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Synthesis of Silica from Chitosan-Modified Sugarcane Bagasse and GPTMS Binding Compound as an Adsorbent for Remazol Yellow Dye Waste

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Abstract

Remazol Yellow, a synthetic dye waste, is carcinogenic and mutagenic, necessitating waste treatment. One of the methods used is adsorption with silica adsorbents and sugarcane bagasse. Silica has unusual features, including mechanical stability at high temperatures, strong ion-exchange capacity, and a large surface area with broad pores. The inclusion of chitosan increases the number of active sites on silica as an adsorbent. The inclusion of the binding agent glycidoxypropyltrimethoxysilane (GPTMS) can improve the link between silica and chitosan as the dye adsorbent concentration increases. The FTIR characterization results indicate absorption bands at 451 cm^{-1}, 796 cm^{-1}, and 958 cm^{-1}, corresponding to the spectra of silica and chitosan chemical groups. The XRD analysis revealed crystalline silica in the form of the cristobalite phase. SEM analysis revealed that the binding chemical increased the surface porosity of the silica-chitosan adsorbent. The optimal adsorption conditions for utilizing chitosan-modified silica adsorbent are a pH of 2 and a contact time of 2 hours. An adsorbent mass of 0.5 g, with an adsorption capacity value of 2.17 mmol/g, influences the growth in adsorption capacity, as does an adsorbate concentration of 80 ppm, with an adsorption capacity value of 2.8 mmol/g.

Keywords: Adsorption, Citosan, GPTMS, Remazol Yellow, Silica

INTRODUCTION

Waste is still a big environmental issue, as is textile manufacturing liquid waste from the dyeing process, which comprises a variety of complex organic chemicals that are difficult to degrade naturally. Discharging untreated textile waste causes significant ecological damage, affecting water quality and destroying aquatic life (Akter et al., 2023; Chang Chien et al., 2024). The batik fabric industry needs effective processing solutions. Toxic synthetic dyes can cause health problems in humans and animals (Sharma P. et al., 2022). The batik textile business, in particular, relies heavily on synthetic dyes such as Remazol Yellow. Remazol Yellow is a reactive dye that contains chromophore groups. Remazol Yellow is a reactive dye that has a chromophore group (Magfiroh L. et al., 2016). Microorganisms in the environment struggle to degrade this dye naturally. Remazol yellow is an azo chemical that is highly soluble in water but does not decompose in aerobic conditions. It also has high chemical stability, making it difficult to degrade and potentially harming the environment and causing disease (Lestari et al., 2023). Moreover, remazol yellow is stable under varying pH and temperature conditions and is resistant to microorganisms, necessitating treatment prior to environmental discharge (Magfiroh L. et al., 2016). If left untreated, it can contaminate ecosystems and accumulate at the bottom of water bodies. Azo dyes are carcinogenic and mutagenic, with harmful environmental implications and the potential to cause illness (Lestari et al., 2023). Because of its environmental impact, waste must be treated before release. The adsorption method is a development technique. Adsorption is preferred because it is simple and effective, and it can be used with various adsorbents, including environmentally friendly and cost-effective bioadsorbents (Taba et al., 2021; Kating C. et al., 2022).

This approach was chosen because it is inexpensive, simple to use, highly effective, and employs easily available adsorbents (Harimu et al., 2019; Badri et al., 2021). Silica is one of the

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adsorbents used. Silica can be obtained by using waste materials. One such material is sugarcane bagasse. At 68.5%, sugarcane bagasse has a high silica content. Silica is a type of support material with a large surface area, good mechanical properties, and high porosity (Ebisike et al., 2020; Nuryono et al., 2025). Because it contains silanol (Si-OH) and silicon (Si-O-Si) groups, which are active groups with a high surface area and numerous pores, silica is employed as an adsorbent (Purnawan et al., 2018; Mujiyanti et al., 2020; Berliana S., 2024). However, silica is less stable and brittle, thus requiring modification (Balavigneswaran et al., 2020).

