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Study on Preparation and Absorption of Alizarin Red from Mg/Al Layered Double Hydroxides

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Abstract. Mg/Al-LDH was prepared by co precipitation method at pH 10.5, 85°C and hot water for 6 hours with controlling Mg/Al ratio (2:1 and 3:1) and mixing (at common stirring and ultrasonic stirring). The samples were characterized by the Fourier transform infrared spectroscopy and X-ray diffractometer analysis. Finally, the four group structure and excellent quality of hydrotalcite materials were obtained. Samples were calcined at 500°C for 4h, and then the effects of different influencing factors on the adsorption of alizarin red by the adsorbent were studied. The results show that the prepared materials are layered with hydrotalcite and have good adsorption performance. When the pH value is 10, adding 1g of hydrotalcite and performing adsorption at low temperature, the adsorption rate reaches a maximum of 99%.

Keywords: Mg/Al-LDH; Alizarin red; Co-precipitation; Adsorption.

1. Introduction

With the continuous development of industry, the environmental and resource crisis has become increasingly serious^[1-5]. In particular, the random discharge of untreated industrial wastewater has caused problems of water shortage and environmental pollution^[6], which seriously affected human life and productivity^[7], and caused widespread concern^[8]. Water pollution has been induced by dyes, heavy metals, pesticides, surfactants and other toxic substances^[9-11]. Among them, the wastewater of the printing and dyeing industry has a high content of organic pollutants, especially chromaticity pollution^[12], which is a major problem that is difficult to handle in industrial wastewater. For difficult-to-degrade dyes such as azo dyes, it is important to find a proper treatment^[13]. As a result, many scientists experiment with different methods, such as physical methods and chemical methods^[14]. Chemical treatment of organic dyes is also an extremely important one^[15]. Generally, chemical oxidation and coagulation sedimentation are commonly used to treat organic pigments in industrial wastewater^[21]. With the development of technology, electrolytic oxidation, micro-electrolytic oxidation, and photocatalytic oxidation have been studied. But chemical methods produce larger toxins^[16]. Among physical methods, the adsorption method is the most widely used. The basic principle of the adsorption treatment method is to contact the adsorbent powder or particles with the wastewater, causing the dye molecules to be adsorbed on the surface of these porous materials and then filtered to remove them^[17]. At present, activated carbon diatomaceous earth and resin and granular activated carbon are the most commonly used adsorbents^[18]. However, these adsorbents are generally not reusable, and they are expensive and have low recycling rates^[19]. LDH and their calcined products can be used as anions due to their large specific surface area, large number of alkaline sites, small and uniform structure size, good stability, easy acceptance of guest water molecules, and "memory effect"^[20]. Because of its special "memory effect", the hydrotalcite-like substance can be reversibly restored to its original structure under certain conditions after its structure is



changed in a certain way. Therefore, it can be reused as an adsorbent and can be used as an environmentally friendly adsorbent for pollutants. The main purpose of this study is to prepare excellent performance based on LDH. A good alizarin red sorbent has been studied by various factors influence, so as to explore its efficient removal of the alizarin red, in order to removal of alizarin red from water by LDH as adsorbent provide theoretical guidance.

2. Experiment

2.1. Materials

Magnesium nitrate ($\text{Mg}(\text{NO}_3)_2$, A.R.), Aluminum nitrate ($\text{Al}(\text{NO}_3)_3$, A.R.), Sodium hydroxide (NaOH , A.R.), Alizarin Red (A.R.). Alizarin Red was purchased from Aladdin Co., Ltd and other chemical agents were provided by Tianjin Tianli Chemical Reagent Factory.

2.2. Synthesis

Weigh two sets of magnesium nitrate and aluminum nitrate ($\text{Mg}:\text{Al} = 3:1$ and $\text{Mg}:\text{Al} = 2:1$), respectively add 150 mL of distilled water, add them to a three-necked flask, and stir with a stirrer at room temperature for 0.5 h. During the stirring process, a 5% sodium hydroxide solution was continuously added dropwise until the pH value of the mixed solution reached about 10, and heated in a water bath at 85°C for 6 hours. After suction filtration and drying, the target Mg/Al -LDH was obtained, which called Sample c and d. Then weigh two sets of magnesium nitrate and aluminum nitrate ($\text{Mg}:\text{Al} = 3:1$ and $\text{Mg}:\text{Al} = 2:1$), add 150 mL of distilled water, and stir with SC-III multi-frequency acoustic chemical generator for 0.5 h. Then, a 5% sodium hydroxide solution is added dropwise until the pH of the mixed solution reaches about 10, and the subsequent steps are as above to finally obtain the target product, which called Sample a and b. Four kinds of hydrotalcite were synthesized by hydrothermal-co-precipitation method with different stirring methods and different nitrate ratios and calcined at 500°C for 4 h to obtain hydrotalcite adsorbent for subsequent adsorption of alizarin red.

