Investigation of Adsorption Kinetics and the Isotherm Mechanism of Manganese by Modified Diatomite

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ABSTRACT: Natural diatomite modified by acetic acid (C-D) and sodium hydroxide (Na-D) for adsorbing manganese (Mn) was studied. The construction and morphology of the modified diatomite were then characterized by different efficient and accurate detection methods (Fourier transform infrared, scanning electron microscopy, and Brunauer−Emmett−Teller), and it was proved that C-D and Na-D increased the surface area and void volume compared to natural diatomite, and the modification of diatomite with acetic acid and alkali also significantly changed the structure of the functional groups of diatomite, especially in Na-D. Therefore, the adsorption rate of Mn by C-D and Na-D was higher than that of natural diatomite. The optimum conditions of Mn adsorption for C-D and Na-D were pH 5.0, 40°C, 30 min and pH 5.0, 40°C, 120 min, respectively, and this was best illustrated by pseudo-second-order kinetics. The Mn adsorption isotherm models showed that Mn adsorption on C-D and Na-D was stable, and the Langmuir adsorption isotherm model fitted adsorption processes of natural diatomite, C-D, and Na-D well as their correlation coefficients were 0.931, 0.940, and 0.991, respectively. These results suggested that modified diatomite with acetic acid and sodium hydroxide significantly increased the adsorption rate of Mn, which had an important application prospect for the remediation of Mn pollution in soil and water.

1. INTRODUCTION

As the material basis of human survival, the protection of land is of great concern. In recent years, industrial, agricultural, and personal activities have polluted many lands, and these activities have generated abundant waste containing large quantities of heavy metals, which can be harmful to health if they are taken overdoses. In China, Hunan Province is rich in mineral resources. Indeed, the Xiangtan Mn Mine is one of the largest mines in China, and with such a large-scale joint production enterprise of mining, beneficiation, sintering, smelting, and electrolysis, Mn pollution is a significant environmental problem in the area surrounding the mine. Zhu et al. demonstrated that the concentration of Mn tailings in the soil has reached 82199.0 mg kg⁻¹, which is 179 times greater than the background value of Hunan Province. Excessive Mn in the soil not only affects the growth of plants but also affects the health of human beings. Therefore, it is necessary to carry out efficient treatment to effectively remove heavy metals from polluted waste waters and soils before toxic metals are released into the environment.

Among various heavy metal remediation methods, the adsorption method is extensively applied in purification for environmental heavy metals because of its low cost and high affinity for heavy metals. As a naturally porous material, diatomite has been widely used for in situ soil remediation because of its adsorption properties. The results of some studies show that diatomite has strong adsorption affinity for heavy metals in soil; diatomite treatments could effectively reduce the leaching content of soil heavy metals, and the application of diatomite would not significantly change the soil structure. Qiu also demonstrated that the application of diatomite had a good curing effect on Cu, Zn, and Cd ions in the soil. Additionally, in order to improve the specific surface area and the number for surface functional groups of diatomite and achieve a goal with more effective adsorption of heavy metals by diatomite, many scholars use different methods to modify diatomite. For instance, Zn₂SiO₄-modified diatomite can achieve a very ideal adsorption effect for Pb and Cd. Furthermore, diatomite respectively modified by...
Mg₃Si₄O₁₀(OH)₂ and MgFe₂O₄ shows the characteristics of larger surface areas and higher adsorption capacities of Cr under ultraviolet treatment; the adsorption rate of Hg by a diatomite-chitosan composite adsorbent reached 77%, and zero-valent iron modified diatomite performance with a high adsorption efficiency of Pb. However, acid and alkali modification methods are effective and inexpensive modification methods, and the adsorption effects of diatomite and its modified reagents on Mn have been rarely studied.

In order to reduce the negative effect of impurities in natural diatomite on heavy metal adsorption, diatomite was modified by acetic acid and sodium hydroxide, respectively, and the Mn adsorption characteristics of modified diatomite were systematically studied. The construction and morphology of the modified diatomite were then characterized by different efficient and accurate detection methods [Fourier transform infrared (FTIR), scanning electron microscopy (SEM), and Brunauer-Emmett-Teller (BET)]. Batch experimental tests were performed to obtain Mn adsorption thermodynamics and adsorption kinetics for the modified diatomite. This study will provide theory foundation and technological innovation of in-site restoration for Mn-polluted soil and water.

