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Original Research Article

Preparation and Comparison of Two Different Nanocomposite Kinds Based on MgZnAl-Layered Double Hydroxide for Simultaneous Removal of Cationic and Anionic Dyes

Fatemeh Tavoosi, Maryam Movahedi*, Nahid Rasouli

Department of Chemistry, Payame Noor University, P.O. Box 19395-3697 Tehran, Iran

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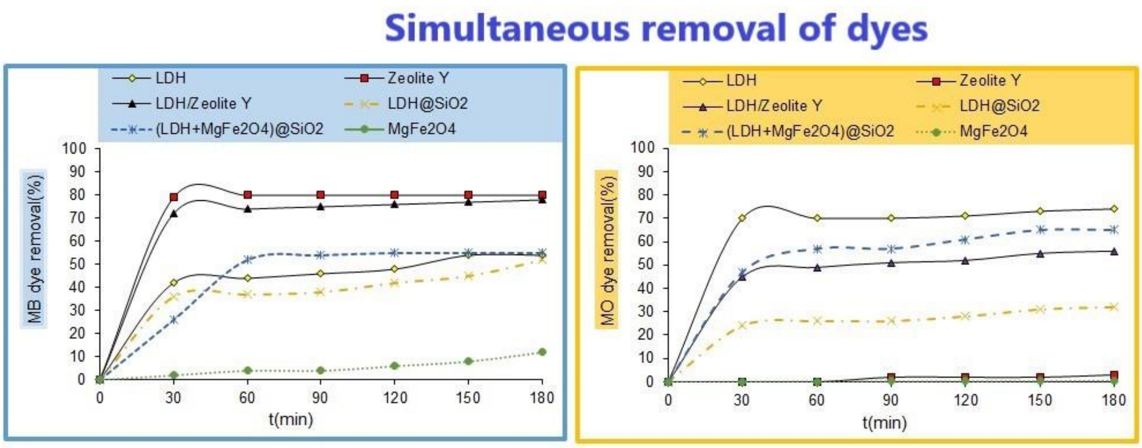
ABSTRACT

In this work, the MgZnAl-LDH/Zeolite Y and (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composites based on layered double hydroxide (LDH) were synthesized and characterized using X-ray diffraction (XRD), field emission scanning electron microscopy (FE-SEM), and energy-dispersive X-ray spectroscopy (EDX) analysis. The efficiency of the samples was assessed for simultaneous removal of cationic and anionic dyes from the solution. In this work, methylene blue and methyl orange as cationic and anionic dyes were used. The performance of prepared composites was also compared with their components. The results demonstrated that the Zeolite Y sample could only remove the methylene blue dye from the solution. The $MgFe_2O_4$ sample is not able to remove any of the anionic and cationic dyes. The simultaneous removal of the methylene blue and methyl orange dyes is observed by the MgZnAl-LDH sample. The MgZnAl-LDH/Zeolite Y and (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composites revealed similar performance to the MgZnAl-LDH. Moreover, the MgZnAl-LDH@SiO_2 composite showed lower efficiency compared with that of the MgZnAl-LDH. In addition, different kinetic models including, the pseudo-first-order, pseudo-second-order, and particle diffusion models were examined for the simultaneous removal of dyes. The kinetic data revealed the adsorption process could be well fitted by the pseudo-second-order kinetic model. The methylene blue dye removal by (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 sample occurred by the adsorption on the surface and intra-particle diffusion.

