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Research on absorption of ammonia by Nitric acid-modified Bamboo Charcoal at low temperature

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ABSTRACT

To solve the problem of low concentration of ammonia (NH_3 and/or NH_4^+) in China North winter Waterworks, Bamboo Charcoal was modified by nitric acid to enhance its absorption capacity. A few important factors such as absorbent dosage, contact time, temperature and initial ammonia concentration were investigated. Scanning electron microscope and infrared radiation were used for characterizing. The equilibrium data were analyzed using the Langmuir and Freundlich isotherm models. The equilibrium adsorption data fitted well into Langmuir adsorption isotherm and showed a maximum ammonia adsorption capacity of 0.65 mg/g. The best efficiency of Modified Bamboo Charcoal dosage, initial ammonia concentration, contact time and temperature were 1.5 g, 5 mg/L, 2 h and 5℃, respectively. Compared with Raw Bamboo Charcoal, Nitric acid-modified Bamboo Charcoal displays a better ammonia removal capacity(from 10 to 40%), which implies a potential application for removing ammonia pollutants from drinking water at low temperature.

Keywords: Nitric acid-modified Bamboo Charcoal; Ammonium; Drinking water; Low temperature; Adsorption

1. Introduction

Ammonium (NH_4^+), one of the important pollutants in municipal sewage and many industrial wastewaters, can cause increase of oxygen demand and eutrophication in rivers and lakes [1]. Several researchers have found that the flocculation process could give high particle removal efficiencies in terms of organic compounds, nitrogen and phosphorus [2], However, because NH_4^+ is in the ionic state, it cannot be removed by flocculation. NH_4^+ can be removed by biological and physicochemical methods: nitrification–denitrification, precipitation, oxidation, adsorption, stripping, etc. [3]. However, some of these methods have limitations and shortcomings. Conventional methods for ammonium removal are mostly based on biological treatments [4], but it is often not effective to be used for ground water at low temperatures (0–5℃) in the reservoir of northern China in winter. Biological systems, especially nitrification processes, are strongly affected by the water temperature [5]. Chlorination and ion-exchange processes have also occasionally been applied to industrial wastewaters containing high levels of ammonia. However, the disadvantages are their high cost and difficult maintenance due to the chemicals used in the oxidation and

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regeneration steps [4,6,7]. From a practical stand point, adsorption is one of the most promising techniques, although pollutants are just transferred from one phase to another rather than eliminated and recovery of pollutants and regeneration of adsorbents by desorption processes have to be carried out [8]. A few ammonium adsorption removal techniques are still considered as the most effective and well-estabished methods, including essentially adsorption onto activated carbon and zeolites [9–12]. However, the high cost of activated carbon and the low abundance of zeolite in many countries, as well as the high costs of regeneration processes by chemical and thermal procedures, are a problem [13]. An adsorbent should be available in large quantities, easily reusable and cheap.

Bamboo charcoal is an environmentally friendly, low-cost and renewable bioresource with a porous structure. The powder contains many pores and gaps in its structure, making it excellent for absorption, electromagnetic shielding and infrared emission [14]. Several studies have found that activated Bamboo Charcoal has an excellent adsorption capacity for a wide variety of substances, such as nitrate-nitrogen [15], heavy metals [16,17], dibenzothiophene [18], and phenol [19], harmful gases, and can be potentially used for the purification of water or air.

The aim of this research is to investigate the adsorption characteristics of Bamboo Charcoal for low concentrations of ammonium in low temperature drinking water. Meanwhile, the Nitric acid-modified Bamboo Charcoal was also studied, to gain a better understanding of the adsorption characteristics for ammonium and to provide the preliminary experimental basis for the processing of modified Bamboo Charcoal for the removal ammonium.

2. Materials and methods

2.1. Materials

All chemicals used were analytical grade reagents from Merck. Analytical grade ammonium chloride salt (NH_4Cl) and distilled water were used for the preparation of a stock NH_4^+ solution of 1,000 mg/L. Ammonium solutions of different concentrations were prepared by diluting the NH_4Cl stock solution with distilled water. The ranges of ammonium concentrations used in this study varied between 1 and 5 mg/L. The initial pH was 7 which was maintained to be very consistent with the pH stated in the China Standards for Drinking Water Quality. The temperature was controlled between 1 and 5℃ to maintain low temperatures.

