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Adsorption kinetics and isotherms of ammonia-nitrogen on steel slag

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ABSTRACT

We found that steel slag, the residue of steel manufacturing, had high NH_4^+-N absorptive capacity. Thus, it has the potential to be a high-efficiency and low-cost adsorbent for NH_4^+-N removal from pollutant water. We investigated the kinetics and isotherms of the NH_4^+-N adsorption by steel slag. Experimental results showed that NH_4^+-N absorptive capacity of steel slag was directly related to NH_4^+-N concentration, shaking rate, and particle size of steel slag. When $NH_4^+\text{-N} \leq 50$ mg/L or the shaking rate ≤ 150 rpm, external diffusion of NH_4^+-N could be promoted. When the particle size of steel slag $\geq$ 3–5 mm, intra-particle diffusion of NH_4^+-N became controlling step. Boyd and Kannan equations illustrated that both of intra-particle and external diffusions were key influence factors of NH_4^+-N adsorption, but only one played key role in some particular condition. The adsorption followed pseudo-second-order kinetics. Langmuir and Freundlich models obeyed to fit well with the experimental equilibrium data, and the maximal adsorption capacity was found to be 0.84 mg NH_4^+-N/g steel slag, and Freundlich models showed the adsorption is preferential type.

Keywords: Steel slag; NH_4^+-N; Diffusion; Adsorption; Isotherms; Kinetics

1. Introduction

Water quality deterioration and eutrophication of rivers in China have attracted more and more attention in the last few decades. It is often caused by ineligible injection–effluent from municipal, industrial, and agriculture sources which contain relatively high concentrations of nitrogen pollutant [1]. Ammonia-nitrogen (NH_4^+-N), one of the most important nitrogen pollutants, is more harmful [2]. It can directly poison humans and upset the equilibrium of water systems. It has also created problems of taste and odor in water, has become toxic to fish, and has led to oxygen depletion and eutrophication of surface waters. Therefore, NH_4^+-N has become one of the most important controlling indexes of water quality in China. A 10% discharge reduction of NH_4^+-N has been set as a target for next five years by government [3]. To look for reliable and economically viable methods to remove NH_4^+-N from contaminated surface water is of great importance.

The traditional methods for NH_4^+-N removal include chemical precipitation [4], steam-stripping [5], catalyzes oxidation [6,7], membrane separation [8],

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biological systems [9], and adsorption [10]. However, the chemical precipitation methods add additional reagents that may result in potential pollutants in the water [4]. The steam-stripping method requires a large stripping tower which consumes lots of energy, and generates the incrustation and effluent with high NH_4^+-N concentration [5]. The catalyze oxidation method, operated at high temperatures (> 400℃) and pressures (> 20 MPa), is only suitable for the treatment of water with high concentration of NH_4^+-N [7]. The membrane separation method used to be costly and difficult to control [8]. On the other hand, the biological treatment is ideal for the removal of low concentration of NH_4^+-N, but it may produce a mass of sludge [9]. The adsorption method plays a vital role in many areas of practical environmental technology, which are mainly applied in the wastewater treatment due to several advantages such as high-efficiency, simple operation, and easy recovery/reuse of adsorbent [10]. Zeolite is usually used as the adsorbent to treat low NH_4^+-N concentration and the NH_4^+-N removal efficiency is as high as 95% [11]. Thus, adsorption method is a cost-effective technique for the removal of low concentrations of NH_4^+-N in the polluted rivers.

Steel slag, a residual waste product or by-product of steel manufacture, is a complex mixture of alkali-earth silicates and aluminosilicates formed at high temperature. It is produced in steelmaking process with the ratio of 15–20% crude steel production. At present, it has usually been used as fill material in road and hydraulic construction, as ballast under the railway tracks, or as raw material for phosphoric fertilizer. There was nearly 696 million tonnes of steel slag generated in China in 2010 [12]. However, the amount of the slag production is much more than that of the consumption. This situation results in the growth of slag piles, which led to the severe environment pollution as well as the waste of land. In recent years, several attempts have been made to use steel slag as adsorbents to replace those high-cost materials, such as activated carbons. In water treatment, the steel slag has been used to remove different types of pollutants from water, such as phosphorus, dye, and As (V) etc. [13–15]. According to the early lab testing by Cheng [16], the steel slag could be used to adsorb NH_4^+-N in contaminated water. However, there are no thorough studies aiming to investigate the mechanism of the NH_4^+-N adsorption process by steel slag. Considering the potential applicability of this technology in environmental industries, it is in need to perform a systematic study of NH_4^+-N adsorption by steel slag.