Chitosan is used during the modification process to enhance the activity of the silica surface-active sites. Chitosan is a biodegradable and non-toxic biopolymer. Because of its special properties, chitosan can be used as a bio-adsorbent. Methylene blue dye and other dye ions can be effectively adsorbed by chitosan's hydroxyl and amine groups (Arunachalam et al., 2021; Mousavi et al., 2020; Rahayu et al., 2020). The active -NH_2 group on chitosan charges its surface positively in aqueous and acidic conditions, allowing it to establish electrostatic (ionic) bonds with anionic dyes. Remazol Yellow is an anionic dye, along with Remazol Red 133 (RR) and Rifaction Yellow HED (RY) (Indah et al., 2024; Attalah et al., 2016). A binding chemical is also required to improve the active side of the adsorbent modification. The presence of binding compounds will facilitate the interaction between chitosan and silica. This is because the interaction between chitosan and silica can be weak, especially under acidic conditions, leading to aggregation and reduced mechanical properties (Liang et al., 2020).

The binding agent used is glycidoxypropyltrimethoxysilane (GPTMS). GPTMS is a coupling agent for silane groups consisting of epoxy and methoxysilane. Epoxy groups on GPTMS molecules will bind with amino or carboxyl groups on the polymer chain, and the silanol group will undergo hydration with the trimethoxy group through an acid-catalyzed reaction. In this reaction, condensation will also occur between two Si-OH molecules to form a Si-O-Si bond, leading to cross-linking via Si-O-Si bonds (Balavigneswaran et al., 2020). GPTMS helps improve the stability and performance of the adsorbent by forming covalent bonds between silica and chitosan (Li C. et al., 2020). The inclusion of the coupling agent enhances interfacial compatibility between silica and chitosan, thereby stabilizing the modified adsorbent surface and reducing particle aggregation, thereby improving surface area (Mousavi et al., 2020). In this research, silica will be modified with chitosan, and a GPTMS binding compound will be added, thereby improving the stability and performance of the adsorbent in adsorbing Remazol Yellow dye waste.

METHODOLOGY

Materials and Instrumentals

The materials used are bagasse, sodium hydroxide (NaOH), hydrochloric acid (HCl), glycidoxy-propyl-trimethoxysilane (GPTMS), chitosan, and acetic acid (CH_3COOH). The instrumental used are UV-Vis, FTIR, XRD, AAS, and SEM.

Methods

Production of silica from sugarcane bagasse

Sugarcane bagasse was dried in direct sunshine. It was dried and then burned for 6.5 hours at 750°C in a furnace. The final product was blended and sieved through a 150-mesh sieve. The next phase is silica extraction from sugarcane bagasse. The initial step in synthesis is to cleanse the sugarcane bagasse ash. Five grams of sugarcane bagasse ash were placed in a glass beaker and rinsed with 2 mL HCl while stirring for 1 minute. After filtering, the outcome was neutralized with distilled water and allowed to stand. The resulting ash residue was returned to the beaker, 100 ml of 1 M NaOH was added, and the mixture was agitated with a stirrer for an hour at 80°C. After that, the residue was separated with filter paper. The resultant residue was returned to an Erlenmeyer flask and titrated with 6 M HCl until the pH reached 7 and a gel formed. The developed gel was allowed to stand overnight. The resultant gel was filtered using filter paper and rinsed with distilled water. After that, the residue was heated to 80°C until dry. The results were then examined with FTIR.

Synthesis of chitosan-modified silica with the binding compound GPTMS (glycidoxypropyltrimethoxysilane)

First, a chitosan solution was prepared. 1.5 g of chitosan powder was dissolved in 100 ml of 2% acetic acid, and the mixture was stirred with a magnetic stirrer until completely dissolved. The silicate solution was prepared by dissolving the precipitate from the first treatment in 100 mL of distilled water, allowing it to settle overnight, and then filtering.

