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Preparation and Characterization of Silica Composite Adsorbent from Palm Oil Boiler Ash and Chitosan

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ABSTRACT

FTIR characterization of silica confirmed the presence of hydroxyl groups (-OH) at wave number 3386 cm^{-1}. Wave number 1059 cm^{-1} Si-O-Si group, 783 cm^{-1}, 984 cm^{-1} symmetric stretching vibration of Si-O-Si group, wave 452 cm-1 Siloxane group, in silica chitosan composite wide absorption band at 3356 cm^{-1} (-OH) group from silanol group (Si-OH) which is a functional group derived from silica as well as (-OH) and ($-NH_2$) groups, 1628 cm^{-1} amide group (-NH) wave 1421 cm^{-1} related to symmetric bending vibration of chitosan oligosaccharide chain namely $-CH_2$ and $-CH_3$ groups which can be influenced by the presence of silica matrix 1079 cm^{-1} Si-O-Si group, wave 593 cm^{-1} and 488 cm^{-1} bending vibration of Si-O-Si and Si-O. The optimum result of Pb^{2+} adsorption using silica chitosan composite adsorbent was 94.70% with an absorption capacity of 1.2467 mg/g. With a concentration of 5.266 ppm it became 0.279 ppm.

Keywords: Adsorbent, Silica-chitosan Composite, Sol-Gel

1. INTRODUCTION

Palm oil boiler ash is waste from palm oil production, based on the analysis results showing the composition of Silicon dioxide (SiO_2) 76%, Phosphorus Pentoxide (P_2O_5) 1.70%, Potassium Oxide (K_2O) 12.60%, Calcium Oxide (CaO) 4.37%, Titanium Dioxide (TiO_2) 0.49%, Vanadium Pentoxide (V_2O_5) 0.006%, Manganese (II) Oxide (MnO) 0.17%, Iron (III) Oxide (Fe_2O_3) 4.61%, Copper (II) Oxide (CuO) 0.247%, Zinc Oxide (ZnO) 0.054%, Rubidium Oxide (Rb_2O) 0.078%, Strontium Oxide (SrO) 0.055%, and Zirconium Dioxide (ZrO_2) 0.037%. Therefore, ash from palm oil husks and shells has the potential to be used as an adsorbent because it contains an active compound, namely silicon dioxide (SiO_2) [1].

Chitosan has hydroxyl and amino groups along its polymer chain, making it highly effective at adsorbing heavy metal cation and cation from organic substances, such as fats and proteins [2]. Chitosan has the potential to be used as an adsorbent due to the presence of active groups, namely $-NH_2$ and –OH groups. Through these active groups, chitosan can adsorb metal ions by binding the metal ions with the amine groups in chitosan [3].

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Modification of silica functional groups with chitosan can increase its adsorption of metal ions. Chitosan has active amine (-NH_2) and hydroxyl (-OH) groups that can increase Pb(II) adsorption. The results of functional group identification using FTIR show that the absorption at wave numbers 1637.02 cm-1 and 1060.1 cm^{-1} indicate the bending vibration of –OH from SiOH and the stretching vibration of Si-O from Si-O-Si. Absorption at wave numbers 1557.99 cm^{-1} and 1420.47 cm^{-1} indicates the bending vibration of –NH and the symmetry vibration of –CN from chitosan [4].

2. EXPERIMENTAL

2.1. Chemicals, Equipment and Instrumentation

The materials used in this study were palm oil boiler ash (ABKS), chitosan powder, hydrochloric acid (HCl), sodium hydroxide (NaOH), distilled water, acetic acid (CH_3COOH), Sodium Tripolyphosphate (NaTPP), $Pb(NO_3)_2$, and HNO_3. The tools used in this study were a furnace, a 200 mesh sieve, a magnetic stirrer, a watch glass, an analytical balance, porcelain, a beaker, a stirring rod, a measuring flask, a desiccator, a volumetric pipette, a measuring cylinder, a pH meter, Fourier Transformed Infrared (FTIR) and Atomic Absorption Spectroscopy (AAS).

