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NaOH Concentration-Dependent Silanol Formation and Pb2+ Adsorption by Waste Glass-Derived Silica Gel

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Abstract

Clear glass waste, a prevalent inorganic waste in Indonesia, presents an opportunity for eco-friendly adsorbent development. This study explored the impact of varying NaOH concentrations (1M-4M) on the Pb2+ ion adsorption capacity of silica gel synthesized from clear glass waste via the sol-gel method.While conclusive determination of the optimal NaOH concentration for Pb2+ adsorption remained outside the study's scope, valuable insights were obtained. FTIR analysis confirmed the presence of silanol (SiOH) and siloxane (Si-O-Si) groups within the silica gel structure, with the SiOH group specifically identified at a wavenumber of 957 cm^{-1}. Increasing NaOH concentration from 0.5 M to 4 M significantly enhanced the formation of silanol groups on the silica gel surface, as evidenced by the increasing area of the corresponding peak in the FTIR spectrum (3.2 to 71.0 arbitrary units). This positive influence translated to an improved Pb2+ adsorption capacity, with the highest performance of 2.3 mg/g achieved at the highest NaOH concentration. However, the relationship wasn't perfectly linear, suggesting the influence of additional factors like silanol distribution and the presence of competing ions. This work demonstrates the potential of waste glass-derived silica gel for Pb2+ remediation while emphasizing the need for further research to optimize synthesis parameters and elucidate the full adsorption mechanisms.

Keywords : *Glass Waste, Silica Gel, NaOH Concentration, Pb^{2+} Adsorption, Silanol.*

INTRODUCTION

Clear glass waste, a prevalent and problematic component of the inorganic waste stream in Indonesia, offers a promising prospect for eco-friendly adsorbent development. This study delves into the impact of varying NaOH concentrations (1M-4M) on the Pb2+ ion adsorption capacity of silica gel synthesized from clear glass waste using the sol-gel method.

The widespread environmental pollution caused by heavy metals like lead necessitates effective and sustainable strategies for their removal(Vidu et al., 2020). Traditional adsorbent materials often come with high production costs and environmental drawbacks. In this context, repurposing clear glass waste as an adsorbent presents a compelling solution, offering environmental benefits by mitigating waste and economic advantages by utilizing a readily available material(Permatasari et al., 2016)(Azmiyawati et al., 2019).

While prior research has explored the synthesis of silica gel from clear glass waste(Khair et al., 2023), the precise influence of NaOH concentration on its Pb2+ ion adsorption capacity remains underexplored. This study aims to bridge this gap by systematically investigating the relationship between NaOH concentration, silica gel properties, and Pb^{2+} ion adsorption.

Through FTIR analysis, we aimed to elucidate the presence and relative abundance of functional groups on the silica gel surface, particularly silanol (SiOH) groups known for their role in metal ion adsorption. Additionally, the relative peak area of the SiOH group at a specific wavenumber was calculated to assess the impact of NaOH concentration on its content.

By unraveling the underlying mechanisms governing Pb2+ ion adsorption in relation to NaOH concentration and silica gel structure(Felss et al., 2020), this study aims to contribute to the development of a sustainable and efficient material for heavy metal removal from contaminated environments(Yang et al., 2019). Furthermore, the findings may provide valuable insights for tailoring the adsorption capacity of this eco-friendly material for specific applications.

METODE

Silica Gel preparation

Clear glass waste underwent rigorous cleaning with running water, soap, and an ultrasonic bath before drying, crushing, and grinding into a fine (100 mesh) powder. Five Erlenmeyer flasks, each containing a precisely measured 10 grams of powder, were used for solutions with varying NaOH concentrations (0.5M, 1M, 2M, 3M, and 4M). Magnetic stirring for 3 hours at 80°C ensured thorough interaction and promoted the desired reaction.

Following 3 hours of stirring, the mixtures were carefully transferred to a 400-watt microwave oven for a 5-minute treatment to expedite the reaction. Afterward, they were allowed to cool to room temperature. Each cooled solution was meticulously filtered to remove unreacted particles or impurities, resulting in separate sodium silicate solutions for further processing.