Bamboo Charcoal (purchased from a market at Zhejiang, China) was dipped in deionized water and stirred at 60℃ for 4 h to remove dust and then dried at 115℃ in a drying oven for 12 h. Bamboo Charcoals were originally in sheet shapes with a mean diameter of approximately 10 mm. The pretreated Bamboo Charcoal materials were pulverized to below 200 mesh, then stored in a desiccator for further use. The main characteristics are listed in Table 1.

2.2. Formation of Nitric acid-modified Bamboo Charcoal

Experiments for Nitric acid-modified Bamboo Charcoal were performed as follows. Firstly, 200 mesh Bamboo Charcoal (1 g) was added to a 1,000 mL round-bottomed flask, mixed with 700 mL of 30% (v/v) nitric acid and refluxed at 80℃ for 3 h. Secondly, the Bamboo Charcoal was recovered from the cooled mixture by centrifugation at 2,000 rpm for 30 min with decanting of the supernatant. Thirdly, the reaction solution was naturally cooled for 2 h, then ultrafiltered, washed by thorough resuspension in deionized water until pH 7 was attained, dried at 115℃ in a drying oven for 12 h, then stored in a desiccator for further use.

2.3. Characterization of the Bamboo Charcoal and Nitric acid-modified Bamboo Charcoal

2.3.1. Fourier transform infrared spectroscopy

Fourier transform infrared (FTIR) spectra (transmission) were recorded on a Perkin-Elmer Model Spectrum One in the range of 4,000–450 cm^{-1} at a resolution of 1 cm^{-1}.

2.3.2. Field emission-scanning electron microscope

Field emission-scanning electron microscope (FE-SEM) was used for morphological observations of Bamboo Charcoal and Nitric acid-modified Bamboo Charcoal. The surface characteristics of both of them, such as specific surface area and porosity, were deter-

Table 1
The main characteristics of the tested Bamboo Charcoal

Bamboo Charcoal sample	
Iodine value	⩾892 mg/g
BET surface area (m^2/g)	⩾157 m^2/g
Pore volume (m^3/g)	>0.87 cm^3/g
Phenol adsorption rate	⩾450 mg/g
Apparent density	0.38–0.45 g/cm^3
Strength	⩾90%
pH	⩾7

mined by N_2 adsorption apparatus (Micromeritics ASAP 2000).

2.3.3. The surface functional groups

The surface functional groups containing oxygen were determined according to Boehm titration [20]. One gram of Bamboo Charcoal sample was placed in 50 mL of the following solutions: sodium hydroxide, sodium carbonate, sodium bicarbonate, and hydrochloric acid, the concentration which was 0.05 mol/L, respectively. The vials were degassed under high purity nitrogen flow, sealed and shaken for 2 days to reach equilibrium and then filtered. Five milliliter of the filtrate was pipetted and the excess base or acid was titrated with 0.05 mol/L hydrochloric acid or sodium hydroxide, respectively. The number of basic sites was calculated from the amount of hydrochloric acid that reacted with the activated carbon. The number of acidic sites was determined under the assumptions that NaOH neutralizes carboxylic, lactonic, and phenolic groups; that Na_2CO_3 neutralizes carboxylic and lactonic groups; and that $NaHCO_3$ neutralizes only carboxylic groups. In each case, the determination was carried out in triplicate [21].

2.4. Measurement of ammonium adsorption

The adsorption of ammonium on the Nitric acid-modified Bamboo Charcoal was investigated in batch mode sorption equilibrium experiments. All batch experiments were carried out in 250 mL conical Erlenmeyer flasks containing 100 mL of ammonium solution, which was continuously shaken at 165 rpm at 1–5℃.

The effect of contact time on ammonium removal by the Bamboo Charcoal and Nitric acid-modified Bamboo Charcoal was investigated ranging from 0 to 720 min at 5℃. The initial ammonium concentration and adsorbent dosage were 5 mg/L and 1 g/L, respectively. According to the results of contact time tests, the optimal contact time was fixed at 6 h for the following experiments. The time span was sufficient to reach equilibrium.

The effect of pH was investigated in the pH range from 3.0 to 7.0 at 5℃. NH_4^+ and ammonia (NH_3) can coexist in an aqueous solution according to the following chemical reaction Eqs. (1) and (2) [22].