GRAPHICAL ABSTRACT



* Corresponding author: Movahedi, Maryam
✉ E-mail: m.movahedi@pnu.ac.ir

Introduction

Adsorption is a process that has been widely utilized to remove dye contaminants and treat the dye effluents [1-4]. The textile, paper, plastic, leather, food, and cosmetics industries often use dyes. The amount of excess dyes used enters the wastewater and if not properly treated, it will find its way to water sources. So far, various adsorbents including, natural adsorbents, composites, and polymer compounds have been employed to remove the dye contaminants from the effluent [5-7]. Among these minerals, oxides, silicates, aluminophosphates, and zeolites have been widely used [8-11]. Most pigments and products from their decomposition are carcinogenic and toxic. Therefore, removing these substances from the effluent is very important. The use of adsorbents for the treatment of industrial effluents and the reduction of pollutants is one of the methods of wastewater treatment. Layer double hydroxide (LDH) can be considered as a class of materials that are easy to synthesize in the laboratory. The simplest and most common method of synthesis is the precipitation method. LDHs with the general formula $[M^{+2}_{(1-x)}M^{+3}(OH)_2]^{X+}(A^{n-})_{x/n}.mH_2O$ where M^{2+} and M^{3+} represents bivalent and trivalent cations respectively, x equal to the molar ratio $M^{3+}/(M^{2+}+ M^{3+})$ and A^{n-} is interlayer anions [12, 13]. So far, LDHs layer compounds have been used as catalysts, modified electrodes, adsorbents, photocatalysts, drug release, and biosensors [14-18]. The preparation of various composites, especially magnetic composites, is considered due to its easier separation from the aqueous solution [19]. Until now, the removal of cationic and anionic contaminants of organic dyes has been investigated using various photocatalysts and adsorbents. Most adsorbents and photocatalysts have selective removal, and few can remove both cationic and anionic dyes simultaneously [20, 21]. In this research study, two different kinds of adsorbents based on LDH were synthesized. Then, the efficiency of the samples for simultaneous removal of methylene blue and methyl orange dyes from solution was compared. Furthermore, different kinetic models such as pseudo-first-order, pseudo-second-order, and particle diffusion models were investigated.

Experimental

Materials

Methylene blue dye, Methyl orange dye, $Mg(NO_3)_2.6H_2O$, $Fe(NO_3)_3.9H_2O$, $Zn(NO_3)_2.6H_2O$, $Al(NO_3)_2.9H_2O$, NH_4OH, C_2H_5OH, Na_2CO_3, NaOH, Tetraethyl orthosilicate and Zeolite Y were purchased from Sigma-Aldrich, and Merck Company.

Instrumentation

The composite materials synthesized in this work were characterized by XRD (Holland Philips Xpert, X-ray diffractometer with Cu-Kα radiation), field emission scanning electron microscopy, and energy dispersive X-ray spectroscopy (FE-SEM, EDX, Vega 2 Tscan). The dye concentrations were analyzed by a spectrophotometer (UV-Vis -Shimadzu-2550)

Preparation of MgZnAl-LDH

First, 100 mL salt solution containing $Al(NO_3)_2.9H_2O$ (1.87 g), $Mg(NO_3)_2.6H_2O$ (2.56 g), and $Zn(NO_3)_2.6H_2O$ (2.97 g) was prepared. The second stage, 100 mL of Na_2CO_3 (0.318 g) solution was added dropwise into the above solution. Then, the pH of the solution was adjusted to 9 by adding NaOH solution (5 M). The product formed was stirred at 70 °C for 24 h. Then the product was washed several times with distilled water and dried in an oven at 60 °C.

Preparation of MgZnAl-LDH/Zeolite Y

For a synthesis of the MgZnAl-LDH/Zeolite Y composite, 0.8 g Zeolite Y was dispersed into the 100 mL solution containing $Al(NO_3)_2.9H_2O$ (0.62

g), $Mg(NO_3)_2.6H_2O$ (0.85 g) and $Zn(NO_3)_2.6H_2O$ (0.99 g) salts. According to the MgZnAl-LDH procedure, 100 mL Na_2CO_3 (0.106 g) solution was added and the pH of the solution was adjusted to 9 by adding NaOH solution. Finally, the product after aging at 70 °C for 24 h was washed and dried in an oven at 60 °C.

Preparation of (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2

First, the $MgFe_2O_4$ sample was prepared. In this synthesis 300 mL solution containing $Fe(NO_3)_3.9H_2O$ (5.38 g) and $Mg(NO_3)_2.6H_2O$ (1.71 g) was prepared. Then, the pH of the solution was adjusted to 9 by adding NaOH solution (3M). The product formed was stirred at 70 °C for 24 h. Then the product was collected and washed with distilled water several times and dried in an oven at 60 °C. Finally, the product was calcined in a furnace at 700 °C for 2 h. Then, for preparation of the (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composite, 0.5 g MgZnAl-LDH and 0.3 g $MgFe_2O_4$ was ground in a mortar agate. Then, the mixture of MgZnAl-LDH and $MgFe_2O_4$ was dispersed in the solution containing of distilled water (20 mL), ethanol (60 mL), and ammonia (1 mL) using an ultrasonic bath for 30 min. Finally, 100 µL of tetraethyl orthosilicate was added and vigorously stirred for 2 h. The product was washed with distilled water and dried in an oven at 60 °C. In this work for the comparative study, the MgZnAl-LDH@SiO_2 sample was prepared with the same procedure.

