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MAKING SILICA GEL BY UTILIZING CANDLENUT AND PALM SHELL ASH

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Abstract

Oil palm shell ash which is the result of boiler combustion in industry has the potential to pollute the environment and have a bad impact in the long term so that special treatment is needed. This study aims to answer this problem by making silica gel from hazelnut shell and oil palm shell ash with variations in the type of acid and the ratio of ash to determine the effect of the type of acid and the ratio of the raw material in the form of ash to moisture content and water absorption of silica gel. Oil palm shell ash is used as the main ingredient because it contains the most SiO2 compounds compared to other compounds (reaching 39.78%). Candlenut shell is also used as a raw material for silica gel because it contains silica (12.58%). The stages in this research include the pretreatment process of the material by washing and drying the material, the combustion process of the main ingredient in the furnace, the process of making sodium silicate and the synthesis process of silica gel. Then proceed with water content analysis, analysis of water absorption by silica gel and characterization by Scanning Electron Microscope. The results of this study were that the type of acid, the concentration of the acid and the time of maturation of the gel had an influence on the moisture content of the silica gel and the absorption capacity of the silica gel. The silica gel produced using H_2SO_4 produced more mass than the silica gel using HCl.

Keywords: Silica Gel, Shell Ash, Candlenut, Palm Oil

INTRODUCTION

Currently, more and more industries are using oil palm shells as boiler fuel in their factories because they are cheap and have a high caloric value. This has an impact on the increasing number of pollutants in the form of oil palm shell ash which can pollute the environment. Oil palm shell ash can be categorized as B3 waste (toxic and hazardous materials) (Izzati, 2015) The main components of oil palm shell ash are silica (SiO_2), alumina (Al_2O_3), and iron oxide (Fe_2O_3), while the rest are carbon, calcium, magnesium, and sulfur. The release of combustion ash into the environment can have a bad impact on the environment, so special handling is needed. One of the ways to overcome this problem is by utilizing oil palm shell ash into a new material that has economic value, for example, is silica gel which has been explained in previous research by (Utama, Yamsaengsung, & Sangwichien, 2016).

Oil palm shell ash has the potential as a material for making silica gel because the element of silica is the most dominant component in POMFA (Palm Oil Mill Fly Ash)

which is 39.02% by weight, followed by Oxygen (30.78% by weight) and Carbon (30.44). % by weight). Besides, palm oil shell ash can be used as an alternative to making silica gel apart from rice husks and quartz sand.

Candlenut (*Aleurites moluccana*), which in English is called candlenut, belongs to the Euphorbiaceae family and the Crotonoideae sub-family.(INDRA, 2020) This plant develops in Indonesia in areas such as West Sumatra, Bengkulu, Lampung, South Sumatra, East Sumatra, Bali, Maluku, West Kalimantan, and its surroundings. Candlenut fruit is round, soft, and slightly flattened. Candlenut fruit has 1-3 spaces containing candlenut seeds. The fruit is green when young and brown when ripe. The shell or skin of the fruit is about 5-7 mm thick. When viewed as a whole, there is the outer skin, pulp, wood layer, shell of the seed shell, and the flesh of the seeds. (Parwati & Suparno, 2017). Candlenut shells are used in this study because the ash of candlenut shells has a silica content of 12.58%.

Silica gel is a synthetic compound that has an amorphous structure and is usually synthesized through a sol-gel process by agglomerating sodium silicate ($NaSiO_2$) with certain types of acids (such as HCl) (Abdurrachim, 2016). Silica gel has the chemical formula $SiO_2.xH_2O$ Silica gel is in the form of a solid and is commonly used as an absorbent to absorb moisture. Silica gel can be applied to maintain moisture in food, jewelry, medicines, and electronic objects. (Fahmi & Nurfalah, 2016) Silica gel that cannot absorb water can be regenerated by absorbing water (by using an electric heating furnace, the residual heat of exhaust gas, or drying hot air) or by the rinse method (by immersing silica gel in saturated steam then rinsing hot water), so that silica gel is water-free and can absorb water again (Syukri, Hindryawati, & NS, 2017).

