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Synthesis and Characterization of Silica Gel from Palm Shell and Coir Ash

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Abstract

This study aims to determine the composition of the oxide, the characteristics of palm shell and coir, and silica gel synthesized. The oxide composition, crystallinity, palm shell and coir ash functional groups were analyzed using XRF, XRD, and FTIR. Analysis of the functional groups and crystallinity using FT-IR and XRD. The results showed the composition of SiO_2 in palm shell and coir ash was 76%, and SiO_2 in the form of quartz and cristobalite was more dominant than amorphous SiO_2. The synthesis success was indicated by the appearance of peaks with low intensity, not sharp, and resembling bumps on the synthesized silica gel diffractogram, which is an amorphous SiO_2 characteristic. This result is confirmed by FT-IR, where absorption at wave number 455 cm^{-1} is a Si-O-Si bending vibration, 783 cm^{-1} is a stretching vibration of Si-O symmetry of Si-O-Si, 3454.51 $cm^{-1,}$ and 3568 cm^{-1} with fairly high intensity are characteristic of the -OH group from Si-OH. The wide and sharp absorption at 1083.99 cm^{-1} is a stretching vibration of the Si-O symmetry of Si-O-Si, 1625.99 cm^{-1} is -OH vibration of water molecule, 798.53 cm^{-1} is Si-O stretching vibration of Si-O-Si and absorption of 462.92 cm^{-1} indicates bending vibration of Si-O-Si.

Keywords: Silica gel, palm shell and coir ash, XRF, XRD, FTIR

INTRODUCTION

Along with the increasing production of palm oil from year to year, the volume of waste has also increased. The waste is in the form of liquid and solid waste. Solid waste in the form of: coir, shells, and empty palm oil bunches. There are two things that need to be done with palm oil waste when it is in the environment, namely handling it to prevent adverse effects on the environment, and secondly is utilization to obtain economic value from the waste (Sulhatun, 2012). Solid waste can be converted into useful materials such as activated carbon because it has a high lignocellulosic content. Haji (2013) states that palm oil solid waste contains hemicellulose, cellulose, lignin, ash, and other components, respectively: 24%, 40%, 21%, and 15%. Activated carbon from palm shells can absorb hazardous liquid waste in the environment, such as laundry liquid waste (Sunarti & Kadtabalubun, 2023; Taufik et al., 2021).

The palm fruit processing factory utilizes palm coir and shells as boiler fuel to produce hot steam to drive turbines to generate electricity. Burning oil palm shells and coir in a furnace at a temperature of 800-1000 ^oC (Toniolo & Boccaccini, 2017) produces ash with a light fraction known as palm fly ash (POFA), and a heavy fraction that settles at the bottom of the furnace known as palm bottom ash (Zarina et al., 2013). The problem that arises from the ashes is that the amount increases every time, and their utilization is not optimal. The resulting ash is usually thrown away around the palm oil processing factory area and causes environmental problems (Ranjbar et al., 2014).

Increasing the palm shell and palm coir ash value can be utilized to become a material with economic value, namely silica (Nguumbur Iornumbe et al., 2021). This idea is based on the main content: silica with the highest composition and a small amount of alkali oxides and impurities and stated. That palm coir ash and palm shells ash contained 58.02% and 61%, respectively.

One of the silica-based materials is silica gel. Silica gel is a solid product of the reaction between HCl and sodium silica (Na_2SiO_3). It undergoes polymerization and is followed by condensation to produce $Si(OH)_4$ gel in the sol-gel process (Besbes et al., 2009). The same is true when synthesizing silica gel is carried out. By smelting siliceous ash with NaOH, sodium silicate is formed. If a sodium silicate solution is added to an acid solution, a condensation reaction will occur from the silicate to form a gel (Mujiyanti et al., 2010).

Silica, as the main ingredient in the manufacture of silica gel, can be extracted from materials containing silica. Hanum et al. (2022) successfully extracted silica from coal fly ash using NaOH and KOH bases by treating various concentrations and reaction times with NaOH and KOH. The extraction was carried out on different fly ash samples coded CFA A and CFA B. Based on the study results. It was found that more CFA was extracted at an extraction time of one hour and a concentration of 1M for KOH solvent. The synthesis of silica gel in this study used palm shell and palm coir ash from the palm processing factory of PT. Nusa Ina Group, East Kobi North Seram District, Central Maluku Regency. The ash and silica gel characteristics from palm shell and palm coir were analyzed using XRF, XRD, and FTIR.