The chitosan and sodium silicate solutions were mixed in a beaker in a 1:2 ratio, followed by the addition of 0.45 ml of 98% GPTMS. The mixture was stirred for 2 hours. Then, 6 M HCl was added dropwise until a gel formed, and the mixture was left for 24 hours. After the gel formed, distilled water was added to dilute it to neutral pH. The products were filtered and oven-dried at 70°C for 6 hours. The obtained solid was crushed and sieved through a 150-mesh sieve before being characterized by XRD, FTIR, SAA, and SEM.

Adsorption study of composite adsorbents on Remazol Yellow (RY) dye

Determining the effect of pH

20 ml of Remazol yellow solution with an initial concentration adjusted to pH by varying the pH to 2, 4, 6, 8, 10, and 12. Then, 0.2 g of the adsorbent composite was added to each solution. The solution was stirred for sixty minutes. The resulting mixture was separated into two components: the residue and the filtrate. The filtrate was examined with a UV-VIS spectrophotometer.

Determination of adsorbent contact time

20 ml of Remazol yellow solution with an initial concentration was added to 0.2 grams of the adsorbent composite. The solution was stirred for 1, 2, 3, 4, and 5 hours. The mixture was separated, and the filtrate was examined using a UV-Vis spectrophotometer.

Determining the effect of adsorbent concentration

20 ml of a 100 ppm Remazol yellow solution was added to each composite at 0.1, 0.2, 0.3, 0.4, 0.5, and 0.6 grams. The solution was stirred for the optimal time and at the optimal pH. The resulting mixture was separated, and the filtrate was analyzed by UV-Vis spectrophotometry.

Determining the Influence of Initial Concentration

20 ml of Remazol yellow solution with varying concentrations of 10, 20, 40, 60, 80, 100, and 200 ppm. Then, 0.2 g of the adsorbent composite was added to the solution. The solution was stirred for the optimal time at the optimal pH. The resulting mixture was separated, and the filtrate was analyzed by UV-Vis spectrophotometry.

Data Analysis

Data analysis utilizing calibration curve data and the implementation of the derived linear regression equation.

RESULTS AND DISCUSSION

FTIR analysis of silica from sugarcane bagasse

The FTIR analysis results of silica synthesis from sugarcane bagasse indicate that at a wavelength of 451 cm^{-1}, there is a bending vibration of the Si-O-Si bond; at 796 cm^{-1}, an asymmetric stretching vibration of the Si-O group within the Si-O-Si bond; at 958 cm^{-1}, a stretching vibration of the OH group in the Si-OH bond; and at 1066 cm^{-1} and 1192 cm^{-1}, stretching vibrations of the Si-O-Si bond (Alshandoudi et al., 2025). The absorption band at 1637 cm^{-1} indicates bending vibrations of the O-H group in Si-OH and the presence of condensed water from the silica group (Alshandoudi et al., 2025).

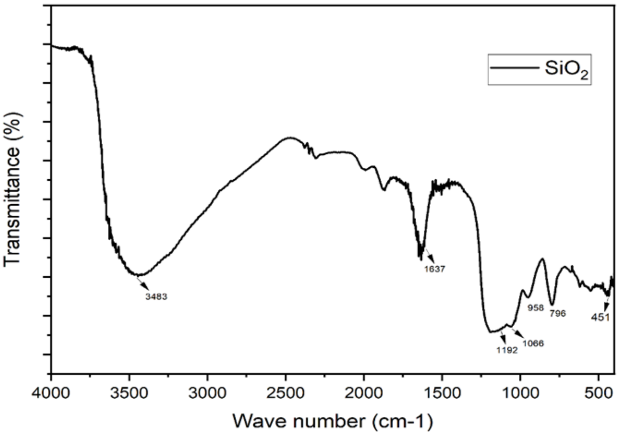

Figure 1. FTIR Spectra Results of Silica from Sugarcane Bagasse

FTIR Analysis of SiO$_2$-Chitosan Composite with GPTMS (Glycidoxy propyl trimethoxysilane) Binding Agent

Following the formation of silica from sugarcane bagasse using the sol-gel process, it will undergo additional modification with chitosan and the incorporation of the binding agent GPTMS. The purpose of adding these compounds is to increase the surface area of the silica compared to not adding them. The results can be seen in the characterization results. Silica has active groups with low acidity and weak donor atoms, which weaken its surface. A way to overcome this problem is by modifying its surface by changing the type of active sites to increase its utilization area (Putra et al., 2022).