The process of synthesizing silica gel begins with making an acid solution. Then, the acid solution was added to the sodium silicate solution drop by drop to pH 7 while stirring with a magnetic stirrer until it formed a gel. Then, the gel that is formed is left to stand for 3 hours for the gel maturation process. After that, the gel has been cooked in the oven at a temperature of 80°C. Then, repeat the steps from the beginning for different variables in the silica gel synthesis process (Syukri et al., 2017)

In a research conducted by (Alkatiri, Insanii, Marjuki, & Fitriyah, 2017), he produced silica gel from oil palm shell ash with a purity level of 92% by applying a leaching process using citric acid (Utama et al., 2016). Apart from oil palm shells, candlenut shell ash also contains 12.58% by weight of silica-based on data from the Palembang Industrial Research and Standardization Center.

Research on the manufacture of silica has been carried out, including (Febriyanti, Zaharah, & Wahyuni, 2014) who succeeded in modifying silica gel from glass waste using tributylamine with a percentage of SiO_2 in silica gel of 74.92% which was proven by X-Ray Fluorescent (XRF) analysis. . Also, research by (Sari, Azmiyawati, & Taslimah, n.d.) has succeeded in synthesizing silica gel with modification by γ-Glycidoxyopropyltrimethoxysilane and Mercaptobenzothiazole to adsorb Cadmium metal.

This success is marked by the results of the analysis using the Atomic Absorption Spectrophotometer (AAS) which shows that a large amount of Cd (II) metal adsorbed by the second adsorbent is for a ratio of 0.05 grams of adsorbent in 10 mL of solution, which is 0.53786 mmol / L or equal to 0.10757 mmol / g with an adsorption power of 98.64%. Besides, (Hayati, Pardoyo, & Azmiyawati, 2017) has also succeeded in synthesizing Nanosilica from rice husk ash. The success of this research is evidenced by the results of the analysis in the form of SEM images of Nanosilica which show the nano-silica synthesized using H_3PO_4 which has a particle morphological size. a surface that is 40-60 nm.

In this research, silica gel synthesis will be carried out using the method used by (Handayani, Nurjanah, & Rengga, 2014) with different samples, namely candlenut shell ash and palm oil shell ash and the variables to be studied are the type of acid used (hydrochloric acid and sulfuric acid) and the fraction of cadlenut ash and oil palm shell ash. Furthermore, the resulting silica gel will be analyzed for moisture content and water absorption. Then continued with silica gel characterization with analysis with Scanning Electron Microscope (SEM) to determine the size of surface morphology and Infra-Red Spectrophotometer to determine silanol (Si-OH) and siloxane (Si-O-Si) groups.

This research is a new study because it uses a mixture of candlenut shells and oil palm shells. The reason for choosing oil palm shell ash was because the silica element was the most dominant component in POMFA (Palm Oil Mill Fly Ash), which was 39.02% by weight. (Utama et al., 2016). Then, candlenut shell ash also has a silica content of 12.58% by weight based on data from the Palembang Industrial Research and Standardization Center.

RESEARCH METHODS

The methodology in this study begins with a material pretreatment process by sifting the candlenut shell ash and palm shell ash. Then, the process of making sodium silicate and synthesizing silica gel is carried out. Furthermore, to determine the characteristics of silica gel, analysis of water content and analysis of water absorption by silica gel was carried out.

The approach used in this research is a quantitative approach which is carried out by calculating the water content and water absorption capacity of silica gel that has been made from the results of the research practicum. The data obtained are in the form of water content and water absorption values which are in percent units. After the data is obtained from eight variables, it will be compared between one variable and another and compared based on the theory of the effect of the type of acid, concentration and duration of maturation of the gel on the amount of silica obtained, the absorption capacity and moisture content of the silica gel.

This research was conducted at the Chemical Analysis Laboratory at the Faculty of Vocational School, Diponegoro University. While the characteristics of silica gel are carried out in the Integrated Laboratory of the Technical Service Unit of Diponegoro University. The samples used were palm shell ash and candlenut shell ash which would then be mixed with a ratio of palm kernel shell: candlenut, which was 3: 1. After that, eight different variables will be created. The variables used were acid type (HCl and H_2SO_4), acid concentration (1 and 2 N) and gel maturation time (3 and 5 hours). Data collection was carried out by carrying out a silica gel making practicum. The data obtained after carrying out the practicum are in the form of tables of observations of acid requirements, observations of water content, observations of water absorption, SEM microscope appearance photos and infrared spectra charts. The data analysis technique is done by comparing the results of the research with the theory and research that has been carried out.

