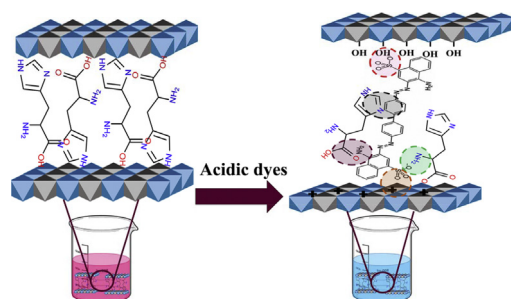


Fabrication of zwitterionic histidine/layered double hydroxide hybrid nanosheets for highly efficient and fast removal of anionic dyes

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GRAPHICAL ABSTRACT



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ABSTRACT

In this work, the bio-nanohybrids of magnesium-aluminum layered double hydroxide intercalated with zwitterionic histidine (His-LDH) was synthesized. The crystal phase, morphology, and nanostructure of the as-prepared His-LDH were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FT-IR), and nitrogen adsorption-desorption methods. The His-LDHs were used to remove anionic dyes, including Congo red (CR), indigo carmine (IC) and sunset yellow FCF (SY) from aqueous solutions. The detailed investigation of the kinetics and the adsorption isotherms of CR, IC and SY from aqueous solutions showed that the dyes adsorb rapidly, in accordance with a pseudo-second-order kinetics and a Freundlich adsorption isotherm model. The remarkably high adsorption capacity of the dyes on the His-LDH (efficiency of CR removal, 99.98%; maximum specific removal q_{max} , 1112 mg g^{-1} ; efficiency of IC removal, 98.98%; q_{max} , 625 mg g^{-1} ; and efficiency of SY removal, 99.78%; q_{max} , 400 mg g^{-1}) is rationalized on the basis of electrostatic interactions as well as π - π and H-bonding interactions between the His-LDH adsorbent and the acidic dyes. Adsorption experiments indicate that the resulting His-LDH has great potential applications as an environmentally friendly material for the swift removal of acidic dyes from aqueous solutions.

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1. Introduction

A growing number of contaminants are entering water supplies through human activities, resulting in inadequate access to clean

water for the growing global population. Organic dyes are widely used as coloring agents in many industries, such as textile, dyeing, paper and pulp, tannery and paint, and hence the effluents of these industries tend to contain dyes in excessive quantities [1]. The discharged organic dyes, almost all soluble in water, are difficult to remove due to their poor biodegradability [2], and some are even toxic or carcinogenic to humans and will jeopardize the environment [3,4]. Increasingly, and due to the demand for advanced

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clean-water technologies, it is becoming more and more urgent to purify water with a lower cost, less energy, improved safety, minimum use of chemicals and a minimum impact on the environment [5,6]. Therefore, their removal from industrial effluents as most urgent task make possible to either a safe and clean environment. To remove dyes and other colored contaminants from wastewaters, several physical, chemical, and biological methods such as membrane separation, flocculation-coagulation, adsorption, ozonation, ion exchange, oxidation, and aerobic or anaerobic treatment have been developed [7]. However, these techniques suffer certain limitations including cost-effectiveness, production of toxic byproducts hazardous to the environment, production of a large amount of sludge, and low adsorption capacity or inadequate affinity in removing dye from wastewater. However, the removal processes based on adsorption are much more popular because of their high efficiency, simple operation and lower price. Adsorption is a complex phenomenon and involves the passive separation of the adsorbate from an aqueous phase onto the solid phase. It occurs between two phases in transporting pollutants from one phase into another [7].

It has been reported that many different types of adsorbents are effective in removing color from aqueous effluents. The most commonly used adsorbent in industrial wastewater treatment systems is activated carbon because it has a large specific surface area, but it is expensive to run such systems [8]. The cost of this process has motivated numerous studies on alternative removal methods that uses less expensive natural materials and waste by-products such as chitosan, bagasse pith, peanut hull, sludge, perlite, rice husk, wood sawdust, montmorillonite, sepiolite, zeolite, diatomite, sawdust, bentonite, organophilic bentonites and layered double hydroxides [9–14]. Among these, layered double hydroxides (LDHs) have attracted more attention not only because of their low-cost but also because of their excellent adsorption performance as well as other potential applications, such as catalysts, ion exchange hosts, decolorizing agents and so on [15,16].