### 2. RESULTS AND DISCUSSION

#### 2.1. Results of Diatomite Characterization.

Natural diatomite contained clay, minerals, and other impurities, which would block some pores in diatomite (Figure 1a). SEM revealed differences in the surface morphology of the modified diatomite. The acetic acid modification had no significant impact on the well-developed columnar porous formation of the diatomite. However, the appearance of C-D was cleaner, reflecting that the impurities existing in the natural diatomite pores were effectively cleaned up and the surface area and pore volume of C-D and Na-D increased significantly through acetic acid modification, which could be profitable in the adsorption of Mn. Moreover, the fragmentation degree of Na-D increased, and complete columnar porous structures were rarely observed (Figure 1c); this greatly increased the surface area and pore volume of Na-D.

Surface modifications of the diatomite with acetic acid and alkali were evidenced in the FTIR spectra, which are shown in Figure 2. The bands of the highest intensity of modified diatomite were quite similar to those of natural diatomite, and the characteristic absorption bands at 3384, 2912, 1650, and 1028 cm⁻¹ are respectively due to stretch vibrations of the −OH, C−H, N−H, and C−O groups in diatomite. The FTIR spectrum showed that the alkali modification changed the structure of the functional groups of diatomite, which...
proved that the alkali would react with diatomite to form the Mn adsorption composite when diatomite was modified by alkali. On the contrary, it was obvious from the FTIR spectrum that the acetic acid modification had no notable influence on the chemical properties of the diatomite.

The surface area and pore size of C-D and Na-D were estimated by BET (Table 1). The pore sizes of C-D and Na-D were 0.092 and 0.129 cm$^3$ g$^{-1}$, respectively, which were bigger than that of natural diatomite (0.084 cm$^3$ g$^{-1}$). The surface areas of C-D and Na-D were 23.087 and 31.383 m$^2$ g$^{-1}$, respectively, which were larger than that of natural diatomite (20.505 m$^2$ g$^{-1}$), and the above results were consistent with previous research.\(^\text{11}\) The reason for the larger specific surface area of C-D and Na-D was the removal of impurities on the surface of modified diatomite and the opening of nanopores. This indicated that the modification of the diatomite enhanced the nanostructural features of the diatomite, which was in accordance with the SEM analysis.

### 2.2. Adsorption Experiment Results

#### 2.2.1. Effects of pH

The adsorption capacities of heavy metals were closely related to the pH value, which would cause the change of metal ion morphology and adsorbent surface charge.\(^\text{12}\) As showed in Figure 3, the pH assessment with respect to Mn adsorption was conducted within the range of 2.0—7.0. The adsorption capacity and removal rate of Mn initially increased and then decreased with increase of the pH value. The maximum values of $q_t$ and $R$ in natural diatomite (1.60 mg g$^{-1}$ and 11.96%, respectively) and Na-D (13.27 mg g$^{-1}$ and 99.52%, respectively) were reached when the pH reached 5.0. With respect to C-D, the maximum values of $q_t$ and $R$ (1.06 mg g$^{-1}$ and 8.01%, respectively) were achieved when the pH increased to 3.0. Therefore, the optimal pH values of the Mn adsorption were 3.0 for C-D and 5.0 for natural diatomite and Na-D.

A gradual increase to the optimal pH value could lead to the reduction of $q_t$ and $R$ values for modified diatomite. This increasing adsorption trend was studied previously that within a certain pH range, the adsorption capacity of hydrolyzable metal cations increased sharply with the increase of pH,\(^\text{13}\) which can be explained as follows: at a low pH (< optimal pH value), there was competitive adsorption with diatomite because of the high H$^+$ concentration solution, which resulted in less absorption of Mn, and as the pH increased, the adsorption competition between H$^+$ and Mn weakened, thereby enhancing absorption of Mn. In contrast, at a high pH (> optimal pH value), the surface potential of diatomite was high, which resulted in weak electrostatic adsorption and reduced the electronegativity of the adsorbent when the values of $q_t$ and $R$ of Mn for the adsorbent decreased. On the other hand, with the increase of the pH value (the point of the optimal pH value) and when the solution was slightly acidic, the surface potential of the adsorbent decreased, and the values of $q_t$ and $R$ of Mn for the adsorbent increased slightly.\(^\text{14,15}\)