RESULTS AND DISCUSSION

A. Synthesis of Silica Gel

Silica gel was synthesized using the sol-gel method with precursor silicon dioxide (SiO_2) which was extracted from palm shell ash and candlenut shell ash by dissolving a mixture of palm shell ash and candlenut shell ash into 2.5 N NaOH solution. clear in color and a pH value of 12.After the sodium silicate solution is produced, the next step is

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to add the acid solution which is added dropwise to adjust the pH level 7. This aims to make the

reaction fast and stable because the optimal conditions occur at pH 7 when the silanol (Si-OH) and Siloxan groups (Si-O-Si) is in the same amount. In this study, 8 variables were used as listed in table 1.

Table 1 Research Experiment Design

Run	Type of Acid	Normality (N)	Gel Curing Time (Hour)	Output
1	HCl	1	3	Moisture Content and Water Absorption
2	H_2SO_4	1	3	
3	HCl	1	5	
4	H_2SO_4	1	5	
5	HCl	2	3	
6	H_2SO_4	2	3	
7	HCl	2	5	
8	H_2SO_4	2	5	

With variations in differences in acids (HCl and H_2SO_4), differences in acid concentration (1N and 2N) and the duration of gel maturation (3 hours and 5 hours), the results will be compared, including moisture content and water absorption of silica gel.

A. Effect of Acid Type, Concentration And Maturation Time on Silica Gel Mass

Table 2 Mass Results of Silica Gel

Run	Type of Acid	Concentration (N)	Gel Curing Time (Hour)	Volume of Acid Requirements (mL)	pH	Silica Gel Results (g)
1	HCl	1	3	10	7	2,11
2		1	5	12	7	2,65
3		2	3	9	7	2,44
4		2	5	7	7	2,95
5	H_2SO_4	1	3	7,5	7	3,02
6		1	5	8	7	3,27
7		2	3	5	7	3,54
8		2	5	4,5	7	4,63

Based on the results of table 2, it can be seen that the silica gel obtained using sulfuric acid obtained more mass when compared to the silica gel obtained using hydrochloric acid. The highest yield of silica was in the H_2SO_4 2N variable with a maturation time of 5 hours with a mass of 4.63 grams, while the silica gel with 1N HCl variable with a maturation time of 3 hours had the lowest yield of 2.11 grams.

This is due to the difference in the number of H + ions. Acids act as catalysts and reactants. The greater the number of H + ions, the more efficient its role as a catalyst will be so that it can produce more silica gel. The more efficient the catalyst is, the less it is used for the reaction process. This is shown by the data in table 4.2 which shows that the need for H_2SO_4 to synthesize silica gel is less when compared to the need for HCl. However, using H_2SO_4 can produce more silica gel than using HCl.

Then, in the data table above, it can be seen that the relationship between acid

concentration and gel maturation time is directly proportional to the amount of silica gel produced. This happens because the amount of acid concentration is increasing and the longer the maturation time of the gel causes more silica gel to be produced.
Then, the factors that influence the results of this study are the accuracy in the practicum, the condition of adequate tools and appropriate practicum procedures.

B. Effect of Acid Type, Concentration And Gel Maturation Time on Silica Gel Moisture Content

Table 3 Results of the water content of the synthesized silica gel

Run	Type of Acid	Concentration (N)	Gel Curing Time (Hour)	Water content (%)
1	HCl	1	3	10,89
2		1	5	8,95
3		2	3	10,63
4		2	5	10,04
5	H_2SO_4	1	3	10,21
6		1	5	8,46
7		2	3	8,95
8		2	5	9,02

Based on table 3, it is obtained data that the silica gel produced using hydrochloric acid (HCl) has a higher water content than the silica gel produced using sulfuric acid (H_2SO_4).

Silica gel with the greatest moisture content is found in silica gel with 1N HCl variable with a maturation time of 3 hours has a moisture content of 10.89%. While the silica gel with the least water content found in silica gel with a variable H_2SO_4 1N with a maturation time of 5 hours had a moisture content of 8.46%. This is because the need for the acid solution used in the use of HCl solution is more than the use of H_2SO_4 solution. Thus, the water content in silica gel using H_2SO_4 solution is less when compared to silica gel using HCl solution.

