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Synthesis and Characterization of Bentonite/Graphene/Chitosan Nanocomposites

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Abstract: Bentonite has been widely utilized as a natural adsorbent; however, it exhibits relatively low effectiveness and selectivity, necessitating its combination with other supporting materials. Graphene and chitosan possess strong capabilities for binding metal ions in water due to their high surface area. Therefore, integrating bentonite with graphene and chitosan can produce an adsorbent with enhanced selectivity. This study aims to develop and optimize a bentonite–graphene–chitosan–based nanocomposite adsorbent for removing Fe3+ ions from contaminated water. The nanocomposite was prepared through the synthesis of graphene oxide, activation of bentonite, and incorporation of chitosan, followed by characterization using XRD and FTIR. The FTIR and XRD results indicate that each component retains its characteristic features, and graphene oxide is successfully formed in an amorphous structure. The combination of these three materials shows strong potential as an Fe3+ adsorbent due to the synergistic interaction among the cation-exchange capacity of bentonite, the active surface of graphene oxide, and the amino functional groups of chitosan.

Keywords: Bentonite, Chitosan, Graphene, Nanocomposite.

Introduction

Advances in technology have driven the development of numerous alternative innovations to meet societal needs. In the field of materials, high-quality materials with superior mechanical properties are increasingly required. Nanocomposites represent one promising alternative for producing materials with enhanced mechanical performance compared to conventional materials. The fabrication of nanocomposites typically involves the use of fillers and a matrix. Fillers serve as reinforcing elements that play a critical role in determining the mechanical properties of the nanocomposite, as they transmit the load transferred from the matrix.

The Jambi Province possesses substantial natural resources with strong potential for use as adsorbent materials, particularly bentonite. Bentonite is a montmorillonite clay known for its high adsorption capacity as an absorbent agent (Srasra and Bekri-Abbes 2020). In Jambi Province, bentonite deposits are distributed across several regions, including Merangin, Bungo, and Batang Hari Regencies, with an estimated reserve of 160,143,200 tons. However, bentonite exhibits limitations in adsorbing pollutants, especially heavy metals, due to its low selectivity. Therefore, modification is required to enhance its adsorption capacity. Several approaches have been employed to improve bentonite's performance, such as bentonite pillaring (Permana et al. 2024), bentonite/chitosan composites (Majiya et al. 2024; Permana et al. 2025), and bentonite/SiO composites (Yahya et al. 2024).

Bentonite is a clay mineral commonly used as a filler in composites due to its hydrophilic nature and high adsorption capability. Graphene, a two-

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dimensional carbon material, provides exceptional mechanical strength and high conductivity, making it suitable as a reinforcing agent in nanocomposites. Chitosan, a biopolymer derived from chitin, is often employed as the matrix owing to its biodegradable and biocompatible characteristics.

The utilization of bentonite/graphene/chitosan nanocomposites is not yet widely recognized by the public; however, these materials hold significant potential in biomaterial applications, such as the fabrication of implants and medical devices. The chitosan matrix functions to distribute the applied load throughout the reinforcing components of the nanocomposite. Typically, matrix materials exhibit ductile behavior.

The combination of bentonite/graphene/chitosan composites can produce an adsorbent with a large surface area and strong metal-ion binding capability. Bentonite possesses a high cation-exchange capacity and a large surface area (Oliveira et al. 2016). Graphene oxide provides an active surface enriched with oxygen-containing functional groups that can strongly interact with heavy metals through complexation mechanisms (Gu and Fein 2015; Joel and Lujanienė 2022). Meanwhile, chitosan, a biopolymer obtained through the deacetylation of chitin, contains nucleophilic amino groups that effectively bind metal ions (Crini et al. 2017; Geeta et al. 2014). Consequently, the resulting nanocomposite is expected to exhibit strong affinity toward Fe^{3+} ions, with nanoscale adsorbent dimensions and a large surface area. A study by Lavelim et al. (2023) demonstrated that bentonite/graphene nanocomposites are effective in adsorbing Pb (II), achieving an adsorption efficiency of up to 87% and a capacity of 271 mg/g. However, the application of bentonite/graphene/chitosan nanocomposites for the adsorption of Fe^{3+} ions remains limited.