Layered double hydroxides (LDHs), also known as anionic or hydrotalcite-like clays, are a class of lamellar compounds that consist of positively charged brucite-like host layers and hydrated exchangeable anions located in the interlayer gallery for charge balance [17]. The charge of the brucite-like layers arises from the isomorphous substitution of a part of the divalent metal ions with the trivalent ones. The chemical composition of LDHs are expressed by the general formula $[M^{II}_1-xM^{III}_x(OH)_2][A^{n-}_x/n, mH_2O]$, where M^{II} and M^{III} represent di- and trivalent metal ions within the brucite-like layers and A^{n-} is an interlayer anion [16–18]. The incorporation of organic guests into LDHs has already received some attention because of the potential uses of the inorganic-organic hybrids produced in catalysis, sorption, photochemistry, and electrochemistry [19]. Recently, the intercalation of biomolecules such as sugar [20], nucleotide [21], DNA [22], amino acids [23,24] and polypeptide [25] for LDH has been investigated in order to prepare novel biomolecule/LDH nanohybrids as bio-nanocomposite materials class.

Biomolecules are widely used as low-cost functional materials for diverse applications in materials chemistry. Amino acids, zwitterion functionalized materials containing both anionic and cationic groups, have received great attention in the past decade due to the presence of oppositely charged ionic groups on their surface and unique structures. Moreover, amino acids, where positive and negative charges are located in close proximity, exhibit alternative ion selectivity to standard anion and cation ion-exchangers [26]. These features make amino acids very promising materials for membrane, ion chromatography, removal of carbon dioxide, removal of heavy metal ions, and removal of organic dyes [26–28].

In this study, we report a new strategy for highly fast and extremely efficient removal of acidic dyes from aqueous solutions using

zwitterionic histidine intercalated layered double hydroxide (His-LDH). This inorganic-organic bio-nanohybrids were preparation as low cost, and green material for removal. Hence, to investigate the effect of zwitterionic histidine on the adsorption capacity and adsorption mechanism of LDHs, two MgAl-LDHs intercalated with different interlayer anions, including NO_3^- and histidine, were synthesized. For the removal of organic acidic dyes, the adsorption kinetics and isotherms were investigated.

2. Experimental

2.1. Chemicals and materials

All chemicals were of analytical reagent grade. L-histidine, Mg $(NO_3)_2 \cdot 6H_2O$ and Al $(NO_3)_3 \cdot 9H_2O$ salts were purchased from Sigma-Aldrich (Milwaukee, WI, USA). Analytical grade Congo red (CR), indigo carmine (IC) and sunset yellow FCF (SY) were procured from Sigma-Aldrich (Milwaukee, WI, USA) and were used without any further purification. The water consumed was purified on a Youngling ultrapure water purification system model Aqua MaxTM-ultra (Seoul, South Korea). Other chemicals applied were of analytical reagent grade or of the highest purity available.

2.2. Preparation of NO_3^- -LDH and His-LDH

His-LDH was directly synthesized using separate nucleation and aging steps [29]. Briefly, aqueous solution A (0.02 mol $Mg(NO_3)_2 \cdot 6H_2O$ and 0.01 mol $Al(NO_3)_3 \cdot 9H_2O$ in 300 mL of CO_2 -free deionized water) and solution B (0.015 mol histidine in 150 mL of CO_2 -free deionized water) were first prepared. The pH of solution B was adjusted to 10.0 by addition of 2 M of NaOH solution and then solution A was added dropwise to solution B while stirring. The solution pH was adjusted at 9.5–10.5. The resulting gelatinous precipitate was aged at 80 °C for 24 h. The final product was collected by centrifuge (4000 rpm for 5 min), washed with water and ethanol five times, and dried at 70 °C for 6 h.

2.3. Characterization techniques

The crystalline structure of the obtained samples was characterized by X-ray diffraction (XRD, X'Pert Pro Super, Philips Co., the Netherlands) with Cu $K\alpha$ radiation (1.5478 Å). The morphologies observations were carried out on field emission scanning electron microscope (FE-SEM, KYKY EM-3200). Energy-dispersive X-ray (EDX) maps were taken on the SEM with an EDX spectrometer. Particle size and morphology were determined by a transmission electron microscope (TEM) of EM10C (Zeiss, Germany) operating at 80 kV accelerated voltage. FT-IR spectra of the samples were recorded on a Thermo Nicolet NEXUS FI-IR spectrophotometer using the KBr pellet method in the wavelength range 400–4000 cm^{-1} at a resolution of 4 cm^{-1} . The specific surface area and pore diameter were characterized by the Brunauer–Emmett–Teller (BET) and Barrett-Joyner-Halender (BJH) methods based on N_2 adsorption isotherms at 120 °C for 12 h with the help of a BET surface area analyzer (ASAP 2020 apparatus, Micromeritics, USA).

2.4. Adsorption experiment

Adsorption experiments were carried out in batch mode. An amount of 10,000 $mg L^{-1}$ of the stock solution of the dye was prepared and diluted to different desired concentrations. Isothermal experiments were performed on solutions at different initial dye concentrations in the existence of a constant amount of the sorbent (10 mg) in 10 mL solutions at room temperature. The adsorption

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