Then, in the table above, it can be seen that the acid concentration and gel maturation time are inversely proportional to the moisture content in the resulting silica gel. This is because the more concentration, the less water content of the silica gel is produced. Then, the longer the gel ripening time also causes less water content of the silica gel to be produced.

Then, the factors that influence the results of this study are the accuracy in the practicum, the condition of adequate tools and appropriate practicum procedures.

C. Silica Gel Adsorption Power Test Results

Table 4 Silica Gel Absorption Power Test Data

Run	Type of Acid	Concentration (N)	Gel Curing Time (Hour)	Water Adsorption Power (%)
1	HCl	1	3	56,44
2		1	5	56,91
3		2	3	53,84
4		2	5	54,44
5	H_2SO_4	1	3	58,33
6		1	5	60,82
7		2	3	54,64
8		2	5	54,37

Based on table 4, the research results show that the silica gel produced using sulfuric acid (H_2SO_4) has more water absorption when compared to the silica gel produced using hydrochloric acid (HCl). Silica gel which has the greatest adsorption power is found in the H_2SO_4 1N variable with a gel maturation time of 5 hours with a water adsorption power of 60.82%. Then, silica gel which has the smallest adsorption power is found in silica gel with HCl 2N variable with 3 hours of gel maturation time has a water adsorption power of 53.84%. This is because the low water content in the silica gel causes a tendency to absorb more water. So it can be concluded that the less water content, the more water absorption of the silica gel.

Then, in the data above, it can be seen that the less acid concentration, the greater the water absorption. However, the gel ripening time did not significantly affect water absorption. Then, the factors that influence the results of this study are the accuracy in the practicum, the condition of adequate tools and appropriate practicum procedures.

D. Characterization of Silica Gel

1) Characterization of Silica Gel by Scanning Elecron Microscope (SEM)

Silica gel characterization by Scanning Elecron Microscope (SEM) was carried out to determine the surface morphology of silica gel. This characterization was carried out at the Diponegoro University Integrated Laboratory Unit.

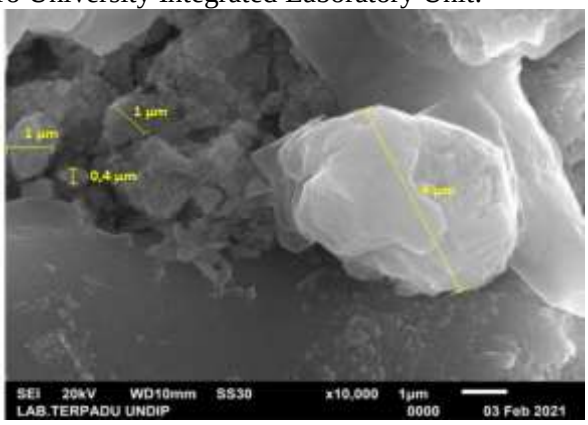

Figure 1 SEM image of silica gel

From the SEM image, it was found that silica gel with 2N HCl variable with a gel maturation time of 5 hours was successfully synthesized with hydrochloric acid catalyst

with a surface morphological size of 0.4 to 4 µm.

2) Characterization of Silica Gel with Infra-Red Spectrophotometer (FTIR)

Characterization of silica gel with an IR Spectrophotometer (FTIR) was carried out to determine the silanol (Si-OH) and siloxane (Si-O-Si) groups in the silica gel. This characterization was also carried out at the Diponegoro University Integrated Laboratory Unit. Silica gel IR spectra image can be seen in Figure 2 Silica Gel IR Spectra.