According to the FTIR spectra results, the addition of chitosan and the binding compound GPTMS (glycidoxypropyltrimethoxysilane) results in spectra at a wavelength of 451 cm^{-1}, which is the Si-O-Si bond's bending vibration; 796 cm^{-1}, which is the Si-O group's asymmetric stretching vibration; 958 cm^{-1}, which is the OH group's stretching vibration in the Si-OH bond; and 1066 cm^{-1} and 1192 cm^{-1}, which are the Si-O-Si bond's stretching vibrations. Silica is present in the composite material, as evidenced by the low transmittance of the absorption band peak at 451 cm^{-1} and 1066 cm^{-1} (Alshandoudi et al., 2025). The disappearance of the absorption band at 1637 cm^{-1} from the Si-OH bond, which undergoes condensation to form the Si-O-Si bond, and the emergence of a broadened Si-O-Si absorption band at 1114 indicate that the silica groups undergo condensation due to the presence of GPTMS molecules (Balavigneswaran et al., 2020).

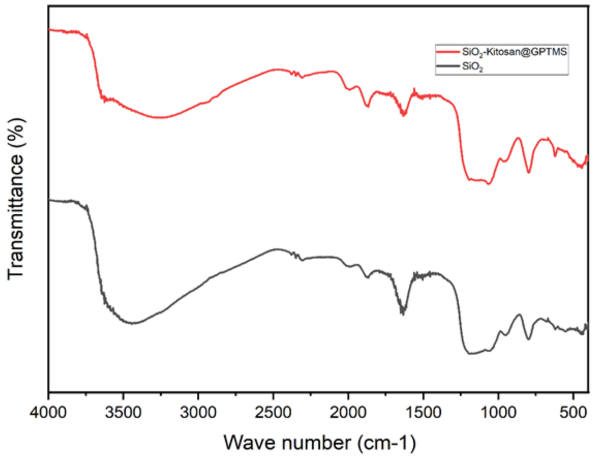

Figure 2. Results of FTIR analysis of SiO2-Chitosan GPTMS composite

The addition of GPTMS and chitosan compounds to the silica surface layer is shown by the appearance of additional peaks in the FTIR spectra. The bending vibration of C-H in the CH_3 group is shown by the spectral peak at the absorption band of 1489 cm^{-1}. The presence of this absorption band suggests that the chitosan compound was successfully cross-linked to the silica surface using GPTMS. The stretching vibrations of the N-H and O-H groups are responsible for the widening at the peak of 3400 cm^{-1}. In contrast, the stretching vibrations of the C-H groups in the basic chitosan structure are responsible for the absorption bands at 2850 cm^{-1} and 2920 cm^{-1}. The absorption band at 1585 cm^{-1} resonates with the amine group in chitosan, which undergoes stretching vibrations in the C=O group within the acetyl group in chitosan (Nuryono et al., 2025).

XRD Analysis of SiO_2-Chitosan Composite with GPTMS (Glycidoxy propyl tri-methoxysilane) Binding Agent

The SiO_2-Chitosan composite with GPTMS (glycidoxy-propyl-trimethoxysilane) binding component reveals well-decomposed XRD characterisation, with peaks at 2θ = 20.87°, 21.95°, 28.46°, 31.42°, 36.14°, 46.98°, 48.61°, and 57.12°. The XRD analysis results show a significant diffraction peak at 2θ = 21.95, indicating an amorphous silica material within the 2θ range of 20°-27° (JCPDS No. 46-1045) (Delpina et al., 2024). This peak, which correlates to the cristobalite JCPDS diffraction pattern 44-1394, indicates that silica crystallized in the cristobalite phase above 700°C (Samik et al., 2022). These results differ from those of Vareda et al (2024) XRD study, which showed the highest peak at 2θ = 18-30°, indicating a semi-crystalline structure of the chitosan-silica bond.