Adsorption kinetic

A solution containing two dyes of methylene blue and methyl orange was used as a dye contaminant. The concentration for each dye was considered to be 5 mg/L. In one Erlenmeyer 0.3 g of adsorbent was added to 200 mL of the contaminant solution and then the resulting suspension mixture was stirred with a magnetic stirrer. The samples were taken at 30, 60, 90, 120, 150, and 180 min and the suspended particles were separated by a centrifuge and the residual dye concentration was measured using UV-Vis. spectrophotometry and dye removal percentage was calculated using the Equation 1. Where C_0 (mgl^{-1}) and C_t (mgl^{-1}) are initial dye concentration and dye concentration at time t.

$$\text{Dye removal}(\%) = \frac{C_0 - C_t}{C_0} \times 100 \qquad (1)$$

The Kinetic data were examined by pseudo-first-order, pseudo-second-order, and intra-particle diffusion models. The linear relationships of the equations are given in Equation 2, 3, and 4, respectively.

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad (2)$$

$$\frac{t}{q_t} = \frac{1}{k_2 (q_e)^2} + \frac{t}{q_e} \qquad (3)$$

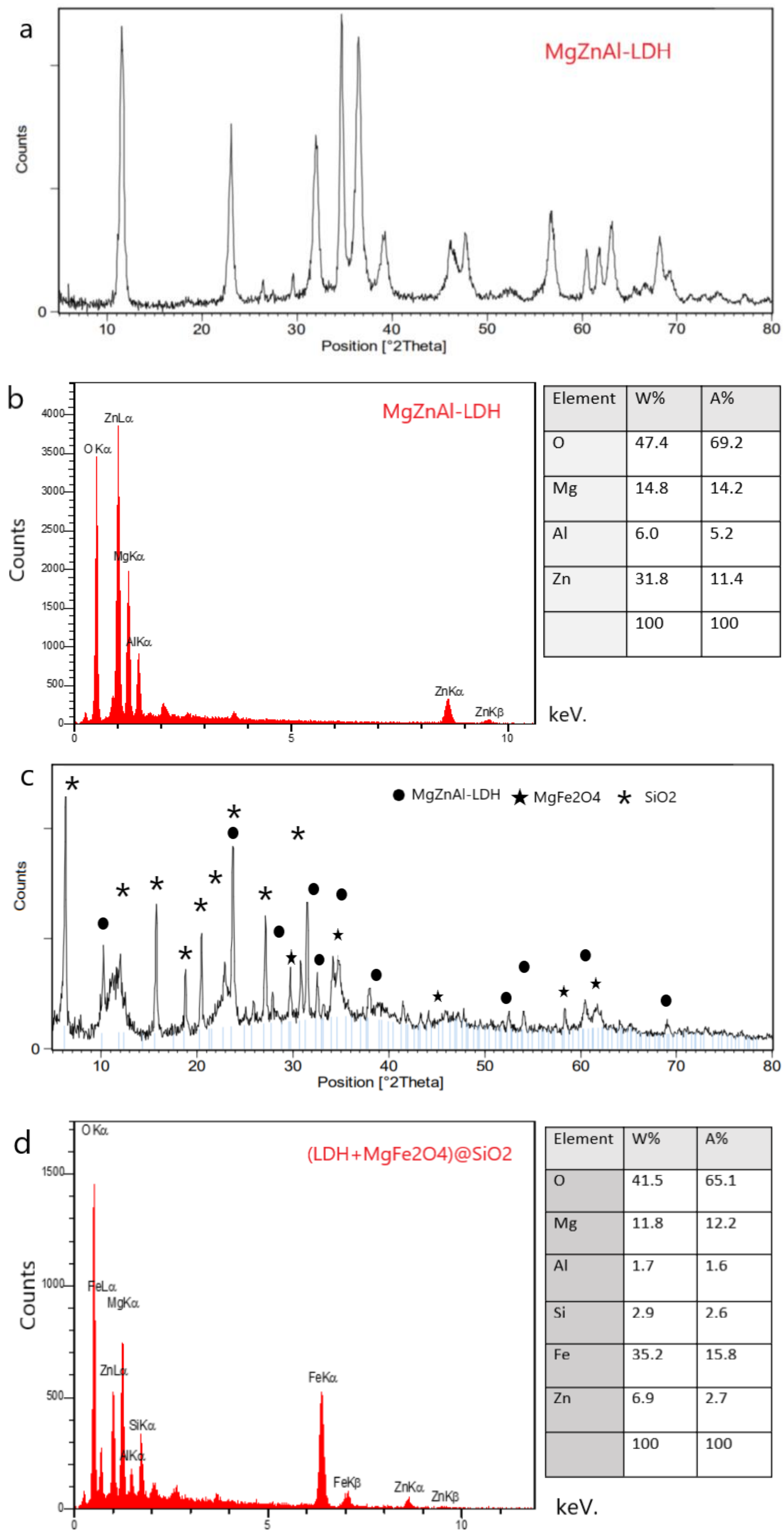
$$q_t = k_{id} t^{0.5} + C \qquad (4)$$

Where k_1, k_2, k_{id} are rate constants; q_e and q_t are the amount of adsorption (mgg^{-1}) at equilibrium and at time t, respectively [22-24].

Results and Discussion

Characterization