#### 2.2.2. Effects of Mn Initial Concentrations

We studied the effects of different initial concentrations of Mn between 50 and 600 mg L$^{-1}$ on heavy metal adsorption by modified diatomite (Figure 4). When Mn concentrations of the solution increased from 50 to 600 mg L$^{-1}$ for natural diatomite, the $q_t$ of Mn was enhanced from 1.09 to 2.81 mg g$^{-1}$, and the $R$ decreased slowly from 65.8 to 14.1%. With respect to C-D, the $q_t$ of Mn was enhanced from 1.09 to 2.81 mg g$^{-1}$, and the $R$ decreased from 1.09 to 2.81 mg g$^{-1}$, and the $R$ decreased slowly from 65.8 to 14.1%.
increased from 1.14 to 3.11 mg g⁻¹ and the R decreased from 68.8 to 15.6%. In general, if the adsorption dose was unchanged, the free solute molecules in the solution would increase as the Mn concentration of the solution increases. Hence, heavy metals more easily come into contact with the adsorbent surface, which made more heavy metals to get absorbed by the adsorbent. Moreover, the Mn adsorption efficiency could be improved by multiple adsorption sites of the adsorbent at low Mn concentrations in the solution. When the concentration of the heavy metal reached the threshold, the adsorption sites of the adsorbent were occupied gradually, and the number of vacancies was obviously reduced. Thus, R tended to become gradually balanced. On the other hand, Na-D had an optimal and stable adsorption capacity for Mn compared with that of natural diatomite and C-D, and the attained removal rate was as high as 98.17−99.85% for any concentration of Mn, which could be attributed to the functional group changing after alkali modification.

2.2.3. Effects of Temperature. As observed in Figure 5, a rise in temperature increased the values of q̇ and R of Mn on natural diatomite and C-D. This could be explained by the fact that the temperature rise would be conducive to Mn ionization and accelerated the diffusion of Mn in the solution, thus promoting the binding of Mn with the adsorbent adsorption sites. With the temperature rise from 20 to 40 °C, the values of q̇ and R of Mn on natural diatomite increased from 0.99 to 1.50 mg g⁻¹ and from 3.24 to 5.93%, respectively. Meanwhile, the values of q̇ and R of Mn on C-D increased from 1.48 to 2.47 mg g⁻¹ and from 5.99 to 8.83%, respectively. On the other hand, the change of temperature had little effect on the Mn adsorption by Na-D because of its strong adsorption to Mn.

2.2.4. Effects of Time. By setting different adsorption times, the optimal reaction times of Mn adsorption by natural and modified diatomite were determined to ensure full and efficient completion of the adsorption reaction (Figure 6). The results showed that natural diatomite achieved stable q̇ and R values of 0.78 mg g⁻¹ and 5.90%, respectively, at 60 min, and C-D achieved stable q̇ and R values of 2.68 mg g⁻¹ and 20.20%, respectively, at 120 min. Meanwhile, Na-D attained stable adsorption in 30 min with q̇ and R values of 13.29 mg g⁻¹ and 99.00%, respectively. The results clearly indicated that the adsorption rate of the adsorbent would significantly increase with time at the initial stage. However, with the continuous adsorption of heavy metals on the adsorbent, the adsorption rate of heavy metals by the adsorbent gradually decreased and finally reached equilibrium and tended to be stable. This was because there were many open adsorption points in the adsorbent at the initial stage of adsorption, which would be occupied by heavy metals in a short time, so the adsorption rate went up sharply at this time. However, with the continuous adsorption of the adsorbent, adsorption points on the surface of the adsorbent gradually reduced, so the adsorption capacity of the adsorbent gradually decreased, and the adsorption rate gradually slowed down and finally remained stable.

2.2.5. Adsorption Kinetics. Adsorption kinetics was a key parameter to describe the adsorption rate of heavy metals in solution. In this study, the pseudo-first-order dynamics (eq 1), pseudo-second-order dynamics (eq 2), and intraparticle diffusion (eq 3) models were adopted to discuss the adsorption mechanism of Mn on a diatomaceous earth-based adsorbent. The equations for these models were as follows:

\[ q_i = q_{eq} \left[1 - \exp\left(-K_1t\right)\right] \]  

\[ q_i = \frac{q_{eq}^2K_2t}{1 + q_{eq}K_2t} \]  

\[ q_i = K_p t^{1/2} + c \]

where \( q_i \) is the adsorption capacity by the adsorbent (mg g⁻¹) of time \( t \) (min), \( q_{eq} \) is the maximum adsorption capacity (mg g⁻¹), \( K_1 \) and \( K_2 \) are the rate coefficients of the pseudo-first-order kinetic (min⁻¹) and the pseudo-second-order kinetic (min⁻¹), \( K_p \) is the spreading rate coefficient [mg (g min⁻¹/2)]⁻¹, and \( c \) is the intercept. Figure 7 shows the dynamic trend of \( q_i \) against \( t \) for the pseudo-first-order kinetic model. Figure 8 shows the dynamic trend of \( t/q_i \) against \( t \) for the pseudo-second-order kinetic model. Table 2 shows the kinetic parameters obtained by the pseudo-first-order kinetic and pseudo-second-order kinetic models.