2.2. Research Procedure

2.2.1 Preparation of Palm Oil Boiler Ash (ABKS) particles

Preparation of palm oil boiler ash (ABKS) samples from palm oil processing plant waste. Boiler ash from palm oil mill waste was calcined in a furnace at 500°C for 5 hours. It was then sieved using a 200-mesh sieve. Ten grams of palm oil boiler ash were soaked in 12 M HCl for 24 hours. The ash was then filtered, neutralized with distilled water, and dried at 120°C for 2 hours. The dried product was weighed, mixed with 4 M NaOH, and heated with stirring until thickened. The resulting solution was heated in a furnace at 500°C for 30 minutes. 100 mL of distilled water was then added and allowed to stand for 24 hours (Wimarsela et al., 2021). The precipitate was then filtered, and the resulting filtrate was added with 3 M HCl drops to a pH of 7, after which it was allowed to stand for 24 hours. The precipitate formed was filtered, then neutralized with distilled water and dried at a temperature of 120°C for 2 hours.

2.2.2 Characterization of ABKS Silica

Functional group analysis of boiler ash samples was performed using an FTIR instrument.

2.2.3 *Preparation of Chitosan and Silica Composite from ABKS*

Weigh 1 gram of chitosan and dissolve it in 100 mL of 1% acetic acid. The solution is then stirred at room temperature for 24 hours at 400 rpm. Then, 20 mL of 2% NaTPP solution is added, after which the solution is stored for 24 hours at room temperature to remove air bubbles. A sodium silicate solution is prepared by adding 2 grams of silica to 100 mL of 2 M NaOH. The sodium silicate solution is then added to the chitosan solution while stirring using a magnetic stirrer. During the stirring process, HCl is slowly added to the pH 7 solution, filtered, and then oven-dried at 70°C until the temperature remains constant.

2.2.4 Adsorbent Characterization

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The resulting adsorbent was then characterized using an FTIR instrument to analyze the functional groups of the resulting adsorbent.

2.2.5 Adsorption of silica-chitosan composites on Pb

A 25 mL sample of Pb solution was placed into a beaker and then an adsorbent made from silica composite from ABKS with chitosan was added as much as 0.1 gram. Stirring was carried out using a magnetic stirrer with varying times of 45, 95, and 105 minutes. The optimum time was varied with a mass of 0.2, 0.3, and 0.4 grams, then analyzed with an AAS instrument.

3. RESULTS AND DISCUSSION

3.1. Preparation of Palm Oil Boiler Ash (ABKS) particles

Palm oil boiler ash is prepared to remove impurities, ensuring a clean, ready-to-use process for silica production. Palm oil boiler ash is calcined at 500°C for 5 hours using a kiln. Calcination oxidizes or removes any remaining carbon present in the ash, as well as any organic materials [5]. After the calcination process is complete, the ash is ground and sieved. This grinding and sieving process is carried out to obtain a more uniform particle size. This will ensure more consistent results in the process of producing silica from boiler ash, as the chemical reactions that occur are more homogeneous in particles with a uniform particle size [6]. The palm oil boiler ash is then soaked in 12 M HCl for 24 hours to activate the silica in the palm oil boiler ash. The washing or neutralization process is carried out to remove the HCl absorbed by the ash during activation. The neutralized ash is oven-dried at 120°C for 2 hours to remove any moisture content.

After achieving a constant ash weight, to produce sodium silicate (Na2SiO3) using the sol-gel method, 4 M NaOH is added to the ash and heated to 80°C while stirring with a magnetic stirrer. Once the solution thickens, the heating and stirring are stopped. The ash is then heated in a furnace at 500°C for 30 minutes. This process evaporates any remaining water in the sodium silicate and removes any volatile compounds that may still be present [7].The resulting solid was then soaked in distilled water. This soaking process optimized the dissolution of the sodium silicate formed during heating. This allowed any remaining undissolved sodium silicate to dissolve optimally during the soaking process.