The X-ray diffraction pattern of the MgZnAl-LDH is demonstrated in Figure 1a. The main peak was seen at 2θ=11, indicating the formation of layer double hydroxide. Figure 1b shows the results of the EDX analysis of the MgZnAl-LDH, indicating the presence of the Mg, Zn, Al, and O elements in the sample. The XRD pattern of the (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composite is depicted in Figure 1c. Moreover, Figure 1d, demonstrates the EDX analysis of the (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 sample. The EDX analysis shows the presence of Mg, Zn, Al, Fe, Si, and O elements. Furthermore, the approximate weight and atomic percentage of the elements are revealed in Figures 1b and 1d.



Element	W%	A%
O	47.4	69.2
Mg	14.8	14.2
Al	6.0	5.2
Zn	31.8	11.4
	100	100

Element	W%	A%
O	41.5	65.1
Mg	11.8	12.2
Al	1.7	1.6
Si	2.9	2.6
Fe	35.2	15.8
Zn	6.9	2.7
	100	100

Figure 1. (a) XRD pattern (b) EDX analysis for the MgZnAl-LDH sample and (c) XRD pattern (d) EDX analysis for the (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composite

The FE-SEM image of the MgZnAl-LDH sample is presented in Figure 2a. In this image, the plate-like morphology is observed that the thickness of the plates is in the range of (14-80) nm. The FE-SEM image of the (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 sample is shown in Figure 2b. According to the FE-SEM images of the MgZnAl-LDH and (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 samples, a similar morphology was observed. For the MgZnAl-LDH/Zeolite Y sample, the particle morphology with a diameter range of 50-150 nm was observed in Figure 2c.

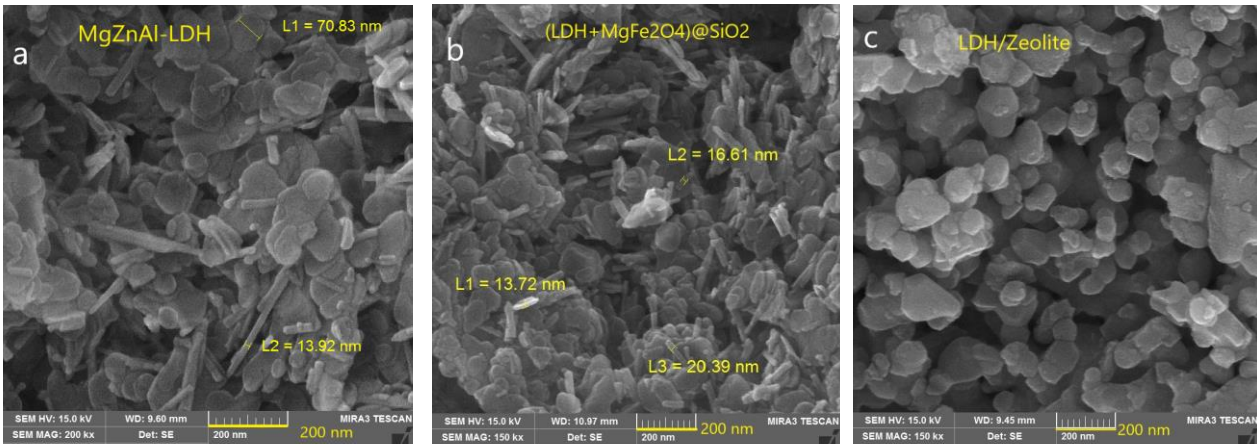


Figure 2. FE-SEM images of the (a) MgZnAl-LDH, (b) (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 and (c) MgZnAl-LDH/Zeolite Y