The main objective of this study was to investigate the adsorption kinetics and isotherms of NH_4^+-N on steel slag. We investigated four impact factors of the adsorption process: the initial NH_4^+-N concentration, contact time, the shaking rate of adsorption, and particle size of steel slag. The experimental results were analyzed using various models: external diffusion, intra-particle diffusion, and pseudo-secondorder. We also discussed the parameters to affect the isotherms of the adsorption process. This study not only generates useful data of NH_4^+-N adsorption by steel slag, but also provides solid evidence of steel slag being a potential low-cost adsorbent for NH_4^+-N removal in pollutant water.

2. Materials and experimental methods

2.1. Materials

Steel slag used in this study was obtained from converter of Taiyuan Iron and Steel Co. in Shanxi province, China. And its chemical composition from analysis is presented in Table 1. The slag was mainly comprised of Ca, Si, Fe, and a few percentage of Mg, Al, and Mn. And its phase composition was mainly composed of Ca_2SiO_4, $Ca_3Si_2O_7$, $Ca_7Mg(SiO_4)_4$, $Ca_2Fe_{22}O_{33}$, $MgFeAlO_4$, and $Ca_2(AlFe)_2O_5$ as shown in the X-ray diffractometer (XRD) patterns (Fig. 1). The received slag was in the form of cm-sized chunks. It was first gently crushed in an iron mortar and pestle to mm-sized, then hand-sieved to constrain different particle size ranges, and washed with distilled water to remove surface dust and soluble ions. Later on, the sample was air dried naturally, and used for the subsequent experiments. These samples were stored in a desiccator. The NH_4^+-N solution for the experiments was prepared by dissolving NH_4Cl in distilled water.

2.2. Adsorption experiments

A series of experiments were conducted to investigate NH_4^+-N adsorption process on steel slag. The experimental procedure is described in detail as follow.

2.2.1. Effect of contact time and initial NH_4^+-N concentration

At room temperature of 25℃, 3.0 g steel slag was added to a 250-mL conical flask with 150 mL of NH_4^+-N solution at desired concentration. The steel slag was 3–5 mm in size. The shaking rate was set to 150 rpm. The initial NH_4^+-N concentration studied is in the range of 5–100 mg/L, respectively. A series of samples were taken for NH_4^+-N analysis at preselected time intervals over a range of 360 min.

Table 1
Main composition of the steel slag

Ingredient	CaO	SiO_2	FeO	MgO	Fe_2O_3	Al_2O_3	MnO	P_2O_5
Contents (%)	47.8	14.22	13.29	9.29	8.79	2.86	1.06	0.56

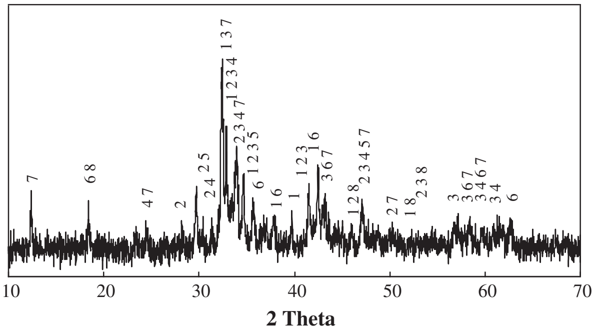

Fig. 1. XRD patterns for steel slag. XRD patterns of steel slag were recorded by an XRD (D/MAX2500PC, Tokyo Rigaku Co.) using Cu Kα radiation. The accelerate voltage was 40 kV, the electrical current was 100 mA and scan ranges from 10° to 80° at the speed of 4°/min.
Note: (1) Ca_2SiO_4 (calcium silicate). (2) $Ca_3Si_2O_7$ (rankinite). (3) Ca_3SiO_5 (calcium silicate oxide). (4) $Ca_7Mg(SiO_4)_4$ (calcium magnesium silicate). (5) $Ca_2Fe_{22}O_{33}$ (calcium iron oxide). (6) $MgFeAlO_4$ (magnesium aluminum iron oxide). (7) $Ca_2(AlFe)_2O_5$ (calcium aluminum iron oxide). (8) Ca(OH).