The resulting sodium silicate solution, in the form of a filtrate, was then separated from the remaining impurities. The resulting sodium silicate filtrate was clear and slightly oily. Acid was then added dropwise to the sodium silicate solution, using 3 M HCl until neutral (7) was obtained, to produce silica gel. HCl was chosen over other strong acids because it can produce a gel with a larger surface area. The gel formed from the addition of acid is called a hydrogel. The resulting hydrogel is then left to mature for 24 hours to allow the gel to mature. This maturing process strengthens the gel, increasing the particle size and pore area of the gel, resulting in a homogeneous gel. After maturation, the silica gel is formed, then washed with distilled water until the pH is neutral. This is done to remove any remaining Na+ ions in the gel. Then, it is heated at 80°C–120°C until dry. This drying process removes any remaining free water in the silica pores, resulting in a stable silica powder [8]. The silica extraction yield from palm oil boiler ash was 6.012 grams, with a yield of 60.12%. This indicates that the silica content in palm oil boiler ash is high enough to be processed into functional materials.

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3.2. Characterization of ABKS Silica

Characterization of palm oil boiler ash silica using *Fourier Transform Infrared Spectroscopy* (FTIR) was conducted to identify the functional groups present in the synthesized silica. This analysis was carried out to identify the functional groups present in the synthesized silica. Therefore, this analysis is very useful for confirming the presence of functional groups in the general silica structure, such as silixane bonds (Si-O-Si) and silanol groups (Si-OH).

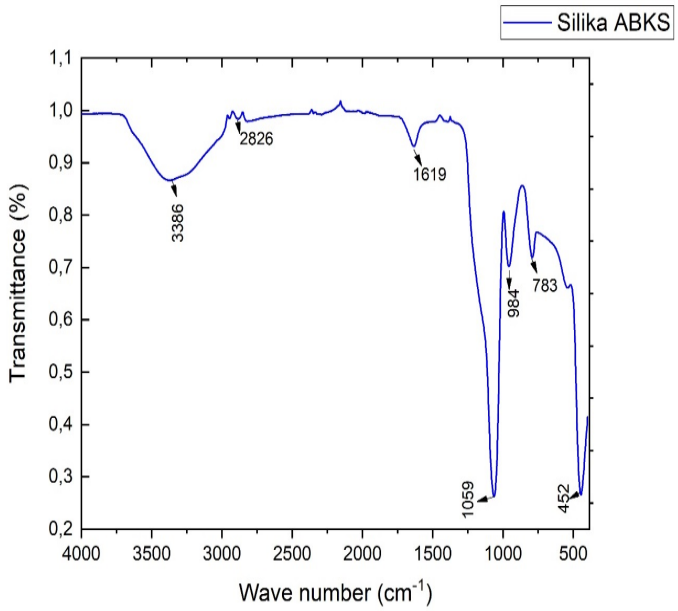

Figure 1. FTIR Spectrum of ABKS Silica

Based on the analysis results in (Figure 1) it can be seen that the FTIR results of palm oil boiler ash silica (ABKS) show absorption at wave numbers 452 cm^{-1}, 783 cm^{-1}, 984 cm^{-1}, 1059 cm^{-1}, 1619 cm^{-1}, 2826 cm^{-1}, and 3386 cm^{-1}. At wave number 3386 cm^{-1} indicates the presence of hydroxyl groups (-OH) which can originate from the assumption of the presence of adsorbed water molecules or silanol groups (Si-OH) contained in silica. The presence of this group indicates that silica is hydrophilic. Furthermore, the peak that appears at wave number 1619 cm^{-1} indicates the presence of bending vibrations or H-O-H bending bonds. The wave number 1059 cm^{-1}, which appears and is strong is a vibration that indicates asymmetric stretching of the Si-O-Si group, and at wave numbers 783 cm^{-1}, 984 cm^{-1} is a symmetric stretching vibration of the Si-O-Si group which is the main indicator of the formation of silica groups. While at wave number 452 cm^{-1} is a typical band of the Siloxane ring found in the synthesized silica [9].