Adsorption study

In this study, the efficiency of the MgZnAl-LDH/ZeoliteY and (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composites were compared with their component (MgZnAl-LDH, Zeolite Y, MgZnAl-LDH@SiO_2 and $MgFe_2O_4$) for simultaneous removal of methylene blue and methyl orange dyes. Adsorption kinetics were also investigated. The results revealed that the Zeolite Y could selectively remove about 80% of the methylene blue cationic dye. The methyl orange anionic dye is not removed by the Zeolite Y. The MgZnAl-LDH sample can remove methylene blue and methyl orange dyes about 54% and 74%, respectively. The results indicated that the MgZnAl-LDH/ZeoliteY sample could remove about 78% and 56% of methylene blue and methyl orange dyes, respectively. Comparison of the (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composite with its components showed that it has a higher efficiency than the $MgFe_2O_4$ and MgZnAl-LDH@SiO_2 samples and can simultaneously remove methylene blue and methyl orange dyes about 55% and 65%, respectively. This study also revealed that the $MgFe_2O_4$ sample as a magnetic component is not suitable for the simultaneous removal of the dyes. The methylene blue (12%) and methyl orange (15%) were removed by the $MgFe_2O_4$ sample. Moreover, the efficiency of the MgZnAl-LDH@SiO_2 sample was investigated. For the MgZnAl-LDH@SiO_2 sample, the results showed the methylene blue (52%) and the methyl orange (32%) were removed. These results are shown in Figures 3a and 3b. The methylene blue is removed due to the hydroxyl group on the silica surface [25-27]. The ion exchange, precipitation, and adsorption mechanisms have been reported for the methylene blue adsorption on the surface of Zeolite [28]. The LDH as a "dual-electronic material" was introduced by many researchers [29]. The methyl orange molecules can be adsorbed on the surface of LDH by various mechanisms such as electrostatic interaction, metal ion complexation, and hydrogen bonding

[30]. Many researchers by the theoretical study depicted that the methylene blue dye removal occurred with "intercalate" between two surfaces of the LDH [31].

