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# Adsorption properties of rare earth soils on Ammonium Nitrogen

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Abstract. The in-situ leaching of ionic rare earth minerals often causes serious nitride pollution in the mining area and surrounding areas. In this paper, the rare earth soil in the Zu Dong mining area of Long Nan, Gan Zhou city, was used as the research object. Through batch equilibrium adsorption tests, the adsorption properties of ammonium nitrogen in different sizes of rare earth soils, the adsorption dynamics and thermodynamics of ammonium nitrogen under different concentrations of ammonium sulfate solution, and the effect of the solution flow rate and concentration in the soil column under dynamic conditions on the adsorption of ammonium on the soil was systematically studied. The results showed that in different diameters of rare earth soils, the adsorption capacity was: clay (within 0.074 mm) > fine sand (range 0.074 to 0.25 mm) > sand (beyond 0.25 mm) > original soil (12.37% clay, 43.9% sandy soil, and 31.56% fine sand), indicating that the clay in the original soil played a major role; the adsorption of ammonium nitrogen in ionic rare earth soils satisfied the Langmuir model (the fitted  $R^2$  values all greater than 0.9), and the dynamics satisfied the quasi-second-order dynamics equation (the fitted  $R^2$  values all greater than 0.95), which showed that the adsorption of ammonium nitrogen in soil was monolayer adsorption and dominated by chemical adsorption process; the concentration of ammonia nitrogen, the speed of leaching, and the content of cations such as rare earths all play an important role in the adsorption of ammonia nitrogen in rare earths.

# 1. Introduction

China is a large country of rare earths, of which more than 50% of our rare earths are located in the southern Gan Zhou city of Jiangxi Province [1]. The mining of ion-type rare earth ore in southern Gan Zhou is currently used in-situ leaching, that is, ammonium sulfate solution is injected through surface excavation injection wells to exchange rare earth ions for collection purposes. But due to the unsatisfactory effect of anti-seepage measures such as man-made baffles, a large amount of leaching agent enters into the mining area soil and surrounding water bodies, and spreads to the surrounding area with the rain brush. Because the negative charge of the soil surface will combine with  $NH_4^+$ , most of the ammonium nitrogen flowing through the rare earth soil will be absorbed by the soil, thus blocking and delaying the migration and transformation of ammonium nitrogen [2,3]. However, there is a certain saturation limit for the adsorption of ammonium in the soil. When the adsorption is saturated, it will enter the groundwater through migration. Through the investigation of the water

Content from this work may be used under the terms of the Creative Commons Attribution 3.0 licence. Any further distribution of this work must maintain attribution to the author(s) and the title of the work, journal citation and DOI. Published under licence by IOP Publishing Ltd 1 around the mine in the Zu Dong rare earth ore district in Long Nan, it was found that the ammonia nitrogen content in the pond near the tailings was between 100~200 mg/L [4], which far exceeds the suitable growth requirement for crops by 25 mg/L, and far exceeds water quality standards of class V [5].

The performance of non-rare earth adsorbing ammonium nitrogen has been studied by a large number of scholars in the world. Wen [6] choosed the soil near the landfill site as a research object to explore its adsorption capacity for ammonium nitrogen in landfill leachate. It showed that metal ions of landfill leachate competed with  $NH_4^+$  for adsorption [7,8], which weakened the soil's adsorption of ammonium nitrogen and was obvious at high concentrations, leaving large amounts of ammonium nitrogen not adsorbed by the soil. Jasmin D [9] studied the adsorption properties of ammonia in soil in the air entrainment belt. The results showed that the adsorption capacity of ammonia in different soils varies greatly with the particle size, which is expressed as: silty clay> sandy clay> silty sand. However, as a special type of soil, ionic rare earths have rarely been studied the adsorption of ammonium nitrogen. After exogenous REEs entered the soil, most of them remained in the soil surface [10], and would affect soil biochemical processes for a long time. Similar to heavy metals, high doses of REEs inhibited ammonia oxidation and organic nitrogen mineralization [11,12]. The study of the application of single rare earth elements La and Ce for spring cabbage found that single rare earth elements increased the content of vitamin C and nitrate [13]. As we know, humans consumed most of the nitrates needed by their bodies through vegetables, but if too much nitrate was reduced to nitrite it would cause Methemoglobinemia in humans [14]. In addition, Sun Liangliang et al [15] studied Arabidopsis root growth and found that by inhibiting the accumulation of nitric oxide, La can significantly relieve the inhibitory effect of La on the cell division of the apical meristem, thereby alleviating the inhibitory effect of La on the growth of the main root. Therefore, it is of great significance to carry out studies on the adsorption performance of rare earth minerals between soils and ammonia nitrogen. Not only can we understand the role of ammonium nitrogen in soil, but also to provide some scientific theoretical basis for further investigation of the transfer and conversion of ammonium nitrogen in soil, adsorption mechanism, and prevention of groundwater pollution.