METHODOLOGY

Materials and Instrumentals

The tools used in this study were: Glassware (Pyrex), Magnetic stirrer, Oven (Memert), XRD (Shimadzu XRD 6000), XRF (Thermo Scientific), Crucible, FTIR (Shimadzu 8201PC), Shaker, pH meter, Pipette, Furnace, Sieve 100 mesh. The materials in this study were: Coir ash and palm shells from PT. Nusa Ina North Seram Central Maluku, 3 M HCl; NaOH 0.1; 4 M, distilled water, Whatman filter paper 42.

Preparation and activation of palm shell and coir ash

Palm shell and palm coir ash were crushed and sieved through a 100-mesh sieve. Furthermore, 40 grams of sifted ash was washed with 300 mL of 2 M HCl. The washed ash was then rewashed with distilled water until neutral. Dried in an oven at 100 ^{o}C until constant weight. The ash was then analyzed by XRF, XRD, and FT-IR (Syukri et al., 2017).

Preparation of sodium silicate

About 20 grams of activated palm coir and shell ash were mixed with 158 mL of NaOH. The mixture was boiled at 85 ^{o}C for 120 minutes while stirring with a magnetic stirrer. After drying slightly, the solution was transferred to a crucible and melted in a furnace at 500 ^{o}C for 30 minutes. After cooling, the ashes resulting from the smelting were added to 200 mL of distilled water and left overnight. The mixture was left overnight. The precipitate was filtered using Whatman 42 filter paper. The resulting filtrate was a sodium silicate solution from palm shell and coir (Yusrin et al., 2014).

Preparation of Silica Gel

Amount 40 mL of sodium silicate solution from palm shell and coir ash was added with 3 M HCl solution (drop by drop), stirring until a gel formed and continued until pH 7. The hydrogel was put in the oven and heated at 80 ^{o}C for 18 hours to produce a gel. Dry silica. The dry silica was crushed, then washed with distilled water until the water used for washing was neutral. The next step was to put it back in the oven and heated at 80 ^{o}C for 9 hours or until the dry silica gel re-formed. Dry silica was crushed and sieved using a 100-mesh sieve. The result of the sieve is a silica gel of coir ash and palm shells. Silica gel was analyzed using XRD to determine its crystallinity and FTIR to determine its functional groups (Yusrin et al., 2014).

RESULTS AND DISCUSSION

Preparation and activation of Palm Shell and Coir Ash

Palm shells and coir ash are used from PT. Nusa Ina Group, North Seram District, East Kobi, Central Maluku Regency, Maluku Province, was initially crushed to reduce the particle size. The ash was then sieved through a 100-mesh sieve to homogenize the particle size.

The palm shell and coir ash activation was done by soaking the ashes with HCl. Washing ash with HCl aims to remove impurities in the form of metal oxides, Fe_2O_3, CaO, and other impurities. In addition, washing with acid can maintain the biogenic structure and increase the silica content of the oil palm shell ash. Basic metal oxides can be analyzed with HCl to form salts and water molecules. SiO_2 contained in palm shells and coir ash cannot be dissolved in HCl because SiO_2 is an acidic oxide sensitive to alkaline solutions, so SiO_2 is still present in the residue (Nur'aeni et al., 2017). The salt produced in this washing process has excellent solubility in air, so the metal oxide, which is an impurity in the form of chloride salt, will dissolve. The next stage is selling the residue and filtrate, then washing it with distilled water until the pH is neutral. The residue obtained was heated in an oven to remove water molecules. Activated ash samples were analyzed using XRF, FTIR, and XRD.

Analysis of the composition of the ash oxide resulting from activation

The XRF analysis is a qualitative and quantitative analysis method. Qualitatively XRF identifies the chemical species in the sample and quantitatively analyzes the number of each chemical

species present in a substance. Collisions between atoms on the sample surface caused by X-rays are the basis for Analysis with XRF (Mujiyanti et al., 2021). The XRF analysis was conducted to determine the mineral oxides in palm shells and coir ash after the activation process using 2M HCl. Based on the analysis results, the oxides' composition in the activated ash was obtained, as presented in Table 1.