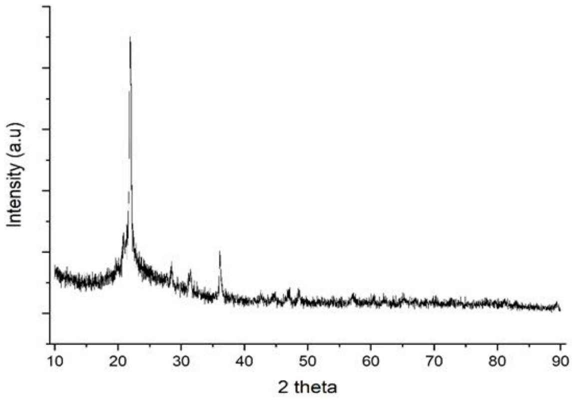

Figure 3. Result of XRD analysis of SiO2-Chitosan@GPTMS composite

Analysis of SiO_2-Chitosan Composite SEM with GPTMS (Glycidoxy propyl tri-methoxysilane) as a Binding Agent

The results of SEM characterization on the SiO_2-Chitosan composite with the GPTMS binding agent are shown in Figure 4. Figure 4 (A) represents the surface morphology of the adsorbent, which is composed of homogeneous particles. Figure 4 (B) shows the adsorbent surface, which seems rough, porous, and unsymmetrical. This surface shows that agglomerates of different sizes have formed (Ebisike et al., 2020). The addition of a binding chemical increases the adsorbent surface porosity (Purnawan et al., 2018).

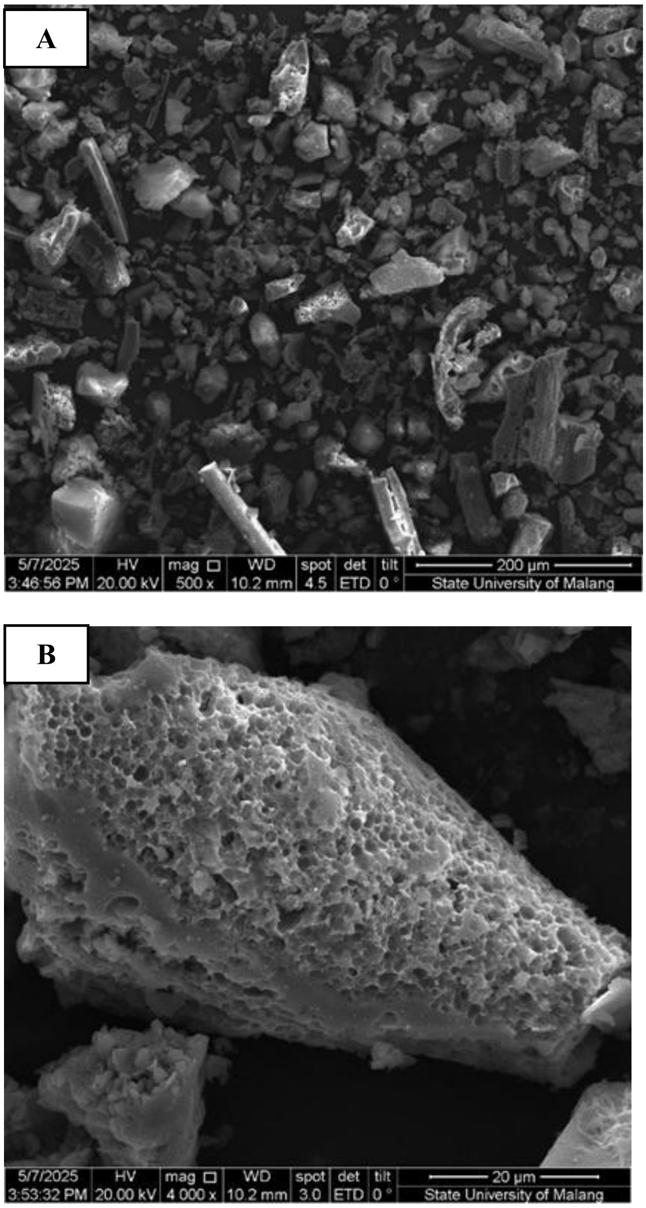

Figure 4. Result of SEM analysis (A) 500x magnification and (B) 4000x magnification