2.3. Characterization

The structures of the products were detected by a X-ray diffractometer (XRD) (D/max-2200PC Japan) using $\text{Cu K}\alpha$ radiation and graphite monochromator with scanning speed $8^\circ/\text{min}$, and sampling width 0.02. The fourier-transform infrared (FT-IR) was recorded on an infrared spectrometer (PRISTIGE-21, Japan) in the range of $4000\text{--}500\text{ cm}^{-1}$ by KBr compression.

2.4. Determination of Alizarin Red Content

- (1) Accurately weigh 0.60 g of hydrotalcite adsorbent into a 25 mL erlenmeyer flask, add 10 mL of Alizarin Red standard solution $2.5\text{ }\mu\text{g}/\text{mL}$, and add hydrochloric acid or hydroxide to adjust its pH to 6, 7, 9, 10, 11, 12, 13. The conical flask was placed in a water bath with a constant temperature shaker (200 r/min) at 25°C for 2 h to study the effect of pH on the adsorption.
- (2) Accurately weigh 0.50 g of hydrotalcite adsorbent into a 25 mL erlenmeyer flask, add 10 mL of Alizarin Red standard solution $2.5\text{ }\mu\text{g}/\text{mL}$, and the pH value is 10. Place the Erlenmeyer flask in a water bath with a constant temperature shaker (200 r/min) at 20°C , 25°C , 30°C , 35°C , 40°C , 45°C , 50°C for 2 h to study the temperature effect on adsorption.
- (3) Accurately weigh 0.20 g, 0.40 g, 0.60 g, 0.80 g, 1.0 g, 1.2 g, 1.4 g, 1.6 g, 1.8 g of hydrotalcite adsorbent into a 25 mL conical flask, and add 10 mL alizarin red standard The solution was $2.5\text{ }\mu\text{g}/\text{mL}$ and the pH was 10. Place the erlenmeyer flask in a water bath with a constant temperature shaker at 25°C for 2 h to study the temperature effect on adsorption.

2.5. Reusable Performance

0.60 g of LDH adsorbent was added to Alizarin Red standard solution, and the amount of adsorption was measured after a certain period of adsorption. Filter the adsorbent, add it to 0.1 mol/L HCl, dissolve it, analyze the alizarin red ion, add 5% sodium hydroxide solution, adjust the pH to about 10, stir, suction filter, and dry, Calcined, and then subjected to adsorption experiments. This process was repeated 10 times to evaluate the change in adsorption amount of LDH.

3. Results and Discussion

3.1. Infrared Characterization of LDH

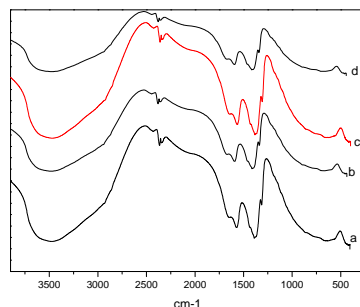


Figure 1. FTIR spectrum of LDH.

It can be seen from the figure that a broad peak around 3400 cm^{-1} is a symmetrical stretching vibration peak of the hydroxyl group between the magnesium-aluminum hydrotalcite layer, and the peak that appears around 1100 cm^{-1} is the hydrogen bond between water and anions in the layer Effect. Thus, the prepared hydrotalcite-like material is Mg/Al-LDH required for experiments.

3.2. XRD of LDH

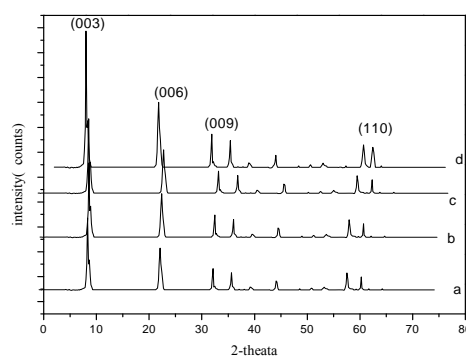


Figure 2. XRD spectrum of LDH.

It can be seen from the figure that four samples prepared have diffraction peaks of hydrotalcite, indicating that the synthesized samples have a typical layered structure of hydrotalcite-like substances. It has good crystallinity and complete crystal form. It was also found that with the increase of $n(\text{Mg}) / n(\text{Al})$, $d(003)$ decreased, and the interplanar spacing increased slightly, because the $n(\text{Mg}) / n(\text{Al})$ increased correspondingly, and the layer The plate charge density decreases accordingly, which weakens the electrostatic attraction between the positive charge of the layer and the anion between the layers, and increases the distance between the layers.

3.3. Effect of pH on the Adsorption of LDH

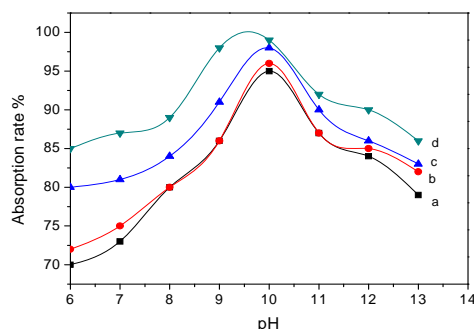


Figure 3. Effect of pH on the adsorption of LDH.

