

Adsorption Study for Removal of Mn (II) Ion in Aqueous Solution by Hydrated Ferric (III) Oxides

Somchintana Puttamat and Varong Pavaraajarn

Abstract—In this study, adsorption characteristic of manganese (II) ion onto hydrated ferric (III) oxides (HFOs) nanoparticle resins was analyzed and fitted with adsorption isotherm models such as Freundlich, Langmuir, Temkin and Dubinin–Radushkevich isotherms. The Freundlich isotherm model gave the highest R^2 value of 0.9739. The fitted Freundlich parameter (n) is 6.757, which indicates favorable sorption. The heat of sorption process was estimated from Temkin isotherm model to be $96.749 \text{ J}\cdot\text{mol}^{-1}$ and the mean free energy was estimated from Dubinin–Radushkevich isotherm model to be $288.675 \text{ J}\cdot\text{mol}^{-1}$. The thermodynamic parameters were calculated and it was found that the adsorption process was spontaneous and endothermic and was favored at higher temperature.

Index Terms—Hydrated ferric (III) oxide (HFOs) nanoparticle, adsorption, equilibrium isotherm and manganese (II) ion.

I. INTRODUCTION

Manganese (Mn) is a heavy metal commonly found in soil, sediment and especially in ground water. Not only it has effect on appearance and taste of the water, it also causes health problems on neurological and muscle function in humans. The Department of Environment Services Health Risk Assessment Program has adopted the health based standard of manganese of $0.84 \text{ mg}\cdot\text{L}^{-1}$.

Manganese can present in water in one of three basic forms i.e., dissolved, particulate and colloids, depending on pH of the solution. Nevertheless, the most common treatment techniques of manganese contaminated water is oxidation/filtration and adsorbing onto ion exchange resin [1]-[3].

Oxidation of the dissolved manganese creates particles of manganese dioxide (MnO_2). The oxidation treatment is more effective in removing high concentration of dissolved manganese than the ion exchange. However, dissolved manganese is generally oxidized so slowly that it takes relatively long time for precipitation, hence it hardly becomes cost effective. The oxidation with chlorine becomes more effective when the pH is greater than 9.5 [4], [5].

Ion exchange technique employing sodium chloride solution can remove low concentration of dissolved manganese when the pH is greater than 6.8. Nevertheless, a simple ion exchange process is only driven by electrostatic interaction and it is nonspecific for heavy metal removal. This

gives rise to the frequent regeneration and high operation cost, rendering ion exchange technology unattractive. The use of chemically stable polymeric chelating exchanger has resolved the problem technically, but it is often too expensive to justify the applications for heavy metals removal from water and wastewater [1]-[7].

The major aim of the present study is to explore the adsorption behavior of Mn^{2+} in single ion system. In particular, we evaluated the equilibrium adsorption data by adsorption isotherms i.e., Freundlich, Langmuir, Temkin and Dubinin–Radushkevich. Also, we intend to evaluate the use of HFOs-resin in removing Mn^{2+} from simulated wastewater.

II. EXPERIMENTAL METHOD

A. Preparation of the HFOs-Resin

Precipitated hydrated Fe(III) oxides or HFOs nanoparticle were prepared by using Kastel[®] C300L (Siam Machinery Thai Co., Ltd.), which is a commercially available macroporous cation exchange resin with polystyrene matrix and sulfonic acid functional group, as the parent cation exchanger. Size of the exchanger resin beads varied between 450 to 550 μm . The preparation of HFOs-resin was consisted of the following three steps [8]. Firstly, Fe(III) was loaded onto the sulfonic acid sites of the cation exchanged by passing 4% FeCl_3 (AR grade, Merck KGaA) solution at an approximate pH of 2.0 through a column packed with the resin. Secondly, desorption of Fe(III) and simultaneous precipitation of Fe(III) hydroxides took place within the gel and pore phase of the exchanger through passage of a solution containing both NaCl (AR grade, Merck KGaA) and NaOH (AR grade, Merck KGaA) solution, each at 5% w/v concentration, through the column. Finally, the column was rinsed and washed with a 50% ethanol (AR grade, Merck KGaA), aqueous solution followed by a mild thermal treatment (333 K) for 6 h under N_2 atmosphere. The obtained HFOs-resin was then kept in vacuum desiccator.