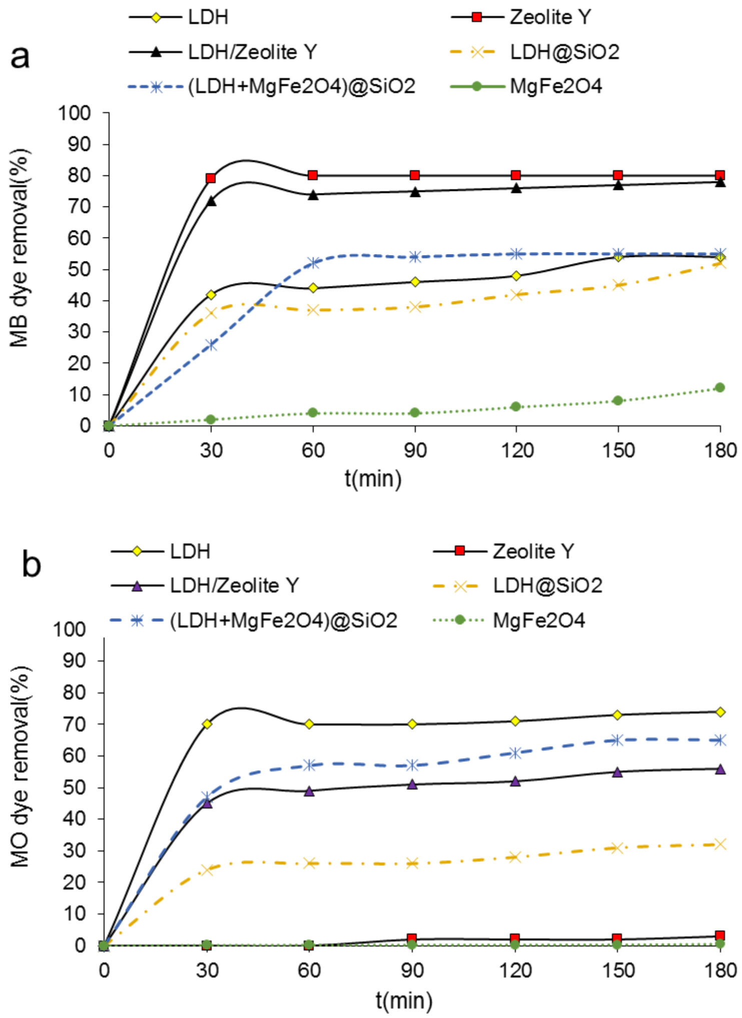
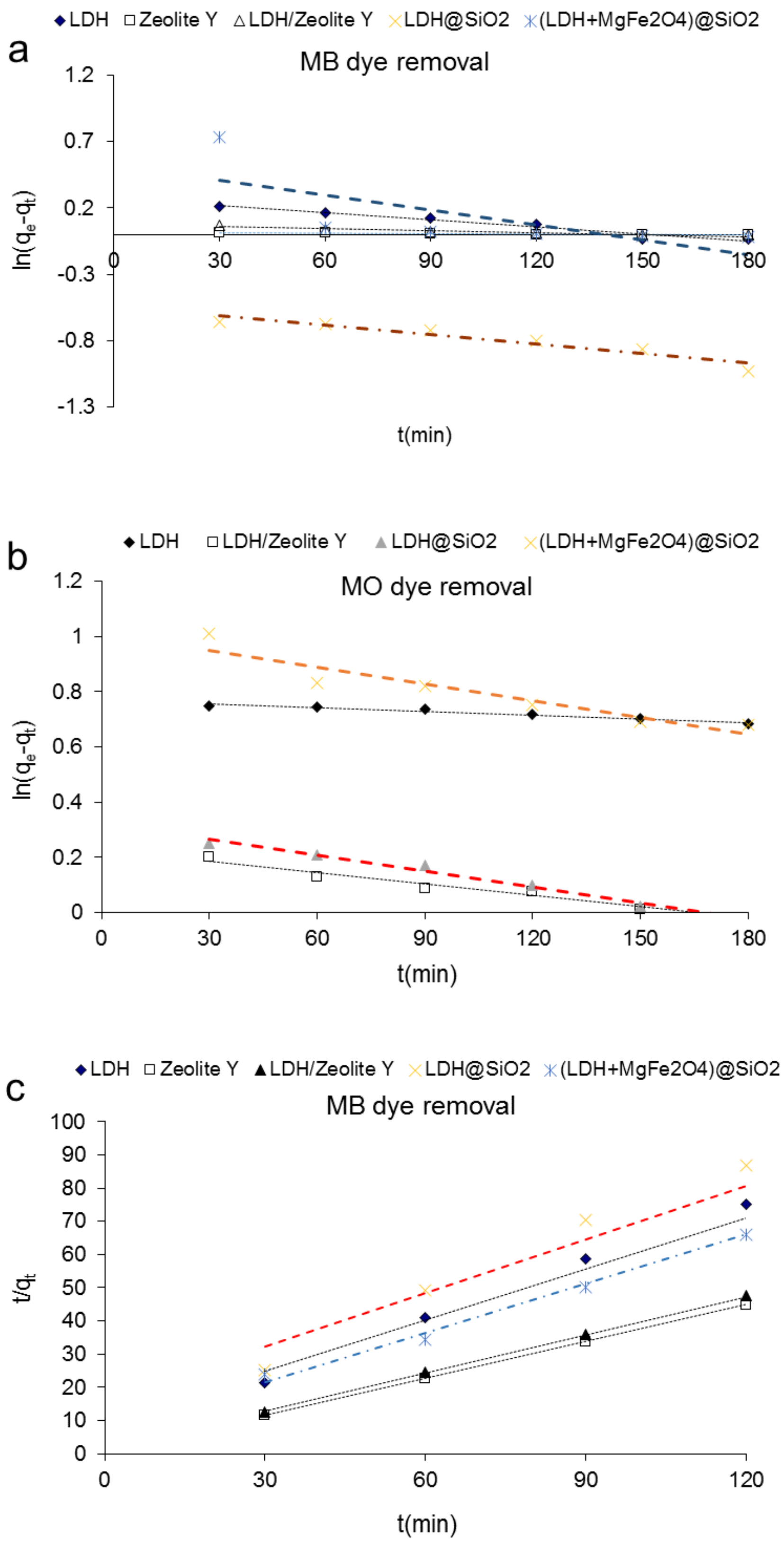