The pseudo-second-order kinetic equation was suitable for calculating the maximum adsorption capacity of Mn at different concentrations, and the calculated value was very close to the experimental value. Besides, the related coefficients \( (R^2) \) of the pseudo-second-order kinetic equation for Mn adsorption by natural diatomite, C-D, and Na-D were 0.999, 0.998, and 0.999, respectively, which were larger than the related coefficients of the pseudo-first-order kinetic equation.
Therefore, kinetics of the Mn adsorption system by natural and modified diatomite corresponded with the pseudo-second-order kinetic equation; the fitting effect of the pseudo-second-order kinetic model for the three materials was Na-D > C-D. It was confirmed that the adsorption type for Mn by natural and modified diatomite was chemical adsorption, which may involve the strong covalent surface interaction between oxygen-containing groups and Mn.20

2.2.6. Adsorption Isotherm. The Langmuir, Freundlich, and Temkin equations were often used as adsorption isotherm models to describe the relationship between the adsorbents and adsorbates in the adsorption process at a certain temperature.21−23 These adsorption isotherm equations were defined as follows:

\[ q_e = q_m \frac{K_L C_e}{1 + K_L C_e} \]  
\[ q_e = K_F C_e^{1/n} \]  
\[ q_e = \frac{R T}{b} \ln K_T + \frac{R T}{b} \ln C_e \]

where \( C_e \) (mg L\(^{-1}\)) is the Mn equilibrium concentration; \( q_e \) (mg g\(^{-1}\)) is the Mn adsorption capacity; \( q_m \) (mg g\(^{-1}\)) is the maximum adsorption capacity of Mn; and \( K_L \), \( K_F \), and \( K_T \) represent the Langmuir, Freundlich, and Temkin adsorption constants, respectively. Additionally, \( n \) is the adsorbed strength, \( R \) is the constant of the gas, \( T \) is the temperature in kelvin (K), and \( b \) is the constant related to the adsorption.

The adsorption isotherm curves for Mn of natural diatomite, C-D, and Na-D are shown in Figure 4, and the related parameters are shown in Table 3. In contrast, from the \( R^2 \) values of different adsorption isotherm models, it was observed that the Langmuir adsorption isotherm model had a good fit with the adsorption characteristics of natural diatomite, C-D, and Na-D. The \( R^2 \) values of natural diatomite, C-D, and Na-D were 0.931, 0.940, and 0.991, respectively. The \( R^2 \) value of Na-D was the largest, reaching 0.991, which indicated that the Langmuir adsorption isotherm model had the best fitting effect on Na-D, and the \( K_L \) values of natural and modified diatomite were less than 1, which indicated that the adsorption reaction between diatomite and Mn was easy to carry out. In addition, for natural diatomite, C-D, and Na-D, the \( R^2 \) values of the Freundlich and Temkin isotherm models were lower than 0.9, which was not suitable for describing the Mn adsorption process (Figure 9).

3. MATERIALS AND METHODS

3.1. Materials. Natural diatomite was attained at the Pengjian Mineral Products Processing Factory (Langshou County, Hebei Province, China). Modified diatomite samples were then prepared. The first sample contained natural diatomite (50 g) mixed with acetic acid (1 mol mL\(^{-1}\) CH\(_3\)COOH; 500 mL; C-D) in a 1000 mL beaker; the second sample contained natural diatomite (50 g) mixed with sodium hydroxide (3 mol L\(^{-1}\) NaOH; 500 mL; Na-D) in a separate 1000 mL beaker. These samples were then heated continuously for 3 h at 80 °C in a thermostatic water bath. Finally, diatomite was repeatedly filtered and washed by ethanol and deionized water. Then, diatomite was dried at 80 °C to a constant weight.