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \quad (1)$$

$$K = \frac{[NH_4^+][OH^-]}{NH_3} = 1.82 \times 10^{-5} \quad (2)$$

The chemical equilibrium of this reaction depends on pH. There are only ammonium ions (NH_4^+) in solution when the pH $\leqslant$ 7. The percentage of NH_3 in moles is increasing as the pH is increasing, For instance, at pH 11, 98.23% of the ammonia content is in the NH_3 form. By increasing the pH value to 12 and 13, the NH_3 form of ammonia becomes 99.82 and 99.98%, respectively (Fig. 1) [22]. The modified Bamboo Charcoal mainly adsorbed the ammonium in ground water in the research, but when the pH $\geqslant$ 7, NH_4^+ will be converted into NH_3 and partial dissolution of acidic functional groups will occur, so the pH value was researched to between 3.0 and 7.0. The initial ammonium concentration and adsorbent dosage were 5 mg/L and 1 g/L, respectively. The ammonium solution was adjusted to the desired pH (3.0–7.0) by adding 1 mol/L of HCl or NaOH. The pH of the solution was monitored by a pH meter.

The effect of Nitric acid-modified Bamboo Charcoal dosage on the ammonium removal was studied in the range of 0.2–1.2 g/L at 5℃. The initial ammonium concentration was 5 mg/L. The pH of the solutions was adjusted to 7.0.

The effect of the initial ammonium concentration and temperature was studied between 1–5 mg/L and 1–5℃. The adsorbent dosage was 1 g/L. The pH of the solutions was adjusted to 7.0.

The thermodynamics process of ammonium removal was studied at pH 7 at 1–5℃. The initial ammonium concentration and adsorbent dosage were 5 mg/L and 1 g/L, respectively. The isotherm equations which are Langmuir and Freundlich are given in Table 2 [23,24]. For all of the batch experiments studied above, the suspension of each test was centrifuged and filtered through a 0.45 μm membrane filter. The initial and final concentrations were analysed by

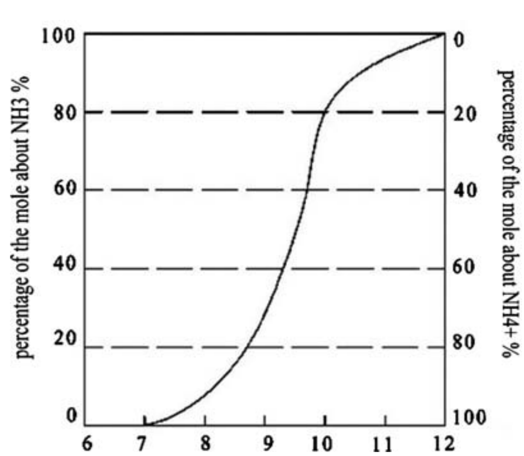

Fig. 1. Effects of pH on NH_4^+ and NH_3 equilibrium.

Table 2
Isotherm models

Isotherm model	Equation	
Langmuir	$\frac{C_e}{Q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m}$	Q_e is the ammonium concentration on the adsorbent (mg/g)
		C_e is the equilibrium ammonium concentration in solution (mg/mL)
		q_m is the monolayer capacity of the adsorbent (mg/g)
		$K_L$is the Langmuir constant and related to the free energy of adsorption
Freundlich	$\ln Q_e = \ln K_F + n \ln C_e$	C_e is the equilibrium concentration of ammonium at the equilibrium time (mg/L)
		n is the Freundlich constant related to adsorption intensity
		K_F is the Freundlich constant related to the relative adsorption capacity (mg/g)

employing a spectrophotometer based on the standard Nesslerization method at an absorbance of 425 nm using a HACH DR/2010 (Method 8038). All the analyses were performed in triplicate, and the result was calculated as the average. The amounts of ammonium adsorbed by the Nitric acid-modified Bamboo Charcoal (q_e, mg/g) and the ammonium removal percentages were calculated by Eqs. (3) and (4).

$$q_e = \frac{(C_0 - C_e)V}{m} \qquad (3)$$

where, C_0 is the initial ammonium concentration (mg/L), C_e is the equilibrium ammonium concentration (mg/L), V is the batch volume (L) and m is the Nitric acid-modified Bamboo Charcoal (g).