Figure 3. Simultaneous removal of methylene blue (a) and methyl orange (b) dyes by MgZnAl-LDH, Zeolite Y, MgZnAl-LDH/Zeolite Y, MgZnAl-LDH@SiO_2, (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 and $MgFe_2O_4$ samples

The kinetic behavior of the samples was investigated using the pseudo-first-order, pseudo-second-order, and intra-particle diffusion models (Figure 4a-f). The kinetic results showed that the simultaneous removal of dyes by adsorbents follows a pseudo-second-order kinetic model. The results are summarized in Table 1 and Table 2. In the particle diffusion model, most of the adsorption process of methylene blue and methyl orange dyes for samples occurs through transfer on the adsorbent surface because it is a straight line diagram that does not pass from the origin. However, many researchers reported that if the data exhibit multi-linear plots, then two or more steps influence the sorption process [32]. The process of removing methylene blue dye by (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 sample occurs by adsorption on the surface and intra-particle diffusion. In this case, the second linear portion is controlling with intra-particle diffusion.


a
◆LDH □Zeolite Y △LDH/Zeolite Y ×LDH@SiO2 ✳(LDH+MgFe2O4)@SiO2
MB dye removal
ln(qe-qt)
t(min)
b
◆LDH □LDH/Zeolite Y ▲LDH@SiO2 ×(LDH+MgFe2O4)@SiO2
MO dye removal
ln(qe-qt)
t(min)
c
◆LDH □Zeolite Y ▲LDH/Zeolite Y ×LDH@SiO2 ✳(LDH+MgFe2O4)@SiO2
MB dye removal
t/qt
t(min)