2.2.2. *Effect of shaking rate*

The effect of shaking rate on NH_4^+-N adsorption by steel slag was investigated in six different shaking rates at 25℃. Each flask (250 mL) contained 3.0 g prepared steel slag with particle size of 3–5 mm and 150 mL NH_4^+-N solution having a concentration of 10 mg/L. The conical flasks were shaken at 50, 100, 150, 180, 210, and 240 rpm on a rotary shaker and the samples were taken for analysis at preselected time intervals over a range of 120 min.

2.2.3. *Effect of particle size*

The effect of particle size on NH_4^+-N adsorption by steel slag was investigated using six different particle size fractions at 25℃. Each flask (250 mL) contained 3.0 g prepared steel slag with 150 mL NH_4^+-N solution (10 mg/L). The particle size fraction studied was 1–3, 3–5, 5–7, 7–9, 9–11, and 11–13 mm, respectively. The conical flask was shaken at 150 rpm rate on a rotary shaker and a series of samples were taken for analysis at preselected time intervals over a range of 120 min.

2.3. *Analytical methods*

Chemical compositions of the steel slag were analyzed by X-ray fluorescence (72000S, ARL, Switzerland). XRD patterns of the prepared samples were recorded by an XRD (D/MAX2500PC, Rigaku) using Cu Kα radiation. The specific surface areas of the slag samples were determined by a N_2-BET method using a Beckman Coulter SA3100-type instrument. The morphology of the samples were observed by scanning electron microscope (SEM) (JSM-6510L, JEOL). UV-visible spectrophotometer (UV-2100, Shinco, China) was used to determine NH_4^+-N concentrations [17]. All adsorption experiments were conducted in triplicates and the data were presented with the average value.

3. Result and discussion

McKay [18] proposed a three-step model for adsorption. The adsorption rate is known to be controlled by factors as following: (i) diffusion of the solute from the solution to the particle surface (external diffusion), (ii) diffusion from the surface to the internal sites (intra-particle diffusion), and (iii) uptake which can involve several mechanisms such as physical–chemical adsorption, ion-exchange, precipitation or complexation. Since the late adsorption step is very rapid, it is assumed that it does not influence the overall kinetics [10]. Therefore, the overall rate of adsorption process will be controlled by either external diffusion or intra-particle diffusion. Boyd equation and Kannan equation are widely used to simulate the external diffusion and intra-particle diffusion, respectively [19]. In this study, effects of contact time, initial NH_4^+-N concentration, shaking rate, and particle size on NH_4^+-N adsorption were evaluated systemically using Boyd equation and Kannan equation.

3.1. *Effect of contact time and initial NH_4^+-N concentration*

The effect of initial NH_4^+-N concentration and contact time on adsorption amount of ammonia-nitrogen (*Q*) by steel slag is shown in Fig. 2. The results show that NH_4^+-N uptake is relatively rapid for the first 30 min and thereafter it proceeded at a slower rate and finally reached equilibrium at almost 120 min within the experimental concentration range.