The percentage of ammonium removal efficiency (η) from the aqueous solution was then calculated from:

$$\eta\ (\%) = \frac{(C_0 - C_e)}{C_0} \times 100 \qquad (4)$$

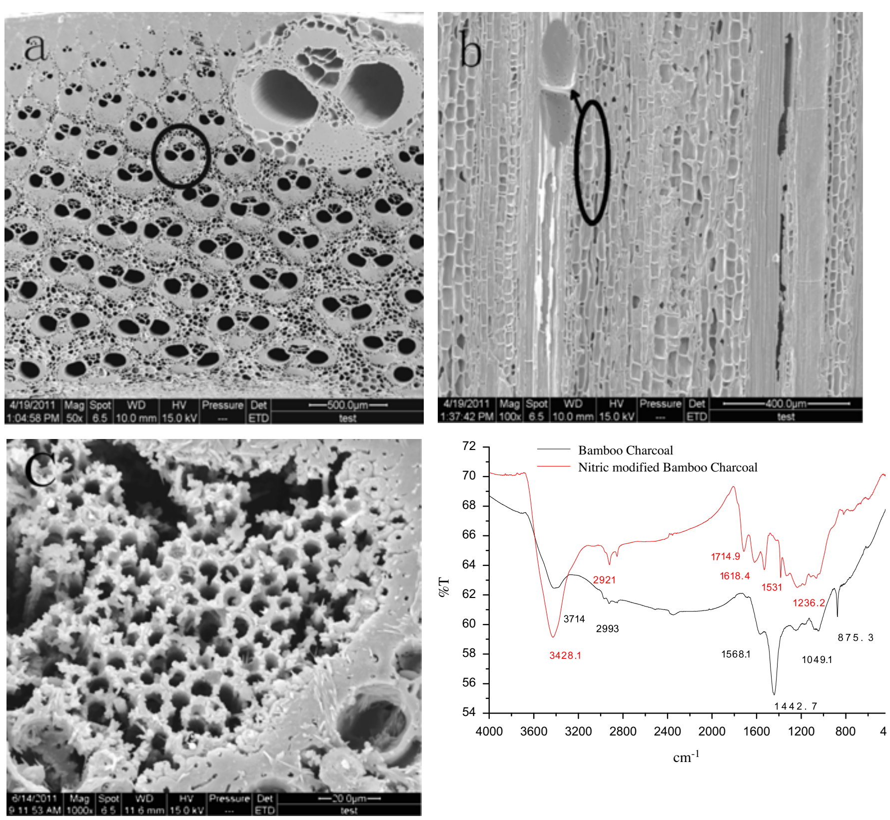

Fig. 2. Scanning electron micrograph and FTIR spectra of modified Bamboo Charcoal (a) ×50; (b) ×100; (c) ×1,000; (d) FTIR.

3. Results and discussion

3.1. Characterization of Bamboo Charcoal

3.1.1. FE-SEM microscope and FTIR spectroscopy

Bamboo Charcoal that has been modified by nitric acid was observed under a SEM as shown in Fig. 2. It can see that Bamboo Charcoal has a relatively smooth surface without large defects. Transverse sections of Bamboo Charcoal are mainly like the Chinese character "pin Structure" Fig. 2(a), Bamboo Charcoal particles were mainly composed of mesopores (2–50 nm), a small portion of macropores and micropores, in which the proportions of mesopores are large, and are regular strip tubes in longitudinal section Fig. 2(b). However, Nitric acid-modified Bamboo Charcoal has some large cavities in the range of 5–20 nm Fig. 2(c). The large pores created by the chemical treatment promote the activation in the internal surface of the carbon particles. Therefore, nitric acid was proved to be an effective activating agent for the production of high-surface area modified Bamboo Charcoal.

Bamboo Charcoal particles are hydrophobic and stay in aggregated forms in aqueous environments, which makes the adsorption of hydrophilic contaminants difficult. Oxidative treatment of Bamboo Charcoal particles is one effective way to increase the hydrophilicity due to the generation of carboxylic acid groups (as well as other minor changes such as hydroxyl groups) on the particles' surface [25].

Nitric acid, a strong oxidizing agent, can attack some imperfect areas of Bamboo Charcoal particles during refluxing, which leads to the formation of carboxylic acid groups (–COOH) at these surfaces. This result was similar to that obtained from the modification of carbon black with nitric acid [26].

