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Comparative Adsorption Performance of Chitosan and Iron-Modified Chitosan for the Removal of a Synthetic Textile Dye †

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Abstract

The present work focuses on the removal of a textile dye from wastewater through adsorption using industrial chitosan and iron-modified chitosan as adsorbents. Characterization was performed through infrared spectroscopy pH point of zero charge analysis. Kinetic results showed that iron modification significantly improved adsorption capacity, achieving 96% removal within 120 min. The initial pH strongly influenced performance: industrial chitosan was more efficient in basic solutions, while iron-chitosan worked best under acidic conditions. Moderate agitation optimized adsorption rate and retention. Findings confirm the promising potential of both materials, particularly iron-chitosan, for textile wastewater treatment applications.

Keywords: chitosan; textile dyes; wastewater treatment; adsorption

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1. Introduction

Water is essential for sustaining life, yet its quality is increasingly compromised by industrialization and human activities that release heavy metals, dyes, pesticides, fertilizers, and other pollutants [1]. Among these, dyes are particularly alarming: over 100,000 are produced annually, exceeding 70,000 tonnes, with nearly 15% lost during dyeing operations [2]. Their complex structures make them poorly biodegradable and often toxic or carcinogenic, posing severe environmental and health risks [3]. Thus, protecting water resources is crucial. Conventional wastewater treatments—adsorption, coagulation, membrane filtration, reverse osmosis, ozonation, sedimentation, advanced oxidation, Fenton, and photocatalysis—are widely applied [4–12], yet balancing efficiency, cost, and sustainability remains challenging. Natural adsorbents offer significant advantages such as biodegradability, low energy demand, and high efficiency [13]. Chitosan, a biopolymer derived from chitin, stands out for its biocompatibility, biodegradability, antimicrobial activity, and functional groups that act as binding sites for dyes [14–16]. This study investigates both commercial and iron-modified chitosan as adsorbents for the removal of trisodium ferric complex of N-methyl-1,8-naphtalimide-4-sulfonate which is a textile dye, providing a sustainable approach to valorize biological waste while advancing wastewater treatment applications.

2. Materials and Methods

2.1. Dye Preparation

In this study, the selected dye corresponds to a trisodium ferric complex of N-methyl-1,8-naphthalimide-4-sulfonate, characterized by a 1:3 metal–ligand coordination structure and extensive use in textile processes

2.2. Adsorbent Preparation

Chitosan was functionalized by dispersing 100 g of chitosan in 200 mL of distilled water containing 10 g of ferric chloride hexahydrate and 5 g of ferrous chloride tetrahydrate. The suspension was stirred at room temperature for 10 min to initiate interaction. Subsequently, 37.5 mL of a 15% aqueous ammonia solution was slowly added under continuous stirring. The resulting material was dried at 60 °C to obtain the iron-loaded chitosan.

2.3. Adsorbent Experiments

Kinetic adsorption experiments were performed at room temperature by combining 300 mL of a 100 mg L^{-1} dye solution with 1 g of chitosan in an Erlenmeyer flask under continuous agitation for 120 min. The concentration of dye remaining in solution was quantified at 710 nm using an Optizen POP UV–Vis spectrophotometer.

3. Instrumental Analysis

Fourier Transform InfraRed Spectroscopy

The FTIR spectra of chitosan and iron-modified chitosan were recorded using a PerkinElmer Spectrum Two spectrometer over the wavenumber range of 4000–400 cm^{-1} (Laboratory of Inorganic and Environmental Chemistry, University of Tlemcen, Algeria). The samples were prepared as KBr pellets by thoroughly mixing approximately 1 mg of finely ground sample with 100 mg of dry spectroscopic-grade KBr, followed by pressing the mixture into a transparent disc using a hydraulic press. The obtained pellets were then placed in the sample holder for spectral analysis.

The Fourier **T**ransform **I**nfra**R**ed **spectrums of both materials are presented in Figure 1.**

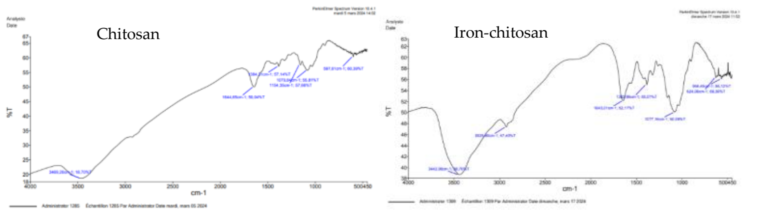

Figure 1. FTIR spectroscopy of chitosan and iron-chitosan.

The infrared spectra of industrial chitosan and iron–chitosan exhibit several characteristic absorption bands [17,18].

For industrial chitosan, a broad band appears at 3469 cm^{-1}, while for iron–chitosan it is slightly shifted to 3442 cm^{-1}, corresponding to the stretching vibrations of N–H and O–H groups. The bands observed between 2925 cm^{-1} and 2975 cm^{-1} for industrial chitosan and at 2925 cm^{-1} for iron–chitosan are attributed to the C–H stretching vibrations of CH_2 and/or CH_3 groups. The absorption bands at 1644 cm^{-1} and 1643 cm^{-1}, respectively, are assigned to the stretching vibrations of the C=O groups.

The bands located at 1550 cm^{-1} in industrial chitosan and 1560 cm^{-1} in iron–chitosan correspond to the N–H bending vibrations. Peaks appearing at 1343 cm^{-1} and 1383 cm^{-1}

are associated with C–H bending in CH_3 groups. Similarly, the bands at 1305 cm^{-1} and 1300 cm^{-1} are due to C–N stretching vibrations. The absorptions at 1154 cm^{-1} for industrial chitosan and 1160 cm^{-1} for iron–chitosan indicate C–O bending vibrations, whereas those at 1079 cm^{-1} and 1077 cm^{-1} correspond to C–O–C stretching vibrations. Finally, the bands observed at 597 cm^{-1} and between 566–624 cm^{-1} are assigned to the C–H bending vibrations.