The aim of this study is to fabricate a bentonite/graphene/chitosan nanocomposite through chemical activation by synthesizing graphene into graphene oxide and activating bentonite, followed by characterization using XRD and FTIR to determine the material characteristics and verify the successful formation of the nanocomposite.

Materials And Methods

Materials

The equipment used in this study includes a set of glassware, an oven, a furnace, an analytical balance, and a 150-mesh sieve. The materials employed were chitosan derived from tilapia fish scales, H_2SO_4 (p.a.), CH_3COOH (p.a.), NaOH, graphite (99.8%), H_2O_2 (p.a.), and distilled water.

Methods

Preparation of Bentonite/Graphene/Chitosan Nanocomposites

Bentonite was physically activated at 250°C, while graphene was synthesized from graphite. The preparation of the bentonite/graphene/chitosan nanocomposite followed a modified procedure based on previous studies (Permana et al. 2025; Lan Huong et al. 2018). A total of 2.5 g of chitosan was dissolved in 350 mL of 0.5% (w/v) acetic acid and stirred at 650 rpm for 2 hours. Simultaneously, 2.5 g of activated bentonite/graphene was dispersed in 100 mL of distilled water and stirred at low speed for 1 hour. The bentonite/graphene suspension was then added to the chitosan solution under continuous stirring. Subsequently, 0.5 M NaOH solution was slowly added until the mixture reached a neutral pH, followed by further stirring for 3 hours. The resulting material was dried at 60°C for 6 hours. The bentonite/graphene/chitosan nanocomposite was characterized using XRD and FTIR to determine its structural properties and functional groups.

Data Analysis

Determination of Particle Size Using X-Ray Diffraction (XRD)

The X-ray diffraction (XRD) data obtained in this study were processed to determine the crystalline particle size using the Scherrer equation, expressed as follows:

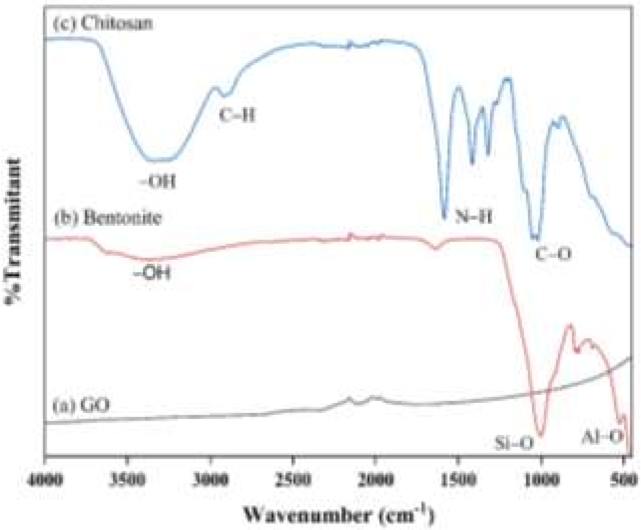

Figure 1. FTIR spectra of graphene oxide (GO), bentonite, and chitosan

$$D = \frac{K\lambda}{\beta \cos(\theta)} \quad (1)$$

Keterangan :

λ : Wavelength of the X-ray used

θ : Diffraction angle

K : Shape factor (0,94)

β : Full Width at Half Maximum (FWHM)

Results And Discussion

The FTIR characterization results for graphene oxide (GO), bentonite, and chitosan are presented in **Figure 1**. **Figure 1a** shows the FTIR spectrum of graphene oxide (GO) synthesized from activated carbon. The spectrum appears relatively flat, with no significant absorption corresponding to specific functional group vibrations. This phenomenon is attributed to the synthesis process, which employed a highly concentrated sulfuric acid solution (6 M), potentially causing degradation of the functional groups originally present in graphene (Zhang et al. 2023).