Comparing the FTIR spectra of Bamboo Charcoal and Nitric acid-modified Bamboo Charcoal revealed the likely presence of ν(C=O) ($\sim$1,714.9 and $\sim$1,531 cm^{-1}), as well as other features typical of oxidation including ν(O–H) at 3,428.1 cm^{-1}, C=C at $\sim$1,618.4 cm^{-1} and C–O at $\sim$1,236.2 cm^{-1}, in the modified Bamboo Charcoal particles Fig. 2(d). These results support the fact that nitric acid, used as an oxidizing agent, had oxidized some areas of Bamboo Charcoal particles during refluxing leading to the formation of carboxylic acid groups (and other moieties) on the surface of Bamboo Charcoal particles.

3.1.2. Specific surface area, pore structural characterization and oxygen-containing functional groups of Bamboo Charcoal

Specific surface area (*S*), Pore volume (*V*)P and functional groups containing oxygen (*G*) of Bamboo Charcoal particles modified by nitric acid are higher than in Bamboo Charcoal significantly (Table 3). It shows that a certain part of micropores and previously blocked pores are expanding or opening due to oxidation, which makes specific surface area (*S*) to increase. Ammonium can enter the expanded pores where the diameter of the pore is bigger than the diameter of ammonium. Meanwhile, the surface of partial pores was burnt out by nitric acid, which led to incomplete structures. The surface defects of Bamboo Charcoal can induce the increased surface activity of charcoal, which strengthens the adsorption ability.

Nitric acid oxidation increased the amount of acidic functional groups on Bamboo Charcoal. As the number of acidic functional groups increase, the number of hydrophilic charcoal particles grows more and more, which strengthens the ammonium adsorption ability too.

3.1.3. Influence of contact time

Fig. 3 shows the ammonium uptake by Bamboo Charcoal and modified Bamboo Charcoal as a function of contact time at 5℃. The result reveals that ammonium removal by Bamboo Charcoal was fast in the initial 360 min but the rate was rapidly reduced afterwards and leveled off. This might be due to the fact that initially there were abundant vacant adsorbent sites and the solute gradient was high. With the progress of the adsorption process, due to a decrease in the adsorption sites, the ammonium removal rate by charcoal decreased significantly and approached zero when it reached the equilibrium. Fig. 3 shows that the ammonium uptake equilibrium by Nitric Bamboo

Table 3
Modification effect of Bamboo Charcoal by nitric acid

Sample	Modification methods	S (m^2/g)	V_P (cm^3/g)	G (m mol/g)
200 mesh Bamboo Charcoal	Unmodified	157	0.87	0.13
	30% (v/v) nitric acid modified	860	0.97	6.94

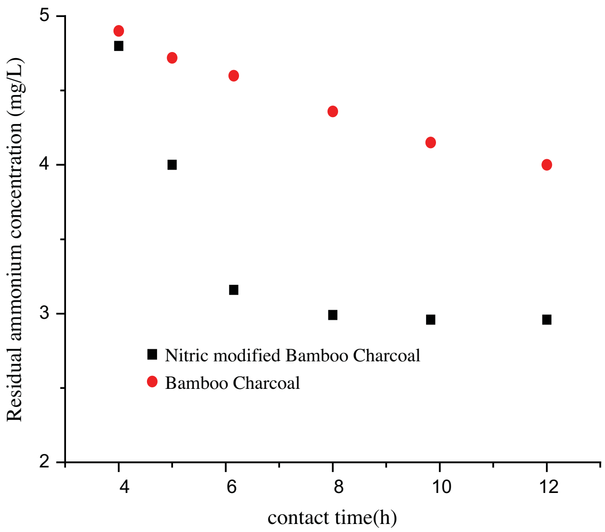

Fig. 3. Effect of contact time on the removal of ammonium (pH: 7; initial ammonium concentration: 5 mg/L; tempe rature: 5℃).