B. The Determination of Bulk Density

The bulk density of each of the sample of Kastel[®] cation exchange resins and HFOs-resin was determined using Archimedes's principle [9] by weighing 10 mL measuring cylinder before and after filling with the samples. The measuring cylinder was then dried and the sample was packed inside, leveled and weighed. The weight of the sample packed in the measuring cylinder was determined from the difference in weight of the filled and empty measuring cylinder. The volume of water in the container was determined by taking the difference in weight of the empty and water filled measuring cylinder. The bulk density was determined using the equation below.

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$$\text{bulk density} = \frac{W_2 - W_1}{v} \quad (1)$$

where:

w_1 = weight of empty measuring cylinder.

w_2 = weight of cylinder filled with sample.

v = volume of cylinder.

C. Effect of the Initial Concentration of Adsorption Isotherm

The equilibrium sorption of the Mn^{2+} (from 4% HNO_3 solution, ICP-OES & ICP-MS standard, SCP Science) was carried out by contacting 5.0 g of the HFO-resins with 250 mL of the solution in various concentrations in the range from 10–200 $mg \cdot L^{-1}$ in 500 mL Duran conical flasks intermittently for 120 minutes on orbital shaker. The mixture was filtered and analyzed using Optimal Emission Spectrometer with inductively coupled plasma (ICP-OES; Perkin Elmer, Optima 8000). The amount of adsorbed ($mg \cdot g^{-1}$) was calculated using the formulae reported by Vanderborgh and Van Griekem [9]:

$$q_e = \frac{v(C_0 - C_e)}{w} \quad (2)$$

where:

q_e = the amount of solute adsorbed from the solution.

v = volume of the adsorbate.

C_0 = the concentration before adsorption.

C_e = the concentration after adsorption.

w = the weight in gram of the adsorbent.

The data was fitted into following isotherms: Freundlich, Langmuir, Temkin and Dubinin–Radushkevich. The removal efficiency was determined by computing the percentage sorption using the formulae in (3).

$$\text{Adsorption\%} = \frac{C_0 - C_e}{C_0} \times 100 \quad (3)$$

D. The Effect of Solution pH

The effect of pH on the removal of Mn^{2+} using HFOs-resin was studied by varying the pH of the metal ion solution from 1 to 10. The solution pH was adjusted by phosphate buffer and 0.1 $mol \cdot L^{-1}$ HCl or NaCl solutions. A mass of 0.05 g of adsorbent was added to 10 mL of the solution containing the metal ions with an initial concentration of 200 $mg \cdot L^{-1}$ and was stirred with 150 rpm agitation for 7 h. The temperature of the solution was kept constant at 303 K [1]. The residual concentration of the ion was determined as explained earlier.

E. The Effect of Adsorption Kinetics

A set of experiments was conducted for studies of adsorption kinetics by placing 0.05 g adsorbent into 10 mL of solution containing Mn^{2+} with an initial concentration of 200 $mg \cdot L^{-1}$. The experiment was performed at pH = 2.0 and temperature of 303 K. The concentration of the metal ion was periodically measure within 10 – 120 min of the experiment [10].

F. The Effect of Adsorption Thermodynamic

The effect of temperature on the adsorption of Mn^{2+} was investigated in order to calculate the thermodynamic parameter; 0.05 g of adsorbent was mixed with 10 mL of 200

$mg \cdot L^{-1}$ metal ion solutions at a range of temperature from 303–333 K [11].

III. RESULTS AND DISCUSSION

A. Characterization of HFOs-resin

The hybrid sorbent HFOs-resin retained the spherical geometry and developed a deep brown color after being loaded with ferric oxide. The content of HFO loaded within the cation exchange resin was about 12.3% in Fe mass (Table D).