Analysis of Adsorption Study of Composite Adsorbent on Remazol Yellow (RY) Dye

The Effect of pH on the Adsorption of Remazol Yellow

One factor that can impact the adsorption process is pH. The analysis showed adsorption capacity at pH 2, as shown in Figure 5. Remazol Yellow will be protonated at basic pH, and the high concentration of -OH will fully convert the $D\text{-}SO_2CH_2CH_2OSO_3^-Na^+$ group into vinyl sulfone ($D\text{-}SO_2\text{-}CH{=}CH_2$). Vinyl sulfone-based RY will easily interact with the adsorbent's active sites (Damajanti et al., 2021). An increase in pH affects adsorption capability. This is due to an increase in OH radicals in the solution, which reduces the electrostatic attraction between the adsorbent and the Remazol Yellow dye (Lestari et al., 2023).

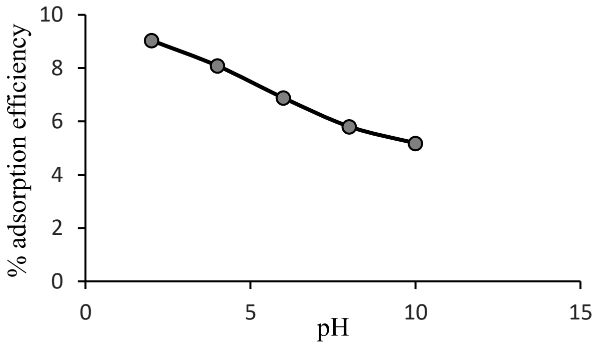

Figure 5. A graph of the effect pH on adsorption capacity

Effect of Contact Time on Dye Color

The influence of contact time is needed to determine how long the adsorbent takes to adsorb the Remazol Yellow dye to its maximum and reach equilibrium. Results are shown in Figure 6. Contact times of 1, 2, 3, 4, and 5 hours were tested. According to the study's results, dye adsorption increased after an initial hour of contact. This is because the more time spent in contact, the more the adsorbent and adsorbate interact, leading to more adsorbed molecules. At 2 hours, adsorption continued to increase; in this condition, the adsorbent's active sites had not yet reached saturation, so it could still adsorb the dye (Annisa et al., 2023). However, at three hours, there was a decrease, and the graph displayed a flat slope at four and five hours. This occurs because the adsorbent's surface is coated in adsorbate and its active sites are already saturated. Furthermore, when the adsorbent reaches equilibrium, the Remazol Yellow dye's bonds are released back to the adsorbent surface due to electrostatic interactions (Lestari et al., 2023).

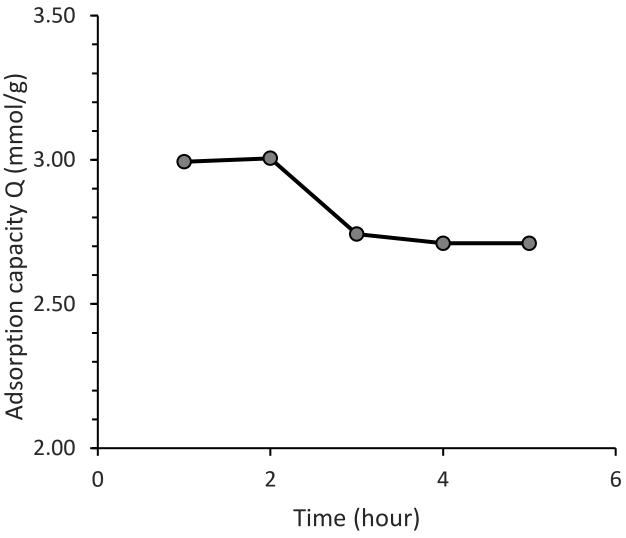

Figure 6. A graph of the effect of Contact time on adsorption capacity

Effect of Adsorbent Mass

The mass of adsorbent affects the adsorption capacity. The research results are shown in Figure 7. The graph analysis shows that the greatest adsorption capacity occurs at 0.5 g of adsorbent, corresponding to 2.17 mmol/g. This suggests that the higher the adsorbent mass, the more active sites can be employed (Paramitha et al., 2019). However, there was a decrease as the amount of the adsorbent increased. The decrease in adsorption capacity is due to the adsorbent's active sites not adsorbing enough adsorbate. The decrease in adsorption capacity is also due to desorption on the adsorbent surface, which releases the adsorbate and converts it into an impurity.