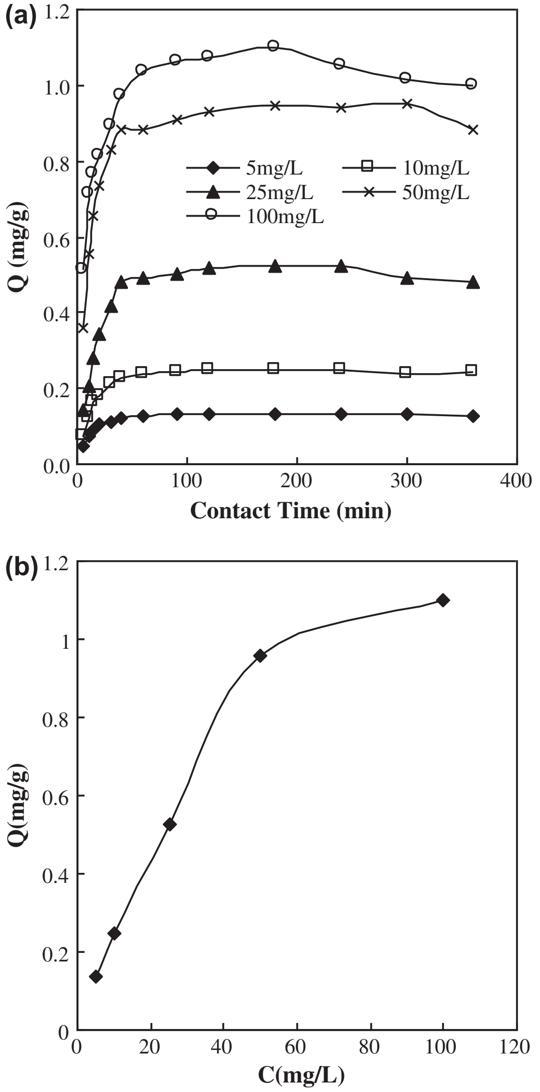

Fig. 2. Effect of contact time on the adsorption amount of NH_4^+-N at different initial concentration of NH_4^+-N. Added 3 g steel slag (particle size: 3–5 mm) into 150 mL NH_4^+-N solution; changed NH_4^+-N concentration (5, 10, 25, 50, and 100 mg/L); shaken for 360 min at 150 rpm to ensure equilibrium at 25℃.

The initial rapid phase may be due to the large number of the vacant sites in the period, and NH_4^+-N concentration gradient between the solution and the adsorbent [20]. Thereafter, a slower adsorption would follow as the available adsorption site gradually decreased. In addition, steel slag mainly consists of silicates, which can ionize in solution, making the surface of steel slag negative charged. Therefore, NH_4^+-N ions would be adsorbed by steel slag through electrostatic attraction. The adsorption amount of NH_4^+-N increased from 0.24 to 1.00 mg/g with the increase of initial NH_4^+-N concentration from 10 to 50 mg/L and the increase of adsorption amount was not significant when further increased the initial concentration from 50 to 100 mg/L. As described by McKay [18], external diffusion is bulk diffusion. In other words, the rate of external diffusion would be affected by initial NH_4^+-N concentration and shaking rate. Therefore, from this experiment, it can be concluded that the higher the initial concentration was, the higher the concentration gradient was, and the faster the NH_4^+-N transferred from liquid to the surface of steel slag. When initial NH_4^+-N concentrations were lower than 50 mg/L, and the adsorption rate was increased with the increasing in initial NH_4^+-N concentration, the adsorption process was controlled by external diffusion. At higher initial NH_4^+-N concentrations (higher than 50 mg/L), the adsorption rate change is not significant, and the resistance of external diffusion was eliminated.

3.2. Effect of shaking rate

The results from shaking rate experiments are shown in Fig. 3. It shows that the adsorption amount of NH_4^+-N (Q) increased with the increase of shaking rate in range of 30–150 rpm. The results implied that the diffusion coefficient of NH_4^+-N in the liquid bulk was increased with the increase in shaking rate. Shaking rate is an important influenced factor during external diffusion. Therefore, it is possible that the adsorption

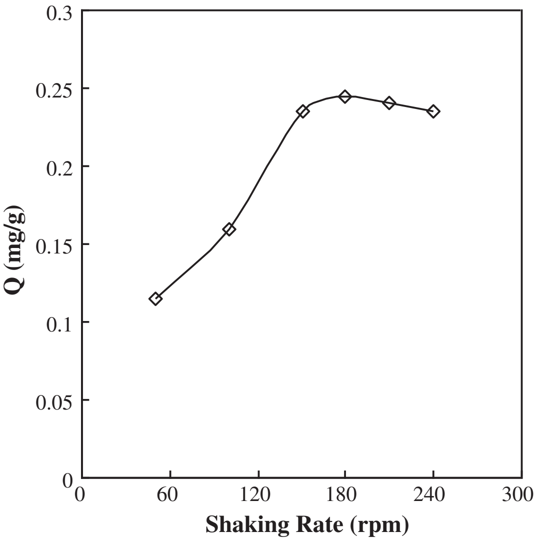

Fig. 3. Effect of shaking rate on the removal of NH_4^+-N. Added 3 g steel slag (particle size: 3–5 mm) into 150 mL NH_4^+-N solution (10 mg/L); changed shaking rate (50, 100, 150, 180, 210, and 240 rpm); shaken for 120 min at 25℃.

process was due to the external diffusion when the shaking rate was lower than 150 rpm. When the shaking rate was further increased, the adsorption amount of NH_4^+-N was almost unchanged at 0.24 mg/g. These indicated that when shaking rate is higher than 150 rpm, other adsorption process might be involved.