4. Results

4.1. Adsorption Kinetics

Adsorption kinetics describe the variation over time of pollutant uptake [19]. To establish the optimal time of contact, dye adsorption capacity was monitored as a function of time (Figure 2). Both adsorbents showed a fast initial adsorption phase, which then slowed down progressively until equilibrium, due to progressive full occupancy of active sites [20]. Equilibrium was reached after 120 min for both materials. At this stage, industrial chitosan achieved a removal efficiancy of 88%, whereas iron-modified chitosan reached 96%, confirming the enhanced efficiency of the iron-modified adsorbent.

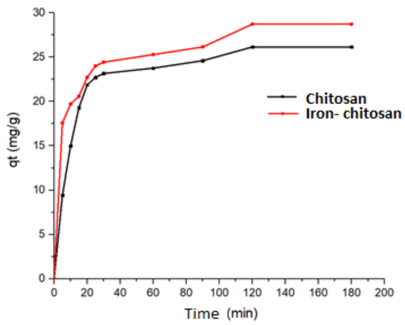

Figure 2. Kinetic study of textile dye adsorption onto industrial and iron-modified chitosan C_{dye} = 100 mg/L, $V_{solution}$ = 300 mL, m_{Ads} = 1 g, pH= natural, constant T, stirring speed = medium.

4.2. Influence of the Initial pH on the Adsorption Process

To assess the effect of initial pH on adsorption performance, 300 mL of a 100 mg/L solution were mixed with 1 g of adsorbent. Solution pH was adjusted from 2 to 12 using 0.1 M HCl or 0.1 M NaOH.

Figure 3 presents the equilibrium dye adsorption on both adsorbents as a function of pH.

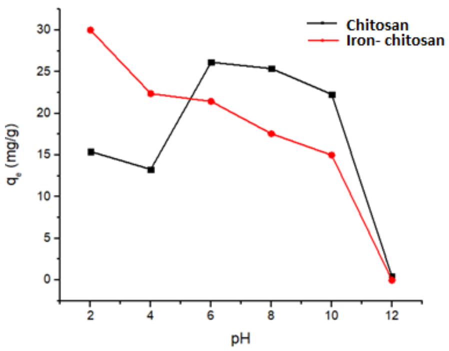

Figure 3. Influence of initial pH on the adsorption process of the textile dye onto industrial chitosan, C_{dye} = 100 mg/L, $V_{solution}$ = 300 mL, m_{Ads} = 1 g, constant T, stirring speed = medium.

The influence of pH on dye adsorption was evaluated, with pHzpc determined as 8 for industrial chitosan and 6.2 for iron-modified chitosan. For industrial chitosan, adsorption increased from pH 2–8 due to electrostatic attraction between the positively charged surface and anionic dye, but decreased above pHzpc because of repulsion. For iron-modified chitosan, maximum adsorption occurred at pH 2 (30 mg/g), then progressively declined with increasing pH, reaching nearly zero at pH 10, also due to electrostatic repulsion.

A comparable pattern was observed in a prior study [21,22], where the influence of pH on the adsorption of anionic dye exhibited similar trends throughout the tested range

4.3. The Impact of Stirring Rate

A 100 ppm dye solution (300 mL) was treated with 1 g of adsorbent to examine how agitation speed affects adsorption efficiency on adsorption was assessed (0, 400, and 900 rpm) (Figures 8 and 9). For both chitosan types, adsorption increased from static to moderate agitation due to improved mass transfer [23], but decreased at high agitation, likely from partial desorption [24]. Across all speeds, iron-modified chitosan showed higher adsorption than industrial chitosan, confirming the positive impact of modification on adsorptive performance.

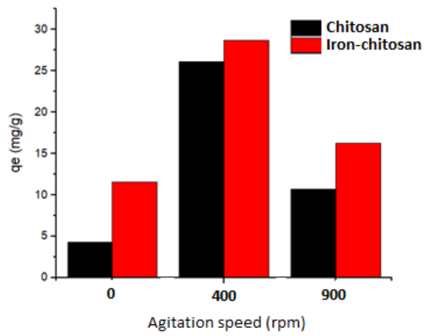

Figure 4. Kinetic of stirring rate effect on the adsorption of the textile dye onto chitosan and iron-chitosan, $C_{dye} = 100$ mg/L, $V_{solution} = 300$ mL, $m_{Ads} = 1$ g, constant T, pH natural.

Kinetic analysis showed that the highest adsorption rates were achieved at medium agitation speed, with rate constants of 0.0989 min^{-1} ($R^2 = 0.99$) for industrial chitosan and 0.0962 min^{-1} ($R^2 = 0.99$) for iron-modified chitosan.

5. Conclusions

This study evaluated the adsorption performance of industrial chitosan and iron-modified chitosan for textile dye removal from wastewater. Kinetic analysis revealed that iron modification significantly improved adsorption efficiency, achieving 96% removal with equilibrium reached in 120 min. The initial solution pH was a key factor: While industrial chitosan performed optimally in basic conditions, iron-chitosan achieved better adsorption in acidic environments. Moreover, medium agitation speed enhanced both adsorption rate and capacity for both materials, outperforming without agitation or high stirring conditions. These findings underscore the potential of chitosan, especially its iron-modified variant, as an efficient biosorbent for wastewater treatment.

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Conflicts of Interest:

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