# 2. Materials and methods

# 2.1. Experimental materials

In this experiment, the unexplored raw ore samples in Zu Dong mining area were selected, which is a type of rare earth ore mainly composed of medium and heavy rare earths and the ion form is the main form of existence of such rare earths [16]. Take back the soil: remove the impurities, air dry naturally, then grind it and put in an oven (105  $^{\circ}$ C) to dry, finally seal it after cooling. The physical and chemical properties of soil were shown in table 1.

Table 1. Physical and chemical	l properties of rare earth soils.
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physical and chemical properties	pН	water content	organic matter	Ammonium nitrogen
content	5.37	28.6%	1.32 g/kg	88 mg/kg

# 2.2. Experimental instruments

The main experimental instruments include: electronic analytical balance (JJ100B, CHANG SHU SHUANG JIE Test Instrument Factory); constant temperature oscillator (SHA-C, JIN TANRONG HUA Instrument Manufacturing Co., Ltd.); spectrometer (SP-756PC, Shanghai Spectrum Instrument Co., Ltd.); XRD (Japan MINIFLEX type).

# 2.3. Test methods

Ammonia-nitrogen content in water was analyzed by sodium-resin spectrophotometry, and the amount of ammonia nitrogen adsorbed by the soil was measured at 420 nm using an ultraviolet spectrometer.

#### 2.4. Adsorption dynamics experiment

The experimental configuration of ammonium sulfate concentrations were 100 mg/kg, 600 mg/kg, and 1000 mg/kg respectively. Add the above configuration solution to soil in an 80 mL centrifuge tube at a ratio of 5:1.At 1 min, 5 min, 10 min, 15 min, 20 min, 30 min, 40 min, 60 min, 90 min and 120 min shaking time, the supernatant was measured after centrifugation to determine the ammonia nitrogen content in the solution. Then calculate the ammonia nitrogen adsorbed in the soil according to formula (1) and discuss the dynamic process of adsorption of ammonium nitrogen in clay soil. Repeat three times for each experiment and average the results. Finally calculate the amount of ammonia nitrogen adsorbed by rare earth soil according to the change of ammonia nitrogen concentration before and after adsorption.

$$Q_t = (C_0 - C_t) \times V / m \tag{1}$$

where  $Q_t$ : ammonia nitrogen adsorption capacity of rare earth soil, mg/kg;  $C_t$ : concentration of ammonia nitrogen in the solution at time t, mg/kg.

#### 2.5. Adsorption thermodynamics experiment

According to China's soil classification method, the original soil was classified into three categories: 60-mesh (greater than 0.25 mm particle size) sand, 60-200 mesh (particle size range 0.074 mm to 0.25 mm) fine sand, and 200 mesh (particle size less than 0.074 mm) clay. In this experiment, the adsorption thermodynamics of soils with four particle sizes (including the original soil) was studied: prepare ammonium sulfate solutions with mass concentrations of 0.1 g/kg, 0.2 g/kg, 0.4 g/kg, 0.8 g/kg and 1 g/kg; weigh 5g of the above 4 kinds of particle size soil; add ammonium sulfate solution in an 80 mL centrifuge tube according to the water to soil ratio 5:1; shake for 90min, adjust shaker temperature 25  $^{\circ}$ C, speed 180 r/min, repeat three times for each treatment; calculate the amount of ammonia nitrogen adsorbed by rare earth soil according to the change of ammonia nitrogen concentration before and after adsorption.

$$Q_t = (C_0 - C_e) \cdot V/m \tag{2}$$

where  $Q_t$ : the equilibrium adsorption capacity of rare earth soil to ammonium sulfate solution, mg/kg;  $C_0$ : ammonia nitrogen initial mass concentration in ammonium sulfate solution before adsorption, mg/L;  $C_e$ : ammonia nitrogen mass concentration in solution during adsorption, mg/L; V: adsorption volume, mL; m: soil quality, g.