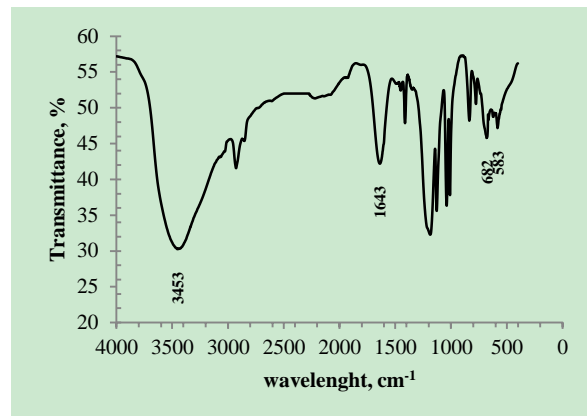


Fig. 1. FTIR chromatogram for HFOs-resin.

The FTIR spectra of HFO showed two major adsorption peaks (Fig. 1). The peak at 3453 cm^{-1} is for the stretching vibration of lattice water and hydroxyl group, while that located at 1643 cm^{-1} is for -OH bending vibration of adsorbed water molecules. The spectra also showed significant band of metal at 682 and 583 cm^{-1} for Fe^{3+} adsorption.

TABLE I: SOME PROPERTIES OF THE CATION EXCHANGER (KASTEL® C300L) AND HFOs-RESIN

	Sorbent	
	Kastel® C300L	HFOs-resin
Matrix structure	Polystyrene	Polystyrene
Functional groups	R-SO ₃ Na	R-SO ₃ Na; HFO
BET surface area ($m^2 \cdot mg^{-1}$)	22.7	29.4
Average pore diameter (nm)	30.8	21.7
Apparent density ($g \cdot m \cdot cm^{-3}$)	0.46	0.62
HFO content (Fe mass%)	0	12.3

B. Adsorption Isotherms of Mn (II) onto HFOs-Resins

The equilibrium adsorption of the Mn^{2+} was carried out as mentioned earlier. The mixture was sampled every 5 minutes to be analyzed for metal ion concentration using ICP-OES. The data was fitted into following isotherms: Freundlich, Langmuir, Temkin and Dubinin–Radushkevich.

1) Freundlich adsorption isotherm is commonly used to describe the adsorption characteristics for the heterogeneous surface. These data is often fitted with the following empirical equation proposed by Freundlich [10]:

$$q_e = K_f C_e^{1/n} \quad (4)$$

where:

K_f = Freundlich isotherm constant ($mg \cdot g^{-1}$).

n = adsorption intensity.

C_e = the equilibrium concentration of adsorbate ($mg \cdot L^{-1}$)

q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium ($mg \cdot g^{-1}$).

Linearized form of the equation (4) can be written as:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

The constant K_f is an approximate indicator of adsorption capacity, while $1/n$ is a function of the strength of adsorption in the adsorption process. If $n = 1$, then the partition between the two phases are independent of the concentration. If value of $1/n$ is below one, it indicates a normal adsorption. On the other hand, $1/n$ being above one indicates cooperative adsorption. The function has an asymptotic maximum as the concentration is increased. As the temperature is increased, the constants K_f and n change to reflect the empirical observation that the quantity adsorbed rises more slowly and higher concentration is required to saturate the surface. However, K_f and n are characteristic parameters of the sorbent-sorbate system, which must be determined by data fitting. Linear regression is generally used to determine the parameters of kinetic and isotherm models. Specifically, the linear least-square method and the linearly transformed equation have been widely applied to correlate sorption data where $1/n$ is heterogeneity parameter. The smaller $1/n$ becomes, the greater the expected heterogeneity is. This expression can be reduced to a linear adsorption isotherm when $1/n = 1$. If n lies between one and ten, this indicates a favorable sorption process [9-10]. The value of $1/n = 0.148$ while $n = 6.757$ indicating that the sorption of Mn^{2+} onto HFO-resin is favorable and the R^2 value is 0.9739 proving that the sorption data is fitted well to Freundlich isotherm model.