3.3. *Effect of particle size*

The adsorption amount of NH_4^+-N (Q) obviously increased from 0.14 to 0.25 mg/g with the decreasing of the particle size of steel slag from 11–13 to 3–5 mm as evident by the data in Fig. 4. The adsorption amount of NH_4^+-N was almost unchanged at 0.25 mg/g, with further decreasing of the particle size. The specific surface area is related to particle size. The N_2-BET of different particle size steel slags is shown in Table 2. It might be because of the fact that the smaller particle size would provide larger specific surface area, which led to higher mass diffusion flux of NH_4^+-N. When the particle size decreased to certain amount, the effect became relatively insignificant. When the particle size ranges from 3 to 13 mm, the adsorption rate is controlled by intra-particle because the intra-particle diffusion is positively related to the particle size. SEM images for the samples surfaces of different particle size before and after adsorption were obtained as in Fig. 5. The micrograph of steel slag before adsorption showed the significant difference on the surface of different particle size. With the decrease of particle size, abundant smaller spheroidal particle with acute angle adhered to the surface of steel slag, which is consistent with N_2-BET results. After adsorption, the surface morphology of steel slag was changed more clearly: small fine particles disappeared and glossy filamentous lumps appeared.

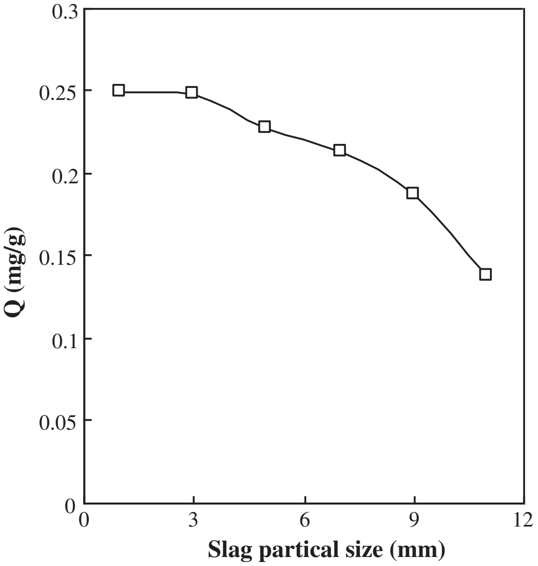

Fig. 4. Effect of particle size of steel slag on the removal of NH_4^+-N. Added 3 g steel slag (particle size: 3–5 mm) into 150 mL NH_4^+-N solution (10 mg/L); changed particle size (3–5, 5–8, 8–10, 13–15, 18–20, and 23–25 mm); shaken at 150 rpm and 25°C.

Table 2
The N_2-BET of different particle size steel slag

Particle size (mm)	1–3	3–5	5–7	7–9	9–11
BET (m^2/g)	0.46	0.32	0.19	0.08	0.01

3.4. *Boyd equation and Kannan equation*

As discussed in previous sections, the adsorption rate of solute on surface of the solid is mainly controlled by external diffusion and intra-particle diffusion. These two processes can be described by Boyd equation and Kannan equation, respectively, as expressed in Eqs. (1) and (2) [21].

$$\text{Boyd equation:} \quad -\ln(1-F) = k_1 t \tag{1}$$

$$\text{Kannan equation:} \quad Q = k_2 t^{0.5} \tag{2}$$

where $F = \frac{Q}{Q_e}$ is adsorption fraction at t moment, and k_1, k_2 are kinetic constant. The Q_e and Q are the amounts of solute adsorbed at equilibrium and at time t (min), respectively.

The plots $-\ln(1-F)$ vs. t, Q vs. $t^{0.5}$ (Fig. 6) should give the kinetic parameters $k_1$and k_2 related to Boyd and Kannan models, respectively. The correlation coefficients (Table 3) for Boyd equation at all concentration are higher. This implies that the adsorption follows Boyd model. In other words, the external diffusion dominates NH_4^+-N adsorption on steel slag. The correlation coefficients for Boyd equation decreased with increasing of initial concentration, which indicates that the effect of external diffuse is weakened at higher initial concentration.