The construction and morphology of modified diatomite were observed by SEM (Zeiss-Supra55, Germany). Surface functional groups of modified diatomite were evaluated using an FTIR spectrometer (Bruker Tensor 27, Germany) in the 4000−400 cm\(^{-1}\) region spectrum. The surface area and pore size of the modified diatomite were calculated by BET (ASAP-...
3.2. Adsorption Experiments. The adsorption thermodynamics and kinetics for Mn adsorption on the modified diatomite were obtained by batch experiments. Mn was dissolved in deionized water to form a standby solution, and different concentrations of Mn test solutions were prepared according to the standby solution. 1.5 g of diatomite was added into 40 mL Mn solutions with different concentrations, which were tested in sealed conical flasks of 100 mL. The sealed conical flasks were put in water bath oscillation with a speed of 150 rpm and 30 °C.

The effects of pH values in the range of 2.0–7.0 upon Mn adsorption characteristics of modified diatomite were discussed under the same initial Mn concentration and temperature (25 °C). The pH values were determined using an acidometer (PB-10). After solid–liquid separation by centrifugation, the residual concentrations of Mn in the supernatant were measured with an atomic absorption spectrophotometer (SOLAAR M6, USA).

The study of Mn adsorption kinetics was conducted under an Mn concentration of 400 mg/L, pH 5.0, and 30 °C. Mn and modified diatomite were blended in 50 mL conical flasks with a solid–liquid proportion of 1:35. All conical flasks were sealed with Tellon screw caps, which were shaken at 150 rpm/min and 30 °C for 30, 60, 90, 120, 150, 180, 480, and 720 min, and about 0.5 mL of the supernatant was extracted from each vial. The supernatants were then added to a 25 mL volumetric flask for Mn analysis.

|                  | Langmuir          | Freundlich       | Temkin           |
|------------------|-------------------|------------------|------------------|
|                  | $q_m$ (mg g⁻¹)    | $K_L$            | $R^2$            | $K_F$           | $R^2$            | $b$              | $K_T$            | $R^2$           |
| diatomite        | 2.803             | 0.10             | 0.931            | 0.269           | 2.803            | 0.827            | 5411.293        | 0.330           | 0.877           |
| C-D              | 3.465             | 0.007            | 0.940            | 0.236           | 2.534            | 0.834            | 4172.441        | 0.260           | 0.865           |
| Na-D             | 20.65             | 0.982            | 0.991            | 8.875           | 2.804            | 0.941            | 622.84          | 7.917           | 0.991           |

Figure 9. Langmuir, Freundlich, and Temkin adsorption isotherm models of natural and modified diatomite.
The adsorption isotherm of modified diatomite for Mn was determined by studying the adsorption effect of modified diatomite on different concentrations of Mn solution, which were set as 50, 100, 200, 300, 400, 500, and 600 mg/L. When the modified diatomite was added with different concentrations of Mn solution, the sealed triangular flasks were shaken at 150 rpm for 3 h. The triangular flasks were set until the solid–liquid was separated, and about 0.5 mL of the supernatant was extracted, which then was added to a 25 mL volumetric flask for a constant volume before Mn analysis.

All batch experiments were run in duplicate, and the control test was designed as a natural diatomite treatment. The adsorption capacity of time \( t (q_t) \) (eq 7) and the removal rate \( R \) (eq 8) of Mn were calculated with the following equations:

\[
q_t = \frac{(c_0 - c_t)V}{w} \tag{7}
\]

and

\[
R = \frac{(c_0 - c_e)}{c_0} \times 100\% \tag{8}
\]

where \( c_0 \) and \( c_t \) are the initial concentration and equilibrium concentration of Mn (mg/L), respectively; \( V \) is the solution volume of Mn (L); and \( w \) is the weight of the modified diatomite (g).

4. CONCLUSIONS

Acetic acid-modified diatomite and alkali-modified diatomite (C-D and Na-D) were successfully synthesized through a simple method. The results showed that C-D and Na-D represented a stronger Mn adsorption than natural diatomite, especially Na-D, and the removal rate of Mn by Na-D can reach 98.17–99.85%, which may be due to the change of the Mn adsorption group of diatomite by alkali modification. The effects of the process parameters, adsorption kinetics, and isotherms of Mn adsorption on Na-D were researched systematically. The results showed that the pseudo-second-order kinetic model could best illustrate the adsorption kinetics process of Na-D. Furthermore, the Langmuir adsorption isotherm model corresponded well with the adsorption isotherm process of Na-D, and the type of Mn adsorption on Na-D was ultimately confirmed to be active and chemical adsorption.

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Notes

The authors declare no competing financial interest.

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