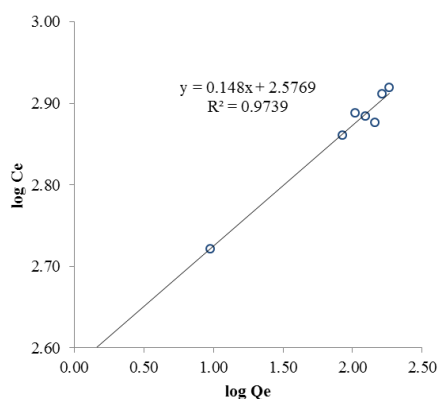


Fig. 2. Freundlich adsorption isotherm.

2) Langmuir adsorption isotherm model describes quantitatively the formation of a monolayer adsorbate on the outer surface of the adsorbent, after which no further adsorption takes place. Thereby, the Langmuir represents the equilibrium distribution of metal ions between the solid and liquid phases. The Langmuir isotherm is valid for monolayer adsorption onto a surface containing a finite number of identical sites. The model assumes uniform energy of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface. Based upon these assumptions, Langmuir represented the following equation [10]:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (6)$$

Langmuir adsorption parameters can be determined by

transforming the Langmuir (6) into linear form.

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L C_e} \quad (7)$$

where:

- C_e = the equilibrium concentration of adsorbate ($mg \cdot L^{-1}$).
- q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium ($mg \cdot g^{-1}$).
- q_m = maximum monolayer coverage capacity ($mg \cdot g^{-1}$).
- K_L = Langmuir isotherm constant ($L \cdot mg^{-1}$).

The value of q_{max} and K_L were computed from the slope and intercept of the Langmuir plot of $1/q_e$ versus $1/C_e$. The essential features of the Langmuir isotherm may be expressed in terms of equilibrium parameter R_L , which is a dimensionless constant referred to as separation factor or equilibrium parameter.

$$R_L = \frac{1}{1 + (1 + K_L C_0)} \quad (8)$$

where:

- C_0 = initial concentration.
- K_L = the constant related to the energy of adsorption (Langmuir Constant).

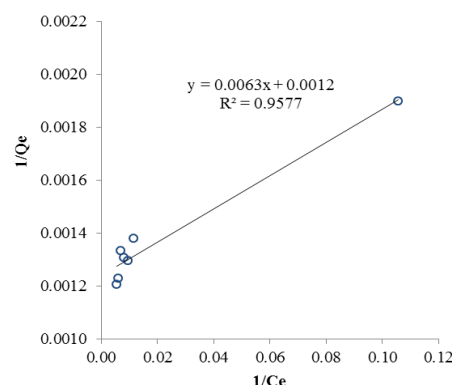


Fig. 3. Langmuir adsorption isotherm.

R_L value indicates the adsorption nature to be either unfavorable if $R_L > 1$, linear if $R_L = 1$, favorable if $0 < R_L < 1$ and irreversible if $R_L = 0$ [10]. From the result, R_L is greater than 0 but it is less than 1 indicating that the Langmuir isotherm is favorable. The maximum monolayer coverage capacity (q_m) from the Langmuir isotherm model was determined to be $830.50 \text{ mg} \cdot g^{-1}$, K_L (Langmuir isotherm constant) is $0.0015 \text{ L} \cdot mg^{-1}$, R_L (the separation factor) is 0.871 indicating that the equilibrium sorption was favorable and the R^2 value is 0.9577.

3) Temkin adsorption isotherm model contains a factor that explicitly taking into the account of adsorbent-adsorbate interactions. By ignoring the extremely low and large value of concentrations, the model assumes that heat of adsorption (function of temperature) of all molecules in the layer would decrease linearly rather than in logarithmic manner with the coverage. As implied in the equation, its derivation is characterized by a uniform distribution of binding energy (up to some maximum binding energy). The fitting was carried out by plotting the quantity sorbed q_e against $\ln C_e$. The constants were determined from the slope and the intercept. The model is given by the following equation [1]:

$$q_e = \frac{RT}{b_T} \ln(A_T C_e) \tag{9}$$

$$q_e = \frac{RT}{b_T} \ln A_T + \left(\frac{RT}{b_T}\right) \ln C_e \tag{10}$$

$$B = \frac{RT}{b_T} \tag{11}$$

$$q_e = B \ln A_T + B \ln C_e \tag{12}$$

where:

- A_T = Temkin isotherm equilibrium binding constant ($L \cdot g^{-1}$)
- b_T = Temkin isotherm constant
- R = Universal gas constant ($8.314 J \cdot mol^{-1} \cdot K^{-1}$)
- T = Temperature at 298 K.
- B = Constant related to heat of sorption ($J \cdot mol^{-1}$)

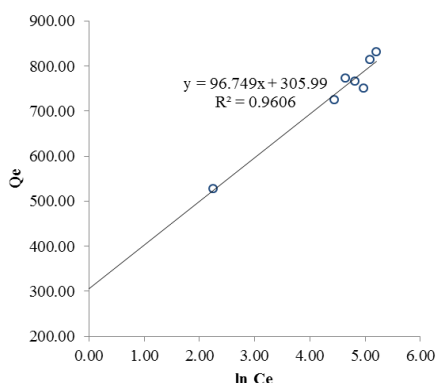


Fig. 4. Temkin adsorption isotherm.

From the Temkin plot shown in fig. 4, the following values were estimated: $A_T = 0.484 L \cdot g^{-1}$, $B = 96.749 J \cdot mol^{-1}$ which is an indication of a physical adsorption and the $R^2 = 0.9606$.

4) Dubinin–Radushkevich isotherm model is generally applied to express the adsorption mechanism with a Gaussian energy distribution onto a heterogeneous surface. The model is often successfully fitted to data with high solute activities and the intermediate range of concentration [9].

$$q_e = (q_m) \exp(-K_{ad} \varepsilon^2) \tag{13}$$

$$\ln q_e = \ln(q_m) - (K_{ad} \varepsilon^2) \tag{14}$$

where:

- q_e = amount of adsorbate in the adsorbent at equilibrium ($mg \cdot g^{-1}$).
- q_m = theoretical isotherm saturation capacity ($mg \cdot g^{-1}$).
- K_{ad} = Dubinin-Radushkevich isotherm constant ($mol^2 \cdot kJ^{-2}$).
- ε = Dubinin-Radushkevich isotherm constant.

The approach was usually applied to distinguish the physical and chemical adsorption of metal ions with its mean free energy, E , per molecule of adsorbent (for removing a molecule from its location in the sorption space to the infinity), which can be computed by the relationship:

$$E = \left[\frac{1}{\sqrt{2B_{DR}}} \right] \tag{15}$$

where B_{DR} is denoted as the isotherm constant. Meanwhile,

the parameter ε can be calculated as:

$$\varepsilon = RT \ln \left[1 + \frac{1}{C_e} \right] \tag{16}$$

where :

- R = the gas constant ($8.314 J \cdot mol^{-1} \cdot K^{-1}$).
- T = absolute temperature (K).
- C_e = adsorbate equilibrium concentration ($mg \cdot L^{-1}$)

One of the unique features of the Dubinin-Radushkevich isotherm model lies on the fact that it is temperature-dependent. When adsorption data from different temperatures are plotted as a function of logarithm of amount adsorbed ($\ln q_e$) and ε^2 , which is the square of potential energy, all suitable data will lie on the same curve, called the characteristic curve. The equation (13) can be linearized to equation (14) which is used in the plot of DRK graph in fig. 5. The constant such as q_m and K_{ad} were determined from the appropriate plot using equation (14). From the linear plot of DRK model, q_m was determined to be $53.774 mg \cdot g^{-1}$, the mean free energy, $E = 288.675 J \cdot mol^{-1}$ indicating a physio-sorption process and the $R^2 = 0.9141$ was lower than of that of the Temkin model.

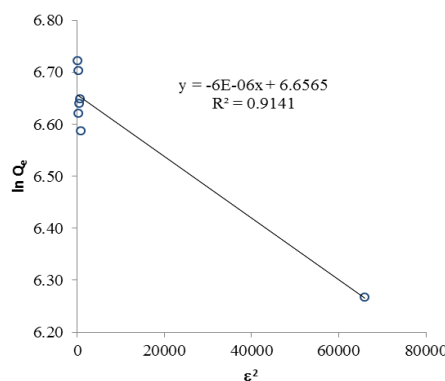


Fig. 5. Dubinin – Radushkevich adsorption isotherm.