It can be clearly seen in the figure that as the pH value rises, the adsorption rate of alizarin red by LDH also gradually increases. When the pH value is 10, the adsorption efficiency is the highest. As the value increases, the adsorption rate decreases; among the four curves, the Mg / Al = 3: 1 hydrotalcite material prepared under ultrasonic stirring has the highest adsorption rate, so it can be concluded that Mg / Al = 3 prepared under ultrasonic stirring LDH with a ratio of 1: 1 has the highest adsorption rate when the pH value is 10, and the adsorbability of LDH with Mg / Al = 3: 1 produced under ordinary stirring is next. Under the environment of acid and alkali, it will affect the surface charge of hydrotalcite, the dispersion degree and the existing form of alizarin red, resulting in the weakening of flocculation and adsorption of dye by LDH, thus leading to the decrease of the adsorption rate.

3.4. Effect of Temperature on the Adsorption of LDH

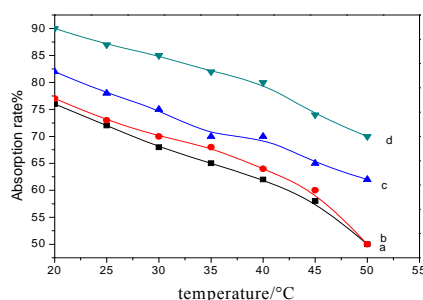


Figure 4. Effect of temperature on the adsorption of LDH.

As shown in Figure 4, the effect of temperature on LDH adsorption of alizarin red is also quite obvious. Obviously two conclusions can be drawn from the figure. First, when the temperature rises, the adsorption rate of hydrotalcite to alizarin red decreases significantly; second, the Mg / Al = 3: 1 LDH prepared under ultrasonic stirring among the four sets of curves has higher adsorption than other hydrotalcite materials. The adsorption rates are high, and the Mg / Al = 3: 1 LDH prepared by ordinary stirring is next. When the temperature rises, the surface tension of the adsorption solution decreases, and the intense thermal movement of molecules is not conducive to the adsorption of pigment by hydrotalcite. As the temperature rises, the crystal water between the hydrotalcite layers will also be affected, resulting in a change in the structure of the hydrotalcite layers and a decrease in the adsorption rate of pigments. The increase in temperature leads to a decrease in the surface tension of the solution, an increase in the thermal movement of the molecules, and the crystal water between the hydrotalcite layers is also affected, resulting in a decrease in the LDH adsorption rate.

3.5. Effect of Amount on the Adsorption of LDH

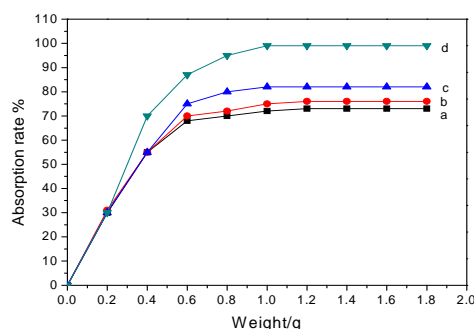


Figure 5. Effect of amount on the adsorption of LDH.

As shown in Figure 5, the effect of the amount of LDH on its adsorption of alizarin red is considerable. It can be clearly seen in the figure that as the amount of LDH is continuously added, the adsorption rate of alizarin red increases rapidly. However, when the amount of LDH is about 1 g, the adsorption rate of hydrotalcite is almost no longer affected. Among them, the LDH with Mg / Al = 3: 1 produced under ultrasonic agitation has better adsorptivity, and the adsorbability of LDH I with Mg / Al = 3: 1 produced under ordinary agitation is second. For this reason, the effective adsorption area of hydrotalcite lies in its surface area ratio. When the amount of LDH added reaches a certain value, the unit surface area ratio becomes a fixed value, so the addition of LDH no longer has a significant effect on the adsorption rate.

3.6. Reusable Performance of LDH Adsorbent

After regeneration LDH was subjected to 10 adsorption-desorption experiments, the adsorption capacity of hydrotalcite for alizarin red decreased by only 4.55% compared with the initial use of hydrotalcite, indicating that the hydrotalcite adsorbent has good reusability.

4. Conclusion

The hydrotalcite-coprecipitation method is used to prepare LDH with different stirring methods and different mixed salt ratios. Under the ultrasonic stirring, the hydrotalcite with a Mg / Al ratio of 3: 1 belongs to both performance and good regularity and crystal form. It was calcined at 500 ° C for 4h, and its adsorption of organic pigment was investigated. It is found that when the pH value is 10, about 1 g of Mg / Al = 3: 1 LDH prepared under ultrasonic stirring is added, and the adsorption effect is best at low temperature, which reusability is good. Compared with other adsorbents in the literature, the adsorption rate of this adsorbent is significantly increased. The adsorption rate of Mg / Al = 3: 1 adsorbent prepared under ultrasonic stirring can reach 99%, and the reusable performance is good. After the experiment only decreased by 4.55%.

Acknowledgements

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