ORIGINAL ARTICLE

Influence of outside environmental variations on ammonia nitrogen adsorption characteristics of HVMT/PC/EPDM composite

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Abstract A new kind of ammonia nitrogen (AN) sewage water treatment composite was prepared by melt blending, with synthetic rubber ethylene propylene diene monomer (EPDM) and polycarbonate (PC) as the matrix, the natural vermiculite (VMT) powder modified by hydrochloric acid solution (HVMT) as the filler. By Fourier transform infrared (FTIR) measurement and XRD measurement, it was confirmed that the HVMT was more suitable as filler than VMT. By considering the influence of pH, temperature, adsorption composite content and initial mass concentration on removal rate and adsorption capacity, it can be concluded that the removal rate increased first and then decreased with the increasing pH. "Room temperature" was the optimum temperature for the composite. The removal rate of AN increased with the increasing adsorption composite content. When the adsorption reached equilibrium, the removal rate remained unchanged; the adsorption capacity of AN increased with increasing initial concentration. The removal rate increased gradually with lower initial concentration of AN. When the initial concentration increased to a certain level, the removal rate started to decline. When the initial concentration and composite content showed adsorption equilibrium, the adsorption capacity remained unchanged. The composite can be recycled to be reused after regeneration for nine cycles; the AN adsorption capacity of composite was decreased by only 4.5 %. If the composite can be prepared in large-scale production lines, the costs of HVMT/PC/EPDM porous composite can be controlled at less than USD 16/kg.

Introduction

Nitrogen is a nutrient essential to all forms of life as a basic building block of plant and animal proteins (Schuur and Matson 2001). Although it is an essential nutrient for living organisms, too much of it can be toxic (Vairavan et al. 2007). The presence of nitrogen excess in the environment has caused serious distortions of the natural nutrient cycle between the living world and the soil, water, and atmosphere. Nitrogen in the form of nitrous oxide, nitric oxide, nitrate, nitrite or ammonia is soluble in water and can end up in groundwater and drinking water (Olav et al. 2005). One of the best documented and best understood consequences of human alterations of the nitrogen cycle is the eutrophication of estuaries and coastal seas (Martin et al. 2011).

The contamination of aquatic bodies by industrial effluents rich in ammonia nitrogen (AN) has received much attention in the last decades. There are many sources of ammonia in water, in addition to a large number of domestic sewage (Ribeiro and Araújo 2002), agricultural fertilizers and animal waste (Hellsten et al. 2007; Lahav et al. 2008). The industrial waste such as oil refining waste, pharmaceutical wastewater and landfill leachate (Liu et al. 2011a, b; Vymazal and Kröpfelová 2009; Bloxham et al. 1999) also contains a lot of AN. The AN pollution of water bodies not only brings a greater human health threat, but

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also causes immeasurable loss of traditional water purification process and water quality. Therefore, the lower concentration of AN in water is urgent and important.

Currently, the AN removal processes from water are biochemical methods (Zhang et al. 2009): air stripping (Lei et al. 2010), breakpoint chlorination method and the adsorption (Selby et al. 1996; Wei et al. 2010). Biochemical methods are influenced by water temperature greatly; the removal efficiency is poor when the temperature is low in winter. For air stripping, the operating temperature must be above 0 °C; otherwise, the ammonia stripping will not work. Breakpoint chlorination method will greatly increase the dosage of chlorine, the cost of water, and the disinfection by-product contents in water will greatly increase. Moreover, many small-to-medium sized industries do not have the economic and technical capacity needed to purify appropriately the waste waters produced in their activities. For these reasons, in recent years, many researchers studied the behaviour of natural materials having high adsorption capacity and which are particularly abundant and inexpensive to use them as low-cost effective sorbents for the removal of AN from various effluents.

It is well known, vermiculite (VMT) has high cation exchange capacity due to the substitution of its main cations, namely Si^{4+} and Al^{3+}, with cations having lower valency, such as Al^{3+} instead of Si^{4+} and Mg^{2+} instead of Al^{3+} (Jiménez de Haro et al. 2003). The extent and location (i.e. tetrahedral or octahedral sheets) of these substitutions give rise to VMT with unique physicochemical properties. In VMT, most of the cation substitutions take place in tetrahedral sheets. Owing to the excellent cation exchange ability, VMT is often used to treat wastewater containing ammonium (Pérez Maqueda et al. 2003), heavy metal ions or organic substance (Froehner et al. 2009), and to make antivirus materials. Many authors reported their researches about the application of VMT.

For the application of VMT, granular VMT can often be directly applied, while the powder VMT is not. However, the insufficiency of granular VMT is that adsorption capacity of granular VMT is much less than that of powder VMT; the effective use time of granular VMT is short. Therefore, the granular VMT has a big problem in practice.

Aimed at the powder VMT using in the water, there is an outstanding issue. For the smaller particle size of VMT, its adsorption capacity is higher and a better treatment effect for the AN sewage water. Therefore, the adsorption capacity of powder VMT was better than granular VMT. However, when the powder VMT was put into the water, its particle size was so small that it washed away, and water quality will be affected (Abusafa and Yucel 2002). Therefore, whether to use granular VMT alone, or the separate use of powder VMT poses a variety of problems.