These sodium silicate solutions were individually transferred to a 500 mL flask and neutralized with dropwise addition of concentrated H2SO4 until reaching a neutral pH. The mixtures were then incubated at room temperature for 48 hours, facilitating the formation of the desired hydrogel product.

The hydrogels were subsequently dried in an oven at 80°C for 18 hours and then activated by heating them in an oven at 500°C for 3 hours. This final activation step yielded the silica gel products ready for further characterization and analysis.

Peak area measurement using Fourier-Transform Infrared Spectroscopy

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The peak area of silanol groups in synthesized silica gels was quantified using a Fourier-transform infrared (FTIR) spectrometer. A pellet was formed from 2 mg of each sample and scanned over a 4000-400 cm^{-1} wavenumber range. The spectra were normalized using OriginPro 9.1 software. The peak area of the Si-OH stretching vibration at 956 cm^{-1} was determined using the OriginPro Peak Analyzer tool. Every sample of synthetic silica gel underwent this process again.

To assess Pb^{2+} adsorption, 0.1 g of optimal silica gel was shaken with 25 mL of 100 ppm $Pb(NO_3)_2$ (pH 4) for 60 minutes at 150 rpm. The Pb^{2+} concentration remaining in the filtered solution was then measured to calculate the silica gel's adsorption capacity.

RESULT AND DISCUSSION

The FTIR results of the synthesized silica gel show a band around 1090 cm-1 is observed in silica gel, which is caused by the Si-O-Si stretching vibration. Additionally, there is a band around 800 cm-1 in both materials, which indicates the Si-O-Si bending vibration.

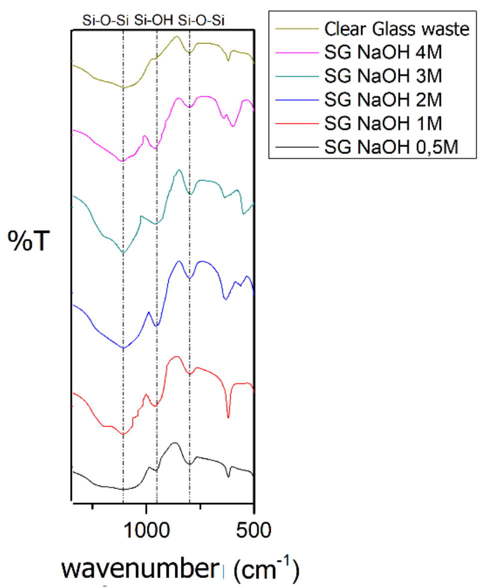

Figure 1. Effect of NaOH concentration on the presence of silanol peak of the silica gel.

However, the key spectrum is the presence of a band in silica gel around 950 cm-1, which is caused by the stretching vibration of the OH group of the silanol (SiOH) group (Cakmak et al., 2021). The peak at 956 cm^{-1} is specifically associated with the Si-OH stretching vibration of the silanol group. Analyzing the 956 cm^{-1} peak avoids this ambiguity and directly measures the presence of silanol.

Silanol groups, characterized by the Si-OH functional group, play an important role in the adsorption properties of silica gel. These groups are polar and can form hydrogen bonds with other polar molecules, such as water and ions.

The increased number of silanol groups on the surface of the silica gel is expected to improve its adsorption capacity for water and ions. This is because the silanol groups can form hydrogen bonds with these molecules, which helps to hold them on the surface of the silica gel.

The results of the Fourier transform infrared (FTIR) analysis of silica gel prepared with different concentrations of NaOH showed that the area of the silanol peak at 956 cm^{-1} increased with increasing NaOH concentration. This is consistent with the assumption that the silanol peak area is proportional to the number of silanol groups in the silica gel(Salsabila et al., 2024). The change in the area of the silanol peak and Pb^{2+} ion adsorption in the Silica gels is displayed in Figure 2.