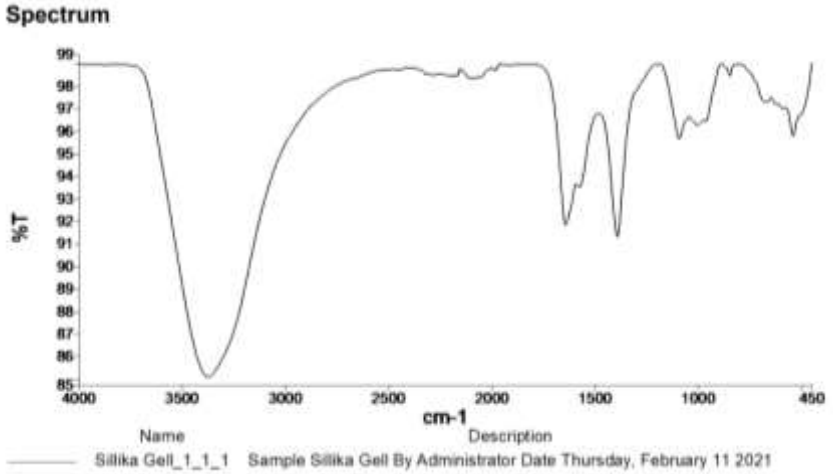

Figure 2 Silica Gel IR Spectra

According to the theory presented in the research by (Hayati et al., 2017), it is explained that the results of silica gel characterization with an IR Spectrophotometer will obtain two possibilities. The first possibility is that the silica gel will be dominated by silanol (Si-OH) groups as evidenced by the absorption at the 900s wave number which shows the vibration of the silanol (Si-OH) group bonds. Then, the second possibility is that the silica gel will be dominated by siloxane groups (Si-O-Si) which is proven by not showing the absorption at the wave number 900's which shows the vibration of the cyanol (Si-OH) group bonds.

Figure 2 shows the IR spectra of variable silica gel HCl 2N with a gel maturation time of 5 hours. When viewed from the IR spectra, it shows that the silica gel synthesized with HCl is dominated by siloxane groups (Si-O-Si). This is evidenced by the absence of absorption at the 900's wave number which shows the vibration of the cyanol (Si-OH) group bonds.

Based on the results of IR spectra on the variable silica gel HCl 2N with a gel maturation time of 5 hours, the absorption band widened at a wave number of 3360.67 cm-1 which showed the vibration of the –OH group strain from Si-OH. Then, the broad absorption band at wave number 1638.99 shows the bending vibration of O-H from H2O. Then, the widened absorption band at wave number 1091.34 indicates the presence of an asymmetric stretching vibration of the Si-O group from ≡Si-O-Si≡. After that, the broad absorption band at the number 803.12 indicates a symmetric stretching vibration of the Si-O group from Si-O-Si≡. Then, the absorption band that widened in the waveform 539.20 showed a bending vibration of ≡Si-O-Si≡.

CONCLUSION

Based on the results of the study, it was concluded that the type of acid, the

concentration of the acid and the time of maturation of the gel had an influence on the moisture content of the silica gel and the absorption capacity of the silica gel. The silica gel produced using H_2SO_4 produced more mass than the silica gel using HCl. Then, the concentration and maturation time of the gel are directly proportional to the mass of silica gel produced. The silica gel produced using H_2SO_4 produced silica gel with the least water content compared to the silica gel produced using HCl. Thus, the silica gel produced using H_2SO_4 has a higher water absorption when compared to the silica gel produced with HCl. This is because the less water content of the silica gel has a tendency to absorb more water.

In this study, the largest mass of silica gel was obtained in the H_2SO_4 2N variable with a gel maturation time of 5 hours with a mass of 4.63 grams, while the smallest mass of silica gel was in the 1N 3 hours HCl variable with a mass of 2.11 grams. Then, the silica gel which has the most moisture content is 10.89% in silica gel with 1N HCl variable with a maturation time of 3 hours, while the silica gel which has the lowest water content is 8.46% in silica gel with H_2SO_4 variable. 1N with a gel maturation time of 5 hours. Silica gel which has the greatest water adsorption power of 60.82% is found in silica gel with a variable H_2SO_4 1N with a gel maturation time of 5 hours, while silica gel with the smallest adsorption power of 53.84% is found in silica gel with variable 2N HCl with a gel ripening time of 3 hours.

In this study, 2N HCl variable silica gel with a maturation time of 5 hours was characterized by a Scanning Electron Microscope having a surface morphological size of 0.4 to 4 µm. Characterization by IR spectrophotometer showed that the variable silica gel HCl 2N with a maturation time of 5 hours was dominated by siloxane groups (Si-O-Si). This is evidenced by the absence of absorption at the 900's wave number which shows the vibration of the cyanol (Si-OH) group bonds.

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