Table 1. Chemical composition of palm shells and coir ash.

Compounds	Content %
SiO_2	76%
P_2O_5	1.70%
K_2O	12.60%
CaO	4.37%
TiO_2	0.49%
V_2O_5	0.006%
MnO	0.17%
Fe_2O_3	4.61%
CuO	0.247%
ZnO	0.054%
Rb_2O	0.078%
SrO	0.055%
ZrO_2	0.037%

The results of the XRF analysis showed that the SiO_2 content in palm shells and coir ash was 76%. The acid concentration affects the percentage of SiO_2 and the dissolution of metal oxides in the palm shell and coir ash. The higher the acid concentration, the higher the dissolution of metal oxides, and the concentration and purity of SiO_2 will increase (Laharimu et al., 2019).

Functional group analysis of shell ash and oil palm coir

Identify functional groups of SiO_2 in palm shell and coir ash using FTIR. Data analysis results with FTIR are presented in Figure 1. The FT-IR spectra in Figure 1 compare the results of rice husk ash and palm shell and coir ash in the research samples. The visible silica absorption patterns are generally silanol (Si-OH) and siloxane (Si-O-Si) groups. The two pictures above have similarities and differences in wave number absorption bands. The wave number similarities of the two are located in wave numbers 455 cm^{-1} and 466 cm^{-1}, Si-O-Si bending vibrations, wave numbers 783 $cm^{-1,}$ and 798 cm^{-1} the Si-O-Si stretching vibration of Si-O-Si. With a fairly high intensity, the wave numbers 3468 cm^{-1} and 3568 cm^{-1} contained -OH groups from Si-OH.

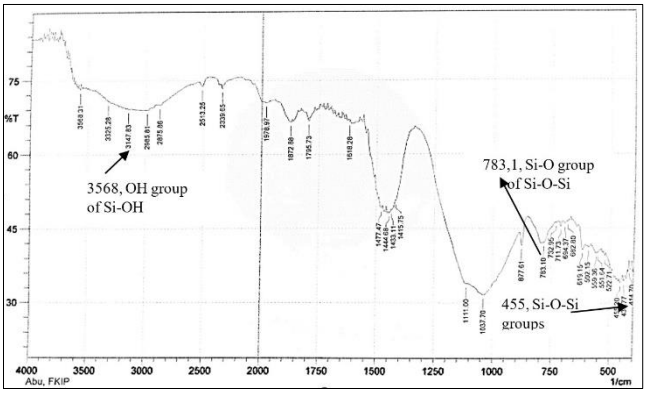

Figure 1. The FT-IR Spectra of Palm Shell and Coir Ash.

Crystallinity analysis of palm shell and coil ash

The crystallinity of palm shells and coir ash can be known from the analysis results using XRD. Diffractogram data can be seen in Figure 2. The diffractogram in Figure 2 shows that palm shells and coir ash have a diffraction pattern of sharp peaks and high intensity. These sharp peaks are identified as the peaks of quartz and cristobalite. The peak for -peak for quartz occurs at 2θ = (20.7469; 26.4666; 40.2578; 42.3227; 50.0645; 60.0120; 68.1050; 81.3055), whereas for cristobalite 2 θ = (21.7972; 31.1986; 36.5604). Peaks with low intensity and in the form of mounds were identified as amorphous SiO_2.