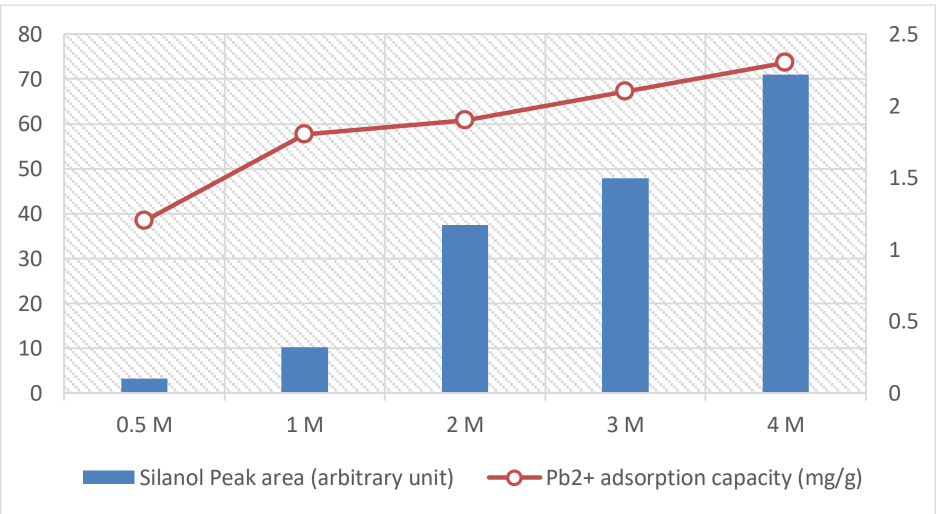

Figure 2. Effect of NaOH concentration on silanol peak area and Pb^{2+}adsorption capacity

Figure 2 clearly demonstrates the correlation between NaOH concentration used during silica gel synthesis, the area of the silanol peak in the FTIR spectrum, and the Pb^{2+} adsorption capacity of the resulting material.

- Increasing NaOH concentration leads to a significant increase in the silanol peak area. This aligns with our earlier discussion about how higher NaOH concentrations promote the formation of more silanol groups on the silica gel surface.
- The Pb2+ adsorption capacity exhibits a less clear and subtler trend, despite the silanol peak area increasing steadily. While it generally increases from 1.2 mg/g at 0.5 M NaOH to 2.3 mg/g at 4 M NaOH, the gains are smaller and not perfectly proportional to the silanol peak area changes. This suggests that other

factors besides the number of silanol groups might also influence Pb^{2+} adsorption.

- The maximum Pb2+ adsorption capacity is achieved at the highest NaOH concentration (4 M), supporting the overall connection between silanol content and adsorption efficiency. However, a smaller increase between 3 M and 4 M NaOH compared to the previous increments indicates a possible saturation effect, where further rises in NaOH concentration no longer translate to significantly improved adsorption.

So, although silanol group formation is undoubtedly enhanced by increasing NaOH concentration, there may be variations in their accessibility to Pb^{2+} ions (Ncube et al., 2017). Even though a group grows in number overall, its efficacy in adsorption may be limited by its location in less accessible areas. This emphasizes how crucial it is to investigate silanol distribution patterns in order to gain a better knowledge of their actual role in the removal of Pb^{2+}. Other ions in the solution, such as impurities or those from the original waste glass, can prevent Pb^{2+} from adhering to the gel by vying for active sites. The observed discrepancy between the silanol peak area's significant increase and the adsorption capacity's smaller increase could be explained by this competition(Shafiq et al., 2021). It's critical to keep in mind that other entities participate in Pb^{2+} adsorption besides silanol groups(Katubi et al., 2021). The interaction between the metal ions and the silica gel is also influenced by other surface characteristics such as pore size and surface charge. Further investigation should take into account their possible contribution to offer a more comprehensive understanding of the adsorption mechanism, even though it is not directly measured here.

CONCLUSION

This study successfully employed a microwave-assisted method to synthesize silica gel from waste glass and explore its potential for Pb^{2+} removal. Increasing the NaOH concentration during synthesis effectively boosted the formation of silanol groups on the silica gel surface, as evidenced by FTIR analysis. These abundant silanol groups demonstrably enhanced the Pb^{2+} adsorption capacity, with the highest performance achieved at the highest NaOH concentration used. However, the relationship wasn't perfectly linear, suggesting the influence of additional factors like silanol distribution and competing ions. Overall, this work highlights the promise of waste glass-derived silica gel for Pb^{2+} remediation while emphasizing the need for further research to optimize synthesis parameters and elucidate the full adsorption mechanisms. By delving deeper into these aspects, we can unlock the full potential of this sustainable material for environmental cleanup applications.

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