According to Kannan model, a plot of Q vs. $t^{0.5}$ should give a straight line if intra-particle diffusion is involved in the adsorption process. In addition, if the plot passes through the zero then intra-particle diffusion is the sole adsorption rate-controlling step. In other words, if the plot does not pass through the zero, the intra-particle diffusion is not the only adsorption rate-controlling step [22]. As shown in Fig. 6(b), intra-particle diffusion was not the only rate-limiting step during absorption process of NH_4^+-N on steel slag because the plot dose not passes through the

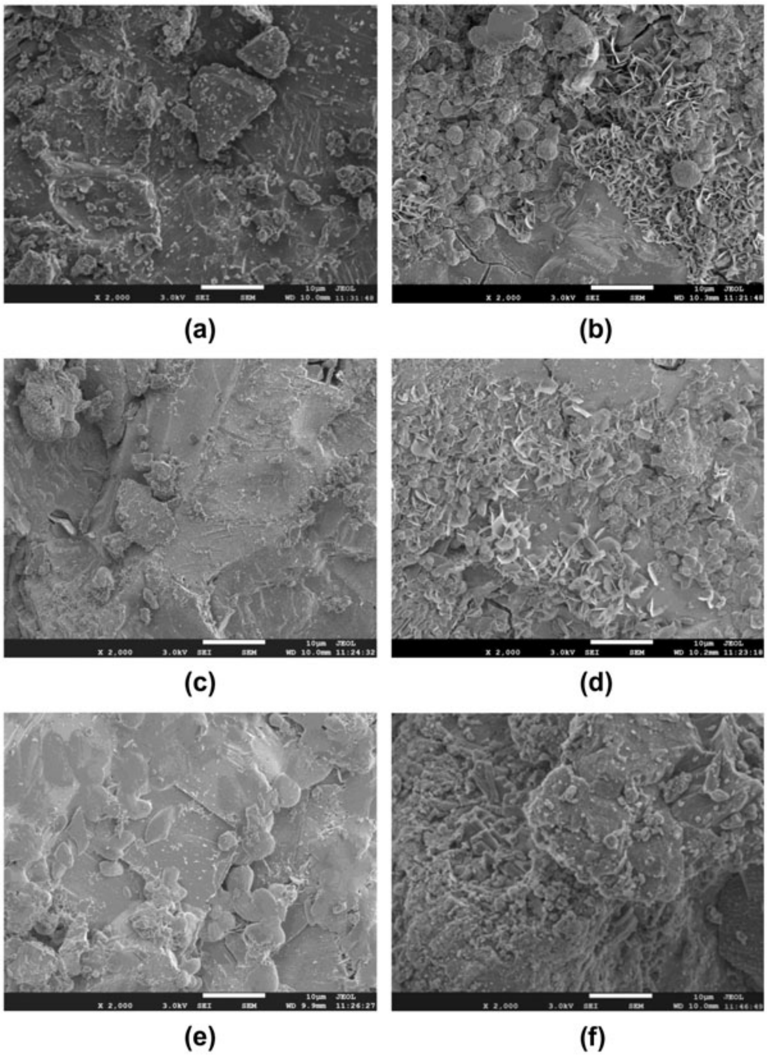

Fig. 5. SEM of steel slag before and after adsorption. (a), (b), (c): SEM of steel slag of 1–3, 3–5 and 5–7 mm before adsorption, respectively. (d), (e), (f): SEM of steel slag of 1–3, 3–5 and 5–7 mm after adsorption, respectively. The surface of steel slag samples were observed using a JEOL JSM-6510L type SEM under following conditions: working distance, 10 mm; accelerating voltage, 3.0 kV, magnification, 2000 × .

zero. As Wu [23] suggested that the experimental data can be modeled to multilinear by Kannan equation as shown in Fig. 6(b). In this case, the multilinear imply two or more steps govern the adsorption process. When initial concentration is higher than 25 mg/L, the experimental data can be fitted into three linear sections: the initial deeper section represents external diffusion, the second linear section represents a gradual adsorption stage where intra-particle diffusion is controlling step and the third section is final equilibrium stage. When initial concentration is lower than 25 mg/L, two linear sections can be modeled by Kannan equation: the initial deeper section represents external diffusion and the second section is equilibrium stage. These results are in good agreement with the date as discussed in sections 3.1–3.3.

3.5. Adsorption kinetics

In this study, NH_4^+-N adsorption from aqueous solution with time was analyzed by using Pseudo-second-order model, which usually describe not only diffusion process but also surface reaction process. The pseudo-second-order model can be expressed in Eq. (3) [24]. And k_3 are the rate constant for pseudo-second-order reaction [g/(mg min)].