#### 2.6. Characterization of adsorbents

X-ray diffraction (XRD): select  $C_u k_a$ , Ni filter (wavelength  $\lambda$ =1.5406 Å, tube voltage 40 KV, tube current 150 mA, scan range=9~90°, step size 0.02°) for phase analysis of rare earths before and after leaching.

#### 3. Results and discussion

# 3.1. Comparative analysis of adsorptive capacity of different particle sizes

Compare the adsorption performance of clay, fine sand, medium sand and the original soil (figure 1), it was found that the concentration of adsorbed ammonia nitrogen in the soil increased with the initial concentration, and the clay adsorption capacity was greater than that of the other three particle sizes, indicating that the clay was the main component of soil in the original soil. In the original rare earth soil, the clay content was 12.37%, the sand content was 43.9%, and the fine sand was 31.53%. The adsorption capacity of the remaining three particle sizes was fine sand> sandy soil> original soil. The reason may be that the smaller the particle size, the larger the specific surface area, and therefore the greater the adsorption capacity. It can also be seen from figure 1 that the adsorption capacity of ammonia nitrogen of soils with different particle sizes at low concentrations was not significantly different because the adsorption sites on the soil surface at low ammonia concentration were not

sufficient enough to be fully occupied; but with the increase of  $NH_4^+$  concentration, the adsorption sites were gradually occupied by ammonia nitrogen under the condition that the adsorption sites on the surface of each particle size were certain, and therefore the adsorption capacity tended to increase.



Figure 1. Soil adsorption dynamics curves and fitting equations for different ammonium sulfate concentrations.

#### 3.2. Dynamics analysis of clay adsorption for ammonium

Chemical reaction and ion diffusion are the main processes of adsorption dynamics. Quasi-first-order dynamics model, pseudo-second-order dynamics model, and particle diffusion equation are widely used in the adsorption experiments to explore the adsorption dynamics equation of adsorption rate variation. In these models, the quasi-first-order dynamics equation was a model based on the premise that the adsorption was affected by diffusion. The adsorption rate was directly proportional to the difference in adsorption amount [17]. The particle diffusion equation was the most accurate description of intraparticle diffusion [18,19].

• First-order dynamics equation

Lagrgren:

$$dq / dt = k_1 \left( q_e - q \right) \tag{3}$$

It can be translated into:

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$$\lg(q_{e} - q) = \lg q_{e} - k_{1}t / 2.303 \tag{4}$$

• Second-order dynamics equation Quasi-second-order dynamics model:

$$dq / dt = k_2 \left(q_e - q\right)^2 \tag{5}$$

It can be translated into:

$$t/q = 1/k_2 q_e^2 + t/q_e$$
(6)

• The particle diffusion equation

$$q = kt^{0.5} + C \tag{7}$$

where q: soil adsorption at time t, mg/kg; t: adsorption time, min;  $q_e$ : adsorption capacity at equilibrium, mg/kg; k: particle diffusion rate constant;  $k_1$ : first-order adsorption rate constant, min<sup>-1</sup>;  $k_2$ : second-order adsorption rate constant, min<sup>-1</sup>.

The kinetic curve and fitting curve of ammonium adsorption on soil under different leaching solution concentrations were shown in figure 1, where the abscissa represented the soil adsorption time, and the ordinate represents the amount of ammonium nitrogen adsorbed by the rare earth soil. From figure 1, we can see that the overall trend of the adsorption curve at the three different concentrations was approximately the same, both as follows: rapid adsorption to saturation and then in a dynamic equilibrium, but the rate of increase was different at different concentrations, and the time required for the adsorption equilibrium would be shorter with increasing concentration of adsorbent. When the adsorbent concentration was 1%, the adsorption amount increased sharply until it reached equilibrium in the first 35 minutes. When the adsorbent concentration was 0.1%, the adsorption rate increased slowly until equilibrium compared to the former. The reason may be that as the concentration of adsorbent increased, the more ammonia that can be adsorbed in the system, the less time it took to reach equilibrium. Because when the adsorption site was constant, increasing the concentration gradient between the soil and ammonium sulfate would increase the initial adsorbent content; When NH<sub>4</sub><sup>+</sup> just entered the soil, there was enough adsorption site on the soil surface. When the concentration of ammonia nitrogen reached a certain concentration, the adsorption site was gradually occupied. However, since there was also physical adsorption in the adsorption, it was in a state of adsorption and separation dynamic equilibrium after the adsorption was stabilized.

