. Conclusion

Silica gel from PKSA with a yield of 34%. XRF characterization shows content of SiO₂ by 45.13 %. XRD characterization shows the nature of the solid silica gel in the form of crystalline phase. The optimum absorption time is 2 weeks for silica gel from PKSA. The effectiveness of the silica gel from the PKSA is 85.65 % as bottle packaging medicine moisture content absorber. Further analysis is needed on how to minimize the level of impurities in silica gel of PKSA to increase the effectiveness as a bottle packaging medicine moisture absorber.

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