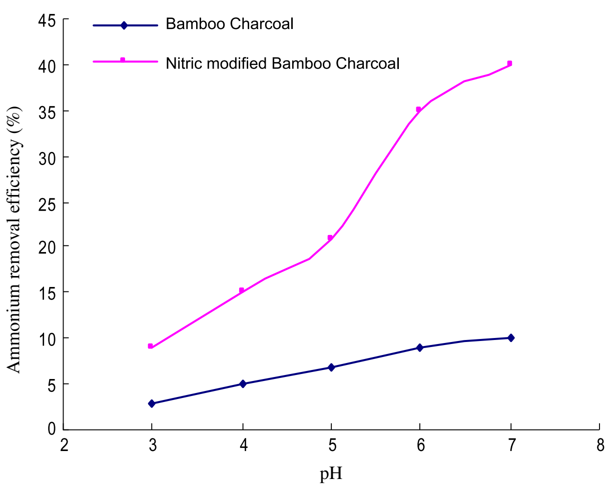

Fig. 4. Effect of pH on the removal of ammonium (adsorbent dosage: 1 g/L; contact time: 360 min; initial ammonium concentration: 5 mg/L; temperature: 5℃).

Charcoal is achieved in a shorter time than by unmodified Bamboo Charcoal. Thus, in the following experiments the equilibrium time was fixed at 360 min.

3.1.4. Influence of pH

pH is an important factor for ammonium adsorption. Ammonium removal by Bamboo Charcoal was observed at pH values ranging between 3.0 and 7.0. The results are depicted in Fig. 4. The figure shows that as the pH of the solution increases gradually, the ammonium removal efficiency of the Nitric

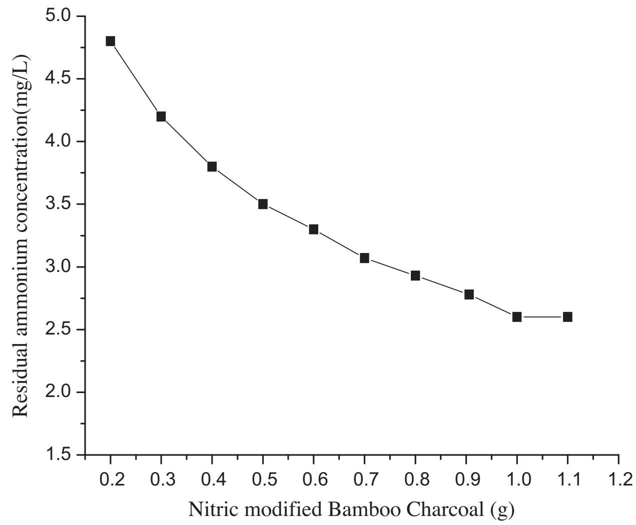

Fig. 5. Effect of adsorbent dosage on the removal of ammonium (contact time: 360 min; pH: 7; initial ammonium concentration: 5 mg/L; temperature: 5℃).

acid-modified Bamboo Charcoal reaches the maximum value (40%) when pH value is 7.0. The result is significantly higher than the removal efficiency of Bamboo Charcoal (10%). At pH values below 7.0, the ammonium concentration in the solution rises when the pH decreases, this nevertheless results in a decline in the removal efficiency, as the hydrogen cation concentration also rises with the decrease in pH and intensifies the competition for adsorption [27].

3.1.5. Influence of absorbent dosage

The results of experiments to determine the effects of adsorbent dosage on ammonium removal are shown in Fig. 5. It reveals that the removal efficiency of ammonium by Nitric acid-modified Bamboo Charcoal increased with an increase in the adsorbent dosage. It rose from 5 to 40% with the increase in the adsorbent dosages ranging from 0.2 to 1.2 g/L. While a rapid increase was observed in the adsorbent dosages ranging between 0.2 and 1.0 g/L, a plateau was seen for those ranging between 1.0 and 1.2 g/L. Increasing the adsorbent dosage above 1 g/L had a negligible effect on the increase in the removal efficiency of ammonium. So the optimum adsorbent dosage of modified charcoal was 1.0 g/L.