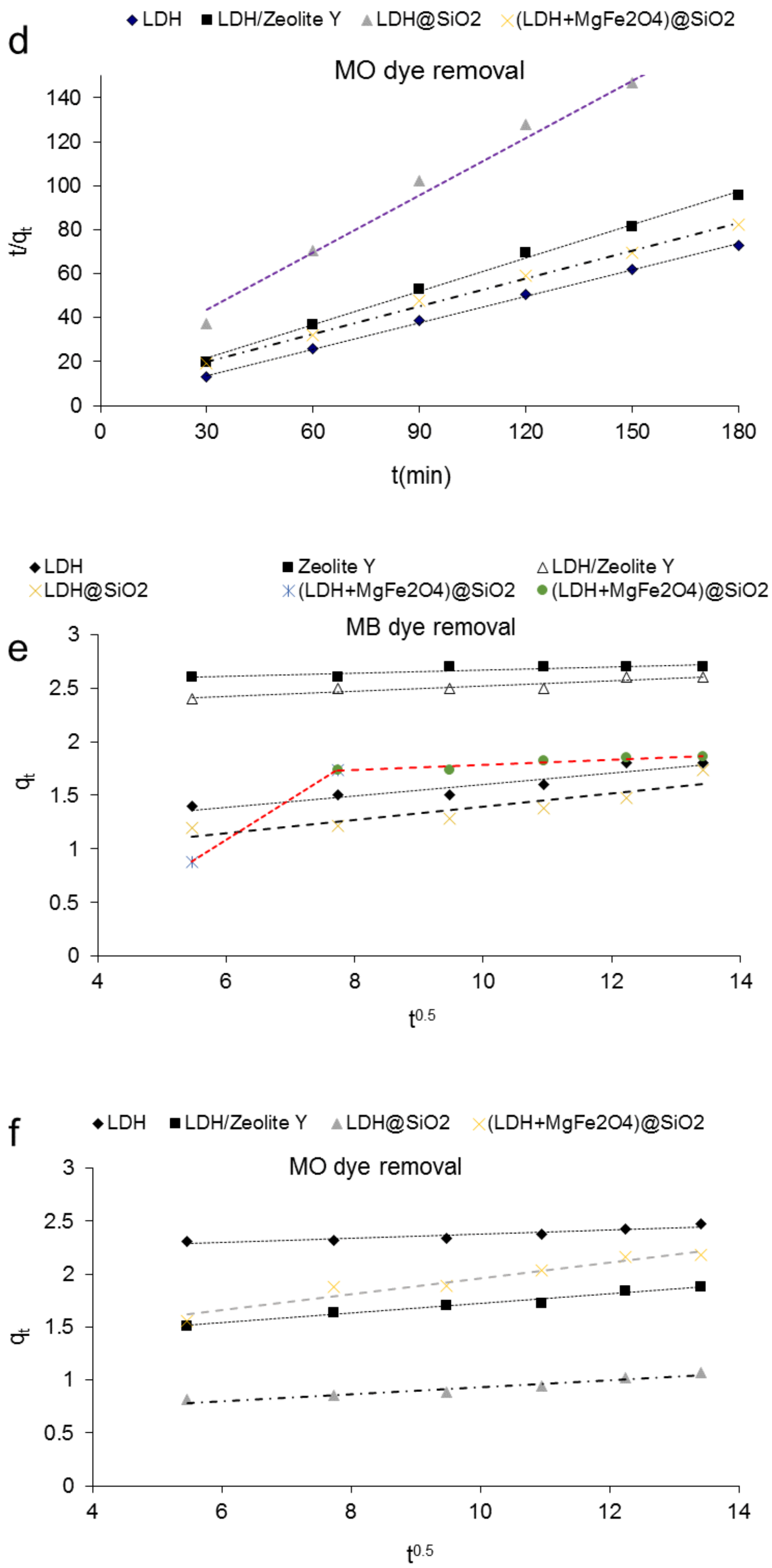


Figure 4. The plots of the pseudo-first-order (a, b), pseudo-second-order (c, d), and intra-particle diffusion (e, f) models for kinetic investigation

Table 1. The pseudo-first-order and pseudo-second-order kinetic parameters for Methylene blue dye removal from dyes mixture solution by MgZnAl-LDH, Zeolite Y, MgZnAl-LDH/Zeolite Y, MgZnAl-LDH@SiO_2 and (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 samples

Sample (For MB dye removal)	q_e^{exp}	Pseudo-first order			Pseudo-second order		
		k_1(min^{-1})	q_{e1}(mg/g)	R^2	k_2(g/mg min)	q_{e2}(mg/g)	R^2
MgZnAl-LDH	1.8	1.8×10^{-3}	1.31	0.9539	2.73×10^{-2}	1.96	0.9878
Zeolite Y	2.7	0.1×10^{-3}	1.01	0.8847	2.080	2.69	1.00
LDH/Zeolite Y	2.6	0.5×10^{-3}	1.08	0.9374	0.100	2.64	0.9999
LDH@SiO_2	1.6	2.4×10^{-3}	1.72	0.9135	1.79×10^{-2}	1.85	0.9507
(LDH+$MgFe_2O_4$)@SiO_2	1.8	3.7×10^{-3}	1.68	0.5049	3.68×10^{-2}	2.00	0.9968

Table 2. the pseudo-first-order and pseudo-second-order kinetic parameters for Methyl orange dye removal from dyes mixture solution by MgZnAl-LDH, MgZnAl-LDH/Zeolite Y, MgZnAl-LDH@SiO_2, and (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 samples

Sample (For MO dye removal	q_e^{exp}	Pseudo-first order			Pseudo-second order		
		k_1(min^{-1})	q_{e1}(mg/g)	R^2	k_2(g/mg min)	q_{e2}(mg/g)	R^2
MgZnAl-LDH	2.4	0.5×10^{-3}	2.16	0.9535	9.09×10^{-2}	2.50	0.999
LDH/Zeolite Y	1.8	1.4×10^{-3}	1.25	0.9654	0.504	1.98	0.9965
LDH@SiO_2	1.0	1.9×10^{-3}	1.38	0.9861	4.42×10^{-2}	1.15	0.9872
(LDH+$MgFe_2O_4$)@SiO_2	2.2	2.0×10^{-3}	2.74	0.8801	2.32×10^{-2}	2.38	0.9968

Conclusion

In summary, the results demonstrated that the Zeolite Y sample was selectively removed 80% of the methylene blue dye after 30 min. The MgZnAl-LDH sample can remove the methylene blue and methyl orange dyes simultaneously. The MgZnAl-LDH sample revealed simultaneous removal of 54% of methylene blue and 74% of methylene orange after 180 min. Moreover, the results showed that the MgZnAl-LDH/Zeolite Y and (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 composites could be used as an adsorbent for the simultaneous removal of cationic and anionic dyes. The kinetic results showed that the pseudo-second-order model provides the best fit to experimental data. The (MgZnAl-LDH+$MgFe_2O_4$)@SiO_2 sample as magnetic hybrid material can be easily separated from the solution. Also, the hybrid material based on natural clay such as Zeolite can significantly reduce the cost of preparation. The simultaneous removal of dye pollutants is important as the mixture of dyes is existence in real effluents.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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