The equilibrium adsorption capacity of the experimental values at the three concentrations was: 43.41 mg/kg, 146.2 mg/kg and 320.01 mg/kg, which were similar to the predicted values of the secondary adsorption model 46.22 mg/kg, 153.36 mg/kg and 325.41 mg/kg. From table 1, the fitted R<sup>2</sup> values of the secondary adsorption model were all greater than 0.9, which was better than the first-order adsorption model and the particle diffusion equation. The quasi-secondary adsorption model contains the entire process of migration, diffusion, physical and chemical reactions that existed in adsorption. It can be seen that the occupation of the adsorption sites, the diffusion of the external liquid film, and the diffusion in the soil affected the adsorption of ammonia nitrogen in the rare earth soil. The removal of ammonia nitrogen in rare earth soils was mainly accomplished through ion exchange and adsorption [20], and controlling the ion exchange process was the main way to control ammonia nitrogen adsorption [21]. Therefore, in the actual ionic rare earth soil, the removal of ammonia nitrogen should be achieved mainly by eliminating chemical adsorption.

#### 3.3. Isothermal adsorption analysis of clay and fine sand

The solid-liquid phase adsorption isotherms were often described by Langmuir and Freundlich model. The isotherm Langmuir assumed that the adsorbent surface was monolayer and the adsorption capacity was the same at each site. But the Freundlich otherm assumed that the adsorption sites are non-uniformly distributed on the adsorbent surface.

Langmuir linear model:

$$\rho_{e} / q_{e} = \rho_{e} / q_{0} + 1 / q_{0} k \tag{8}$$

Freundlich linear model:

$$\ln q_e = \ln K_f + \ln \rho_e / n \tag{9}$$

where  $q_e$ : adsorption of ammonium nitrogen concentration per unit mass of soil;  $\rho_e$ : concentration of ammonium nitrogen in the solution during adsorption equilibrium;  $q_0$ : maximum adsorption capacity.  $K_f$ : Freundlich constant, indicating the adsorption capacity of the soil; 1/n: heterogeneous factors.

According to different temperature, the isothermal analysis of the adsorption of ammonium nitrogen on clay soil and fine sand soil was carried out. Taking ammonium bicarbonate as the abscissa for the effluent and ammonium nitrogen as the ordinate for the soil with unit mass, the isothermal adsorption curves of clay and fine sand soil were fitted.



**Figure 2.** Adsorption isotherms and Freundlich, Langmuir fitting curves for different soils at different temperatures.

According to the relevant literature, during the adsorption process of non-rare earths, temperature was an important factor affecting the soil's adsorption of ammonium nitrogen, and the increase of temperature can promote the adsorption of ammonia nitrogen [22-25]. But the effect of temperature on the adsorption of ammonia nitrogen was small in this experiment. As can be seen from figure 2, the overall trend of soil adsorption was 15 C>25 C>35 C, indicating that low temperature promoted the adsorption of ammonia on the soil. Rare earth soils and non-rare earth soils showed opposite characteristics. The reason for the difference may be that the adsorption of ammonia on the soil was a balance between physical and chemical processes. On the one hand, because soil adsorption was a weak exothermic process, reducing the temperature would have an accelerating effect on adsorption;

on the other hand, ammonia in the water solution had a chemical balance, and the temperature rose in the system would increase the concentration of  $NH_4^+$ . This leaded to an increase in the amount of solid phase adsorption. In different soils, the opposite effect of these two processes showed different adsorption results for temperature changes.

Compared with 60-mesh sand, the clay adsorption capacity of 200-mesh clay was greater than that of fine sand. The Langmuir, Freundlich and particle diffusion equations were used to fit the process of ammonia adsorption in soil. From the three fitting equations (table 2), Langmuir and Freundlich can better fit the isotherm adsorption process of ammonia on the clay soil, which  $R^2$  were all greater than 0.95. The fitting effect of Langmuir was slightly better than that of Freundlich, indicating that the soil surface was monolayer and the adsorption capacity of each site was the same. In the Langmuir equation, the  $k_f$  value represented the adsorption binding energy. The larger the value, the more stable the ammonia nitrogen adsorption in the soil [26], in which the temperature value was inversely proportional to the binding energy of clay and fine sand, indicating that the adsorption capacity between soil and ammonia nitrogen in winter was greater than that in summer; In the Freundlich equation, the  $k_f$  value was proportional to the adsorption capacity. But from figure 2 it can be seen that the lower the temperature, the stronger the adsorption capacity, which was consistent with the conclusion drawn by the Langmuir equation.