Figure 1b shows the FTIR spectrum of the activated bentonite. The absorption band at 3360 cm^{-1} corresponds to the vibrational stretching of –OH groups. The characteristic vibration of the Si–O bond appears at 1006 cm^{-1}, while the Al-O bond vibration is observed at 535 cm^{-1} (Chen et al. 2013; Daou et al. 2025).

The FTIR spectrum of chitosan is presented in **Figure 1c**. The characteristic peak at 3311 cm^{-1} corresponds to –OH vibrational stretching, while the absorption at 2890 cm^{-1} represents the C–H stretching vibration. The N–H absorption band appears at 1580 cm^{-1}, and the band at 1030 cm^{-1} is attributed to the characteristic C–H functional group (Mozaffari et al. 2019; Delgado-Cedeño et al. 2022; Fuhr et al. 2025).

The XRD characterization results of bentonite, chitosan, and graphene oxide (GO) are presented in **Figure 2**. **Figure 2a** shows the diffractogram of bentonite. The peak at $2\theta = 28.65°$ corresponds to quartz, while the peaks at $2\theta = 33.91°$, 47.75°, and 52.17° represent characteristic reflections of montmorillonite (Mudasir et al. 2020; Permana et al. 2025). Based on these XRD results, it can be concluded that montmorillonite is the primary mineral component of the bentonite. **Figure 2b** displays the diffractogram of chitosan, where a sharp peak at approximately $2\theta = 27.61°$ is observed, which is typical of pure chitosan (Satpathy et al. 2021). **Figure 2c** presents the diffractogram of graphene oxide (GO). The absence of high-intensity peaks and the presence of a broad hump at around $2\theta = 20–30°$ indicate that the synthesized graphene oxide possesses an amorphous structure (Tiwari et al. 2025; Stobinski et al. 2014).

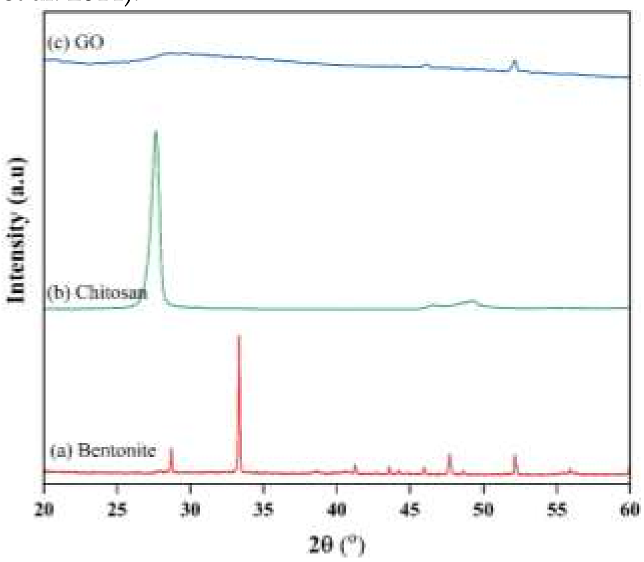

Figure 2. XRD diffractograms of bentonite, chitosan, and graphene oxide (GO)

Conclusions

This study successfully synthesized a bentonite/graphene/chitosan nanocomposite through bentonite activation, oxidation of graphite into graphene oxide (GO), and subsequent incorporation with chitosan. The FTIR and XRD results indicate that each component retained its characteristic features, and the graphene oxide was successfully formed with an amorphous structure. The combination of these three materials demonstrates strong potential as an Fe^{3+} adsorbent due to the synergistic interaction among the cation-exchange capacity of bentonite, the active surface of graphene oxide, and the amino functional groups of chitosan.

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Conflict of Interest: The authors declare that there are no conflicts of interest concerning the publication of this article.

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