Table 1. Interpretation of Silica Gel Spectra

Gugus Fungsi	Bilangan Gelombang (cm^{-1})	Interpretasi
-OH dari	3600-3000	Vibrasi Stretching

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Si-OH		–OH dari Si-OH
-OH dari Si-OH	1650-1620	Vibrasi bending –OH dari Si-OH
Si-O-Si	1200-1020	Vibrasi Stretching asimetris Si-O-Si
Si-O-Si	450-470	Pita bending Si-O-Si

The data in the table above confirms that silica from palm oil boiler ash was successfully synthesized due to the presence of –OH groups from Si-OH stretching and bending vibrations, as well as Si-O-Si groups, which are the main groups in silica.

3.3 Preparation of Chitosan and Silica Composite from ABKS

To prepare a 1% chitosan solution, acetic acid (CH_3COOH) is added while stirring with a magnetic stirrer. This will create a slightly yellow liquid chitosan solution. Then, 1% NaTPP is added. NaTPP acts as a crosslinker, forming chitosan-TPP through intermolecular or intramolecular bonds with ionic reactions [10]. To prepare a 2% sodium silicate solution, 2 grams of silica are added to 100 mL of 2 M NaOH slowly while stirring with a magnetic stirrer for 30 minutes. The sodium silicate solution is then added to the 1% chitosan solution and slowly added 2 M HCl drops until a neutral pH (7) is achieved. The resulting white, viscous chitosan-silica composite is filtered and oven-dried at 70°C until a constant weight is achieved, resulting in a pure white composite The addition of HCl to the composite forms a white, viscous gel. When HCl is added, it protonates the NH_2 group to NH_3^+, then an electrostatic bond is formed between the silicate anion and NH_3^+. A hydrogen bond will form between the silanol group and the hydroxyl group [4].

3.4 Adsorbent Characterization

Characterization of silica-chitosan composites using *Fourier Transform Infrared Spectroscopy* (FTIR).

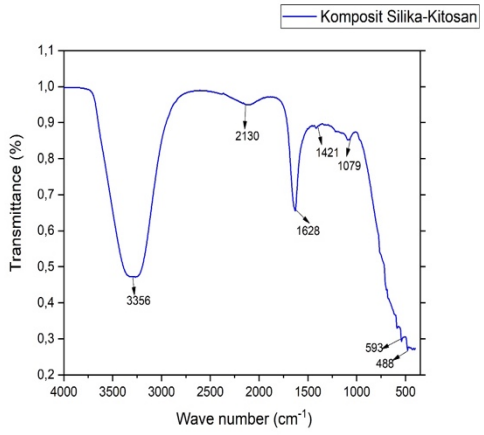

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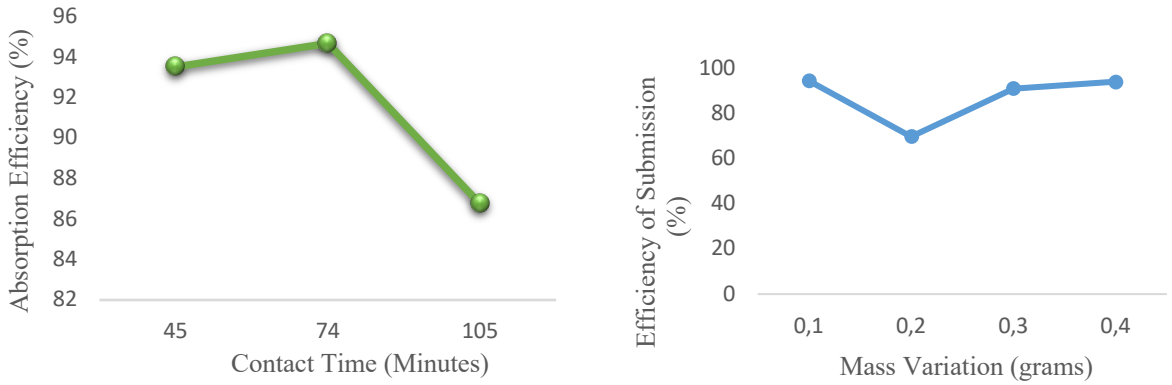
Figure 2. FTIR Spectrum of Silica-Chitosan Composite