C. The Effect of Solution

The Mn (II) ion uptake onto the HFOs-resin were extremely dependent on the initial pH of the solution because the pH affects the surface charge of the adsorbent, the degree of ionization of the surface groups and the nature of the adsorbing ions [2]. The results showed that the adsorption capacity of metal ions was low at low pH, which is attributed to the fact that the high concentration and high mobility of H^+ ions in the solution favors its adsorption onto the surface rather than the adsorption of Mn^{2+} . The adsorption capacity of the HFOs-resin increases with increasing pH from 2 to 6 for the metal ions. The adsorption experiments could not be performed at $pH > 6$ because Mn (II) ion begin to precipitate as $Mn(OH)_2$. Hence pH of 6 was used as the optimum pH throughout this study.

D. The Effect of Initial Metal Ion Concentration

The increase in initial Mn (II) ion concentration decreased the percentage adsorption from 97.39% to 91.41%, which could be due to the limited available adsorption sites. No significant change in adsorption behavior of the HFOs-resin was observed when ion concentration was changed.

E. The Effect of Adsorption Kinetic

Adsorption is a time dependent process. In this section, our main objective is to study the effect of pre-equilibrium contact time in order to better comprehend the adsorption kinetics of heavy metal ions onto the HFOs-resin. It was found that the amount of adsorbed ions increased rapidly in the first 25 min, which indicates that there were enough adsorption sites for the ions to be accommodated. Then, the process slows down as the adsorption sites became gradually saturated. The adsorption equilibrium was reached at about 120 min. Also, the adsorption of metal ions increased with agitation time, perhaps due to a decrease in mass transfer resistance in the bulk solution and an increase in the kinetic energy of the hydrated ions. The kinetics of adsorption is one of the most important aspects defining the efficiency of adsorption and also provides important information about of the rate at which the pollutant is removed from the wastewater for better design batch adsorption systems [7]. In this study, the experimental data were analyzed using a number of kinetic models in order to investigate the mechanism of heavy metal ion adsorption onto the adsorbent. The potential rate-controlling steps are transport of the ion to the surface and surface adsorption itself.

F. The Effect of Temperature

In order to investigate the effect of temperature on the adsorption capacity of Mn (II) onto the HFOs-resin, it is observed that adsorption capacity increases with increasing temperature in the range 303–333 K, demonstrating that the process of adsorption is endothermic. This increase can be due to the increased mobility of metal ions and to their tendency to adsorb from the solution to the surface of the adsorbent as well as due to a greater activity of binding sites as the temperature is increased [10].

IV. CONCLUSIONS

In this paper, HFOs-resin were prepared by using Kastel® C300L resin as the starting material and applied as an adsorbent. Adsorption experiments have shown that the HFOs-resin has high adsorption capacity and can be used as a sorbent for the removal of Mn (II) ion in aqueous solution. The maximum adsorption capacity of the HFOs-resin was 111.925 mg·g⁻¹. The pseudo-second order kinetic model was applied to the experimental data, confirming that sorption was chemically controlled and followed the intra-particle diffusion model. However, intra particle diffusion was not the sole rate-controlling step. The isotherm adsorption data were well fitted by the Freundlich model. It gave the highest R² value, the sorption intensity which indicates favorable sorption and the correlation value were 6.757 and 0.9739 respectively. Also from Langmuir isotherm model, the maximum monolayer coverage was determined to be 830.50 mg·g⁻¹, the separation factor indicating a favorable sorption experiment 0.871. The heat of sorption process was estimated from Temkin isotherm model to be 96.749 J·mol⁻¹ and the mean free energy was estimates from Dubinin-Radushkevich isotherm model to be 288.675 J·mol⁻¹. Based on the thermodynamic parameters (ΔG° , ΔH° and ΔS°), it was found that the adsorption process was spontaneous and

endothermic over the temperature range considered.

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