temperature/°C	particle size	thermodynamic model	$R^2$	п	$k_{f}$
15	clay	Freundlich	0.97	1.28	4.5
25			0.96	1.25	3.91
35			0.96	1.14	2.59
temperature/°C	particle size	thermodynamic model	$R^2$	k.Q	Q
15	clay	Langmuir	0.98	1.90	951
25	5	0	0.97	1.18	985
35			0.97	1.4	1493
temperature/°C	particle size	thermodynamic model	$R^2$	k.Q	Q
15	fine sand	Langmuir	0.97	2.04	1022
25		C	0.97	1.27	1266
35			0.97	1.24	1241
temperature/°C	particle size	thermodynamic model	$R^2$	n	$k_{f}$
15	fine sand	Freundlich	0.96	1.23	3.55
25			0.96	2.9	2.9
35			0.96	1.17	2.76

**Table 2.** Thermodynamic simulation parameters for adsorption of ammonium nitrogen in different soil particle sizes.

#### 3.4. Characterization results of adsorbents

The soil was taken from raw ore samples in the mining area. The semi-quantitative analysis of soil samples by X-ray fluorescence spectra (XRF) was a rare earth mine enriched with yttrium oxide. The specific results were shown in table 2. From table 2, we can see that the grade of aluminum was 14.735%, and the grade of rare earth was 0.09%. After analyzing the rare earth ore sample by means of a diffraction spectrum (figure 2), we can obtain that the mineral content of the soil was mainly quartz, mica and clay minerals. Among them, Re<sup>3+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, K<sup>+</sup> and Na<sup>+</sup> were exchangeable cations in the soil and played an important role in the exchange process with NH<sub>4</sub><sup>+</sup> and other cations [27].

From the XRD pattern before and after the immersion liquid (figure 2), it can be seen that the material before the immersion liquid was highly dispersed and the minerals and rare earths were

precipitated after immersion. The white mica after immersion showed a corresponding peak at the angles of 45.47  $^{\circ}$ , 26.83  $^{\circ}$  and 17.77  $^{\circ}$ , and the aluminum silicate compound also showed clearer peaks at 14.36  $^{\circ}$  and 25.8  $^{\circ}$ .

# 4. Conclusions

Through the static adsorption, the performance of ionic rare earth soils for adsorption of ammonium nitrogen is investigated. The following conclusions are obtained:

- In rare earths with different particle sizes, the adsorption capacity is: clay (grain size within 0.074 mm)> fine sand (grain size range 0.074 to 0.25 mm)> sand (grain size beyond 0.25 mm)> original soil (the clay content is 12.37%, sandy soil is 43.9%, and fine sand is 31.53%), which indicates that clay is the main component in the original soil.
- The general trend of the adsorption curves at the three different concentrations is approximately the same, both as follows: rapid adsorption to saturation followed by dynamic equilibrium, but the rate of increase in different concentrations is different. The equilibrium adsorption capacities for the three concentrations were: 43.41 mg/kg, 146.2 mg/kg and 320.01 mg/kg, respectively. The estimated values for the secondary adsorption model were 46.22 mg/kg, 153.36 mg/kg and 325.41 mg/kg, which are close. The fitted value R<sup>2</sup> of the secondary kinetics is greater than 0.9, which is superior to the first-order kinetics and particle diffusion equation. The fitting effect of Langmuir is slightly better than that of Freundlich fitting, indicating that the soil surface is monolayer adsorption and the adsorption capacity of each site is the same.
- Langmuir and Freundlich can better fit the isotherm adsorption process of ammonia on the clay soil and  $R^2$  is greater than 0.95. In this experiment, the temperature has little effect on the adsorption of ammonia nitrogen, and the overall trend of soil adsorption is 15 C>25 C>35 C.
- RE<sup>3+</sup>, Ca<sup>2+</sup>, Mg<sup>2+</sup>, K<sup>+</sup> and Na<sup>+</sup> were exchangeable cations in the soil and played an important role in the exchange process with NH<sub>4</sub><sup>+</sup>.

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