3.1.6. Influence of initial ammonium concentration

In order to analyze the effect of the initial ammonium concentration, the following tests were carried out where in the initial ammonium concentration was

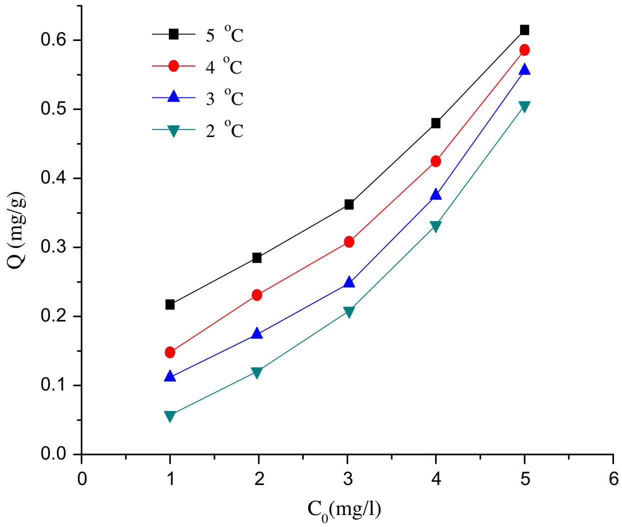

Fig. 6. Effect of initial ammonium concentration on the removal of ammonium (contact time: 360 min; pH: 7; adsorbent dosage: 1 g/L; temperature: 1–5℃).

set at 1–5 mg/L, respectively, temperature was 5℃ and pH of the solution was 7.0. Fig. 6 depicts the ammonium removal efficiency of modified charcoal and unmodified charcoal with five specific initial ammonium concentrations, respectively. It indicates that a high ammonium concentration would add loads of Bamboo Charcoal and increase the ammonium removal efficiency. With increasing concentrations of the solution, the adsorption capacity increases. The initial concentration provided an important driving force to overcome all mass transfer resistance of ammonium between the aqueous and solid phases, hence a higher initial concentration of ammonium may increase the adsorption capacity [28].

At 5℃, when the initial ammonium concentration was from 1 to 5 mg/L, the adsorption capacity of modified Bamboo Charcoal increases from 0.21 to 0.65 mg/g. At an ammonium concentration of 5 mg/L, with the change in temperature from 1 to 5℃, the uptake capacity increased from 0.45 to 0.65 mg/g. It indicated that the initial ammonium concentration was a vital factor for ammonium removal efficiency.

3.2. Adsorption equilibrium study

Adsorption isotherm models are widely used to describe the adsorption progress and to investigate the mechanisms of adsorption. An equilibrium isotherm expresses the relation between the amounts of adsorbate removed from solution at equilibrium by unit of mass of adsorbent and the adsorbate concentration remaining in solution. In this work, equilibrium data were simulated and analyzed by Langmuir and Freundlich.

The Langmuir and Freundlich parameters for the adsorption of ammonium onto modified Bamboo Charcoal are listed in Table 4. It is evident that the adsorption of ammonium onto modified Bamboo Charcoal is fitted better into the Langmuir isotherm model than into the Freundlich isotherm models, as indicated by the R^2 values and the adsorption capacity values in Table 4. Calculations from the Langmuir isotherm equation define the maximum capacity of the adsorbent for ammonium. Modified Bamboo Charcoal shows a higher adsorption capacity than unmodified charcoal and the maximum adsorption capacity of ammonium is 0.651 mg/g at 5℃. The adsorption capacity increases with an increase in the temperature.

4. Conclusions

The present work has showed that Nitric acid-modified Bamboo Charcoal could be considered as a promising material to remove low concentrations of ammonium even in low temperature water. The main mechanisms involved in the removal of ammonium are physical or chemical adsorption onto charcoal porous medium. Moreover, it appears that hydroxyl and carboxyl groups are the predominant contributors to

Table 4
Langmuir and Freundlich isotherms parameters for the adsorption of ammonium by modified Bamboo Charcoal

Equilibrium model	Parameter	Value			
		2℃	3℃	4℃	5℃
Langmuir isotherm	q_m (mg/g)	0.5627	0.6254	0.6104	0.651
	K_L (L/mg)	0.3553	0.4266	0.580	0.6656
	R^2	0.9881	0.996	0.9985	0.997
Freundlich isotherm	K_F (mg/g)	0.1664	0.2121	0.2543	0.2935
	N	0.477	0.4301	0.3568	0.3291
	R^2	0.9592	0.9616	0.9593	0.9498

the complexation of ammonium. The use of Nitric acid-modified Bamboo Charcoal presents an interesting option for both low temperature drinking water (as a possible physicochemical adsorbent in pretreatment process) and wastewater treatment (as a filter media or immobilized microorganisms supporter).

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