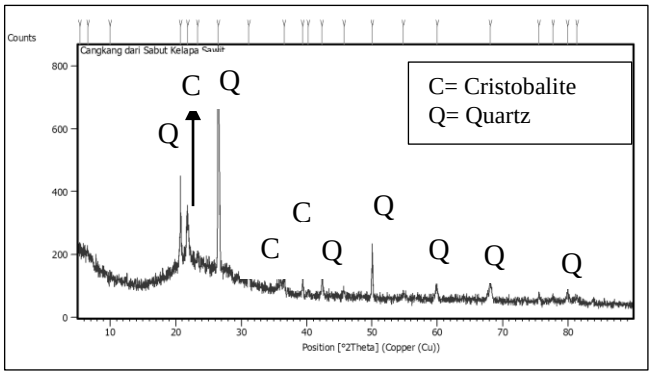

Figure 2. The Diffractogram of palm shell and coir Ash

Based on the diffractogram data above, it can be concluded that the crystalline phase SiO_2 in palm shells and coir ash is higher than the amorphous phase. The dominance of the crystalline phase is caused by the firing temperature of palm shells and coir ash in the furnace to heat the kettle, where at temperatures of 800-900 is dominated by SiO_2 cristobalite, at temperatures more than 900 ^oC is dominated by silica quartz (Trivana et al., 2015), whereas at temperatures of 500-600 ^oC, SiO_2 is stable in the amorphous phase (Mujiyanti et al., 2010).

Manufacturing of Sodium Silicate

The material in the manufacture of silica gel is sodium silicate. Sodium silicate solution was prepared by dissolving shell ash and palm coir with NaOH and heating at 85 °C. NaOH will dissolve SiO_2 in the ash, and heating will complete the dissolving process. The heating temperature must be kept stable to avoid excessive solution evaporation (Fathurrahman et al., 2020). Then the solution was melted at 500 °C. Melting at this temperature causes NaOH to completely dissociate to form Na^+ and OH^- ions (Trivana et al., 2015). Dissolution followed by melting will increase or accelerate the formation of sodium silicate (Mujiyanti et al., 2010). The predicted reaction mechanism for the construction of sodium silicate is shown in Figure 3.

Melting at high temperatures causes NaOH to dissolve and completely dissociate, forming Na^+ and OH^- ions. The high electronegativity of the O atom in SiO_2 causes Si to be more electropositive. The unstable $[SiO_2OH]^-$ intermediate is formed, and dehydrogenation will occur. The second OH^- ion then binds to hydrogen to form a water molecule, and two Na^+ ions will balance the negative charge of the SiO_3^{2-} ion to form sodium silicate (Mujiyanti et al., 2010).

Preparation of Silica Gel

Silica gel was prepared by adding 3 M HCl solution. Sodium silicate solution has a pH of 11-12 so adding HCl forms silicic acid monomers to form a gel. In this process, the formation of H_2SiO_3 occurs, followed by the reaction of forming an acid sol $Si(OH)_4$. The reaction between sodium silicate and HCl can be assumed as follows (Yusrin et al., 2014):

$$Na_2SiO_{3(l)} + 2HCl_{(aq)} \rightarrow H_2SiO_{3(aq)} + 2NaCl_{(s)}$$
$$H_2SiO_{3(aq)} + H_2O_{(l)} \rightarrow Si(OH)_{4(aq)}$$

According to Scot (Meilina, 2010), the reaction mechanism for silica gel's formation is shown in Figure 4. Adding HCl causes the concentration of H^+ in the sodium silicate solution to increase. This condition causes the silicates to turn into silicic acid (H_2SiO_3), which results in siloxane groups (Si-O-) forming silanol groups (Si-OH). The silanol groups polymerize by forming ≡Si-O-Si≡ cross-links to form silica gel (Yasrin et al., 2020).

The absorption pattern of silica gel from palm shell and coir ash is similar to that of Utama et al. (2018). The similarity is where a broad absorption at wave number 3454.51 cm^{-1} indicates -OH vibrations of Si-OH, absorption wide and sharp at 1083.99 cm^{-1} indicates the presence of Si-O symmetrical stretching vibrations of Si-O-Si, and absorption at 1625.99 cm^{-1} indicates the presence of -OH vibrations of water molecules. The absorption band at 798.53 cm^{-1} indicates Si-O stretching vibrations from Si-O-Si, and the absorption band at 462.92 cm^{-1} indicates bending vibrations from Si-O-Si. Analysis using XRD aims to determine the characteristics of silica gel crystals. The synthesized silica gel diffractogram is presented in Figure 6.