Based on Figure 2 FTIR analysis results on silica-chitosan composite, there are several groups that appear seen from the wave numbers produced in the spectrum. In the wide absorption band at wave number 3356 cm^{-1} is the stretching vibration region of the (-OH) group from the silanol group (Si-OH) which is a functional group derived from silica as well as the (-OH) and ($-NH^2$) groups found in chitosan. The presence of absorption band broadening indicates that there is an interaction between silica and chitosan through hydrogen bonds which indicates the formation of a composite between the two materials. At the peak wave number 1628 cm-1 is the bending vibration of the amide group (-NH) of chitosan as well as bending vibrations originating from the H-O-H bond in the water that is still bound. At wave number 1421 cm^{-1} which is related to the symmetric bending vibration of the chitosan oligosaccharide chain, namely the $–CH_2$ and $–CH_3$ groups which can be influenced by the presence of the silica matrix. Furthermore, the absorption at wave number 1079 cm^{-1} is the main functional group that indicates silica material in the form of symmetric stretching vibrations of Si-O-Si, the presence of this absorption band is evidence that the presence of silica appears in the silica-chitosan composite. Furthermore, at wave numbers 593 cm^{-1} and 488 cm^{-1} are areas that are bending vibrations of Si-O-Si and Si-O which are also characteristics of the silica structure.

2.5 Adsorption of silica-chitosan composites on Pb

The absorption efficiency of Pb using a silica-chitosan composite adsorbent was 93.54% at 45 minutes, then increased to 94.79% at 75 minutes, then decreased drastically at 105 minutes to 86.78%. The data obtained showed that the absorption efficiency for Pb was highest at 75 minutes. Before and after reaching 75 minutes, there was a decrease in absorption efficiency, indicating that the optimum time was 75 minutes.

Based on mass variations, there was an increase and decrease in the adsorbent's ability to absorb lead ions in Pb solutions. Increasing the adsorbent mass can increase the adsorption capacity, resulting in an increase in the adsorbate adsorbed. Increasing the adsorbent mass also increases the active surface area interacting with the adsorbate. With increasing mass, the pores of the adsorbent become larger, trapping more adsorbate. However, after reaching the optimum mass, there will be a decrease in adsorbate absorption. This is because the active side of the adsorbent does not fully bind to the adsorbate, when the mass is optimum, the absorption is high [11].



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Figur 3. Pb Adsorption with Mass Variation and Contact Time Variation

4. CONCLUSION

Silica characterization confirmed the hydroxyl group (-OH) at wave number 3386 cm-1. 1059 cm-1 Si-O-Si group, 783 cm-1, 984 cm-1 symmetrical stretching of the Si-O-Si group, wave 452 cm-1 Siloxane group, in the silica chitosan composite 3356 cm-1 (-OH) group of the silanol group (Si-OH) is a functional group derived from silica as well as the (-OH) and (-NH2) groups, 1628 cm-1 amide group (-NH), 1079 cm-1 Si-O-Si group, wave 593 cm-1 and 488 cm-1 bending of Si-O-Si and Si-O. The optimal result of Pb adsorption of silica chitosan composite is 94.70% with an absorption capacity of 1.2467 mg/g.

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