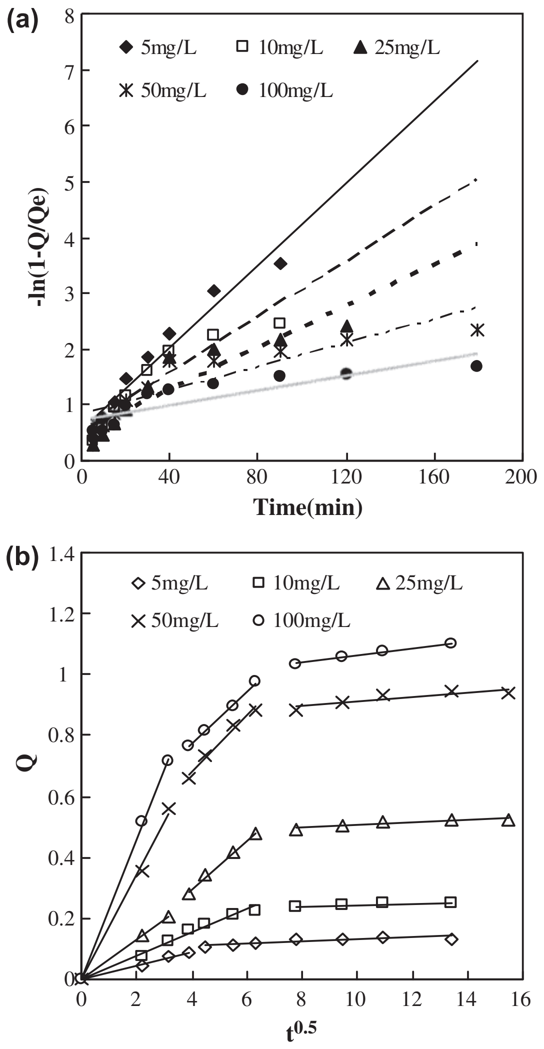

Fig. 6. Boyd equation and Kannan equation at different initial concentration. Experimental condition is the same as the condition of Fig. 1.

Table 3
The correlation coefficients for Boyd equation

Boyd equation	
Initial concentration (mg/L)	R^2
5	0.9476
10	0.9748
25	0.9228
50	0.9215
100	0.9092

$$\text{Pseudo-second-order equation:} \quad Q = \frac{Q_e^2 k_3 t}{1 + Q_e k_3 t} \tag{3}$$

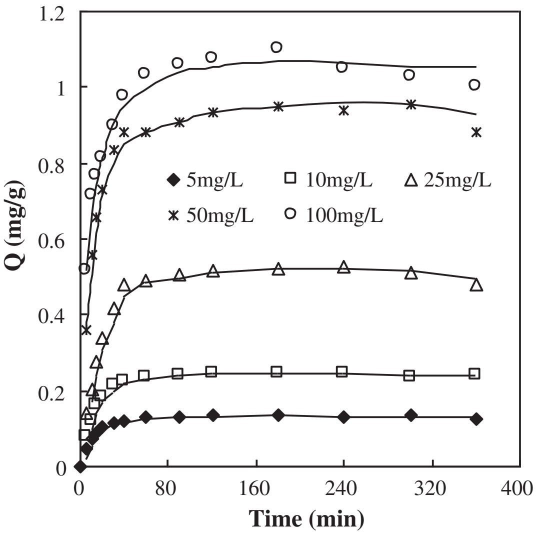

Fig. 7. Pseudo-second-order kinetics of NH_4^+-N adsorption onto steel slag particles at various concentrations. Experimental condition is the same as the condition of Fig. 1.

Shown in Fig. 7 is the result from fitting pseudo-second-order linear equation to the experimental data. The fitting parameters are listed in Table 4. Higher coefficients R^2 indicate that NH_4^+-N adsorption followed pseudo-second-order kinetics.