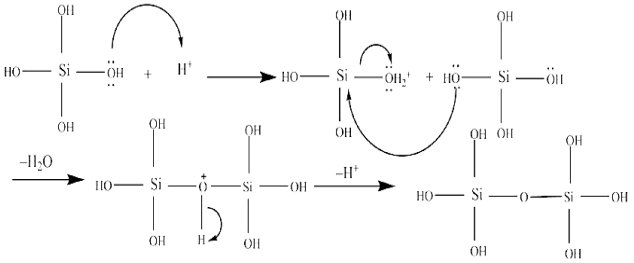

Figure 4. The reaction mechanism for the formation of silica gel

Characterization of Silica Gel

FTIR and XRD carried out the characterization of silica gel results. Characterization of silica gel with FTIR aims to determine the presence of functional groups in silica gel, namely silanol (Si-OH), siloxane (Si-O-Si), and other groups. The results of the Analysis are presented in Figure 5.

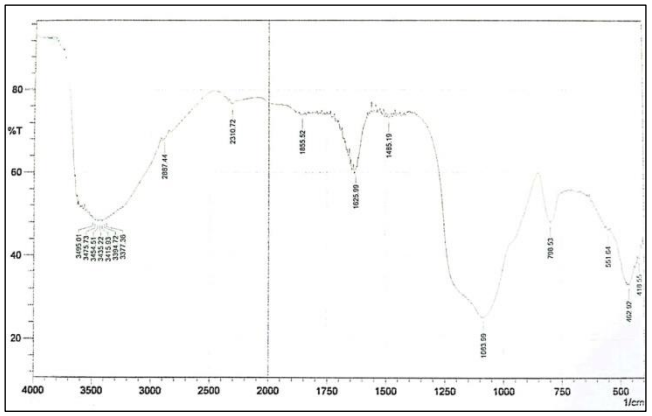

Figure 5. Infrared spectra of silica gel from palm shell and coir ash

The diffraction pattern of silica gel from silica gel palm shells and coir ash is shown in Figure 6. There are no sharp peaks and low intensity. The peaks of crystalline SiO_2 in the form of crystobalite and quartz did not appear, and the amorphous form of silica was dominant. Widened and mound-like peaks at 2θ between 15° - 22.4° are characteristic of amorphous SiO_2. This result is consistent with the main study by et al. (2018), where broad peaks show

the characteristics of amorphous SiO2 at a diffraction angle of 2θ between 15° to 35°.

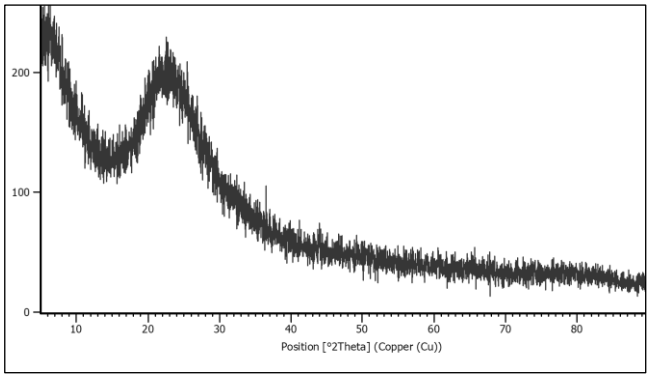

Figure 6. The silica gel of palm shells and coir ash diffactogram

CONCLUSION

The composition of SiO_2 in shell and palm coir ash from PT Nusa Ina Group is 76%, dominated by SiO_2 minerals in quartz and cristobalite. Si-O-Si bending vibration was identified at wave number 455 cm^{-1}, wave number 783 cm^{-1} is a symmetrical stretching vibration of Si-O from Si-O-Si, 3568 cm^{-1} with a fairly high intensity indicates the presence of -OH groups from Si-OH. The synthesized silica gel is amorphous. The silanol and siloxane functional groups identified at wave number 3454.51 cm^{-1} are the -OH vibrations of Si-OH, the wide and sharp absorption at 1083.99 cm^{-1} is the stretching vibration of Si symmetry -O of Si-O-Si, 1625.99 cm^{-1} is -OH vibration of a water molecule, 798.53 cm^{-1} is stretching vibration of Si-O of Si-O-Si, and absorption at 462.92 cm^{-1} is the bending vibration of Si-O-Si.

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