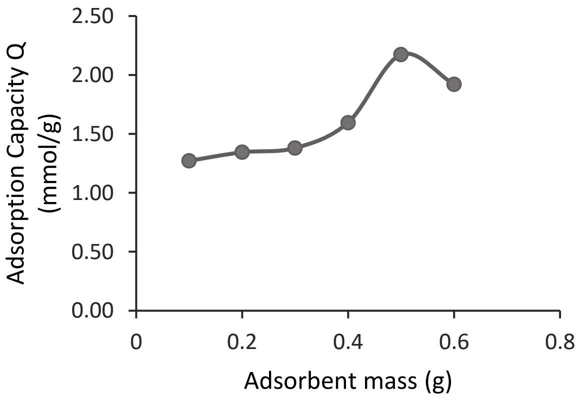

Figure 7. A Graph of the effect of adsorbent mass on adsorption capacity

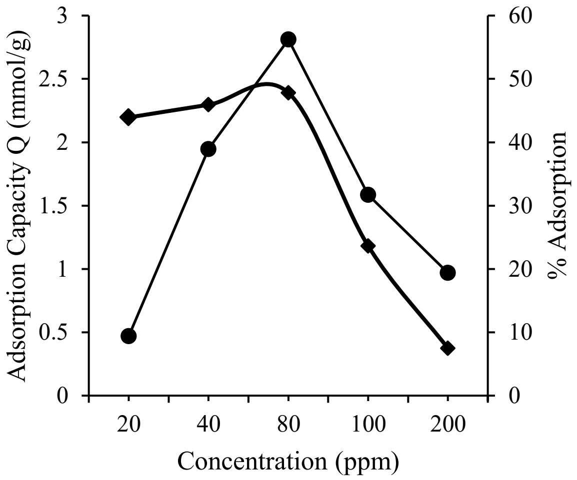

Figure 8. A Graph of the the influence of initial adsorbate concentration on adsorption

Effect of Initial Adsorbate Concentration

Effect of adsorbate concentration on the adsorbent's adsorption capacity and absorption efficiency. The analysis results show that at a concentration of 80 ppm, the highest adsorption capacity is 2.8 mmol/g (Figure 8). The adsorbate concentration impacts an adsorbent's adsorption capability. An increasing concentration of adsorbate in solution will push it towards the active side of the adsorbent (Lestari et al., 2023). owever, the adsorption capability decreases as the adsorbate concentration increases. This is because the adsorbent's active sites have already been saturated. This circumstance also affects the optimal adsorption percentage (Figure 8) at 80 ppm, which is 47.8%. The higher the adsorbate concentration, the lower the adsorption percentage. It is because the adsorbent's active site is supersaturated, as the surface is already covered with adsorbate.

CONCLUSION

The combination of silica adsorbent with chitosan, with the addition of the GPTMS binding agent, as analyzed by FTIR, XRD, and SEM, shows that the presence of the GPTMS binding compound increases the surface area of the material adsorbent, resulting in homogeneous particle shapes, a more porous surface, and minor aggregation on the adsorbent surface. The results of the adsorption test analysis of the adsorbent for Remazol Yellow dye show that adsorption is influenced by pH 2 and a contact time of 2 hours. The adsorption capacity was determined as a function of adsorbent mass, with an optimal mass of 0.5 g and a capacity of 2.17 mmol/g. The effect of adsorbate concentration showed an optimal adsorption capacity of 2.8 mmol/g at 80 ppm.

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