3.6. Adsorption isotherms

In terms of adsorption phenomena in certain temperature, it usually uses Langmuir equation and Freundlich equation to study the relationship between solid surface adsorption capacity and equilibrium adsorption of solute in the solution. The experimental data for absorbed NH_4^+-N by steel slag was compared using these two isotherm models. Langmuir and Freundlich equations were expressed in Eqs. (4) and (5) [25].

$$\text{Langmuir equation:} \quad Q = \frac{Q_m k_L C_e}{1 + k_L C_e} \tag{4}$$

Table 4
Pseudo-second-order models

C_0 (mg/L)	Q_e (mg/g)	k_2 [g/(mg min)]	R^2
5	0.14	3.1	0.9807
10	0.25	1.8	0.9786
25	0.53	1.3	0.9699
50	0.95	0.9	0.9489
100	1.10	0.8	0.9407

Table 5
Langmuir and Freundlich isotherms (T = 25℃)

	R^2	Q_m (mg/g)	K_L (L/mg)	k_f (mg/g)	n
Langmuir	0.9691	0.87	3.05	–	–
Freundlich	0.9527	–	–	18.54	1.87

Freundlich equation: $$Q = k_f C_e^{\frac{1}{n}} \tag{5}$$

where C_e is the equilibrium concentration (mg/L), Q_m is the maximum adsorption capacity (mg/g), k_L is the Langmuir constant related to the free energy of adsorption, k_f is Freundlich constant and n is the heterogeneity factor.

The different equation parameters of these models often provide insight into the adsorption mechanism, the surface properties and affinity of the adsorbent. The parameters for Langmuir and Freundlich models are listed in Table 5 and nonlinear plots are presented in Fig. 8. As result shown, both Langmuir and Freundlich can simulate the adsorption progress well as evidenced by high correlation coefficients. Moreover, it suggests that the Langmuir model gave slightly better fitting than that of Freundlich model due to a higher R^2 values in Table 5. The theoretical adsorption capacity of steel slag is 0.87 mg/g. Compare to the reported adsorbents, steel slag is ideal for low-cost wastewater treatment because of inexpensive and abundant. Besides, it is a solid waste and can be used as an adsorbent, which improve reuse of resource and protect the environment. It is also evident from these data that the surface of NH_4^+-N–steel slag is made up of homogenous adsorption patches than heterogeneous adsorption patches.

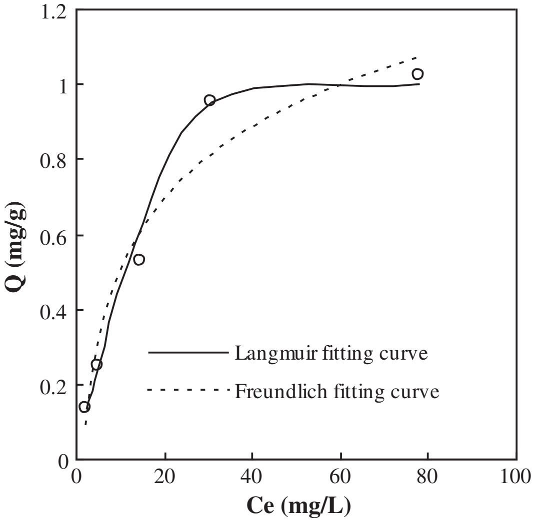

Fig. 8. Langmuir and Freundlich isotherms for NH_4^+-N adsorption on steel slag. Experimental condition is the same as the condition of Fig. 1.

The Langmuir constant k_L is a characteristic parameter related the binding energy of solute and adsorbent. It reflects spontaneous of adsorption reaction. Namely the greater of the k_L, the higher of the spontaneous adsorption, so it has more stable of the product and better of adsorption capacity of NH_4^+-N [24]. In the experiment the k_L value is relative low, it means the adsorption product of steel slag and NH_4^+-N is not stable and it easy to desorbed. The Freundlich constant n is a measure of the deviation from linearity of the adsorption, and its value usually between 0–10. If $n \geq 2$, it means the adsorption process is privilege type. If $n > 1$, it implies that the adsorption is favorable type [26]. Therefore these results suggest that NH_4^+-N adsorption on steel slag is preferential type.

4. Conclusions

This study provides useful data and ample evidence on the adsorption of NH_4^+-N on steel slag and tries to establish base for further utilization of steel slag as a low-cost adsorbent for NH_4^+-N removal. The controlling step of NH_4^+-N adsorption varied under different experimental conditions. The adsorption kinetics could be accurately described by Boyd equation, Kannan equation and the pseudo-second-order kinetic model. The isotherm of NH_4^+-N adsorption by steel slag was found to follow both the Langmuir and Freundlich models, but it has slightly better fitting on Langmuir model. According to the Freundlich models, the adsorption could be categorized as the preferential type.

Acknowledgments

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