To overcome these problems, a new kind of AN sewage water treatment composite was prepared by melt blending, with synthetic rubber ethylene propylene diene monomer (EPDM) and polycarbonate (PC) as the matrixes, with modified VMT powder (HVMT) as the filler. To make the composite applied to be more environmental, the mechanical properties of the composite should be raised; therefore, a two-component matrix is adopted in the preparation of composite. PC as one of the matrixes is hydrophilic. During the preparation, addition of the appropriate amount of pore forming agent produced many passages to allow water molecules conveniently into the composite. The AN in water can be adsorbed into the composite being encompassed by a cross-linking network. Because the density of the composite was lower than the density of the water, it can float on the water, for which the composite was easily used. The composite is an ecological environment material and it has a good adsorption capacity of AN in the water.

Experimental

Materials

In this study, EPDM (ethylene content 69 %, the third monomer is ethylidene norbornene) (3720, Dupont Co. America) and PC (3103, Bayer Co. Germany) were used as the composite matrix. Dicumyl peroxide (DCP) (Shanghai Chemical Co., China) was used as the cross-linking agent. Polyethylene glycol (PEG) (molecular weight: 2,000, Tianjin Chemicals Co., import sub package, China) was used as the leavening agent. AN standard stock solution (TA-0017, 1,000 mg/l, Beijing Chemicals Co., China) were used to configure the analog sewage. VMT (Lingshou Hebei Province, China), the elemental composition of the clay was determined by the inductively coupled plasma atomic emission spectrometry (ICP-AES) after solubilization

Table 1 Characterization of organic VMT

Element	Content (%)
SiO_2	44.87
Al_2O_3	16.46
MgO	16.41
MnO	0.13
K_2O	3.61
Fe_2O_3	13.50
TiO_2	0.82
CaO	4.20
Na_2O	0.24
P_2O_5	0.06

by acid digestion in a microwave oven and is reported in Table 1. It is possible to suppose that potassium is the principal exchangeable ion present in the interlayer of this clay, as confirmed by its high percentage in the chemical composition of VMT, besides possible coordinating cations (Si, Al, Fe, and Mg).

Pre-treatment and surface modification of VMT

Pre-treatment of VMT

As a pre-treatment, VMT was cleaned with de-ionized water and then dried, and it was crushed and sieved into 200 meshes.

Surface modification of VMT (HVMT)

Surface modification of VMT was prepared by pumping 5 g pre-treated VMT into 100 ml of 1 mol/l hydrochloric acid solution; the reaction medium was maintained under stirring for 2 h at 83 °C, washed and dried in an oven at 60 °C for 24 h, and stored in a desiccator.

Preparation of HVMT/PC/EPDM composites

Compounding and melting of sample

EPDM rubber and PC were melted by blending on a closed mill (Ningbo Rubber and Plastic Machinery Factory, China) for 5 min and then a small amount of HVMT, PEG-2000 and DCP were added. These components were mixed continuously to apparent homogeneity. The temperature was 220 °C. Compounded rubber was taken out and masticated on an open mill (Wuxi No. 1 Rubber Machinery Factory, China) (160 mm, linear rate of front roll 10.97 m/min, linear rate of front roll to rear roll 1/1.35) for 10 min and stabilized to remove the strain overnight (24 h, if possible) before being vulcanized.

Vulcanization

The mixed compound was placed in $80 \times 60 \times 2$ mm^3 molds, preheated for 5 min, and cured for 14 min on the plate rheometer (Jilin University Science Instrument Factory, China) to obtain a closed-cell rubber sheet. After the press was over, the mold was taken out and the composite was prepared.

Preparation of simulated AN wastewater

A solution of 50 mg/l of AN was prepared by dissolving 50 ml of AN standard stock solution in 1,000 ml of distilled water. The pH values of the solutions were kept neutral and standing 1 h before used.

Preparation of simulated AN wastewater in different pH

A solution of 100 ml simulated AN wastewater was configured; then the wastewater was adjusted at different pH with 1 mol/l H_2SO_4 and 2 mol/l NaOH. The range of pH investigated was 2.0–9.0. Eight replicates were performed in a 250 ml grinding mouth flask with stopper for each experiment. The initial concentration in each flask was all 50 mg/l and 1 g composite was added into each flask.

Preparation of simulated AN wastewater in different temperatures

A solution of 100 ml simulated AN wastewater was configured; then the pH of wastewater was adjusted at 7.3; the range of temperature investigated was 10–50 °C. Five replicates were performed in a 250-ml grinding mouth flask with stopper for each experiment. The initial concentration in each flask was all 50 mg/l and 1 g composite was added into each flask.

Preparation of simulated AN wastewater in different composite content

In this study, the pH of wastewater was adjusted at 7.3; the temperature was 20 °C (room temperature). Ten replicates were performed in a 250-ml grinding mouth flask with stopper for each experiment. The 100 ml (50 mg/l) simulated AN wastewater was in each flask. Different qualities of composites were added into flasks (No. 1–No. 9), as shown in Table 2.

Preparation of simulated AN wastewater in different initial mass concentration

In the study, the 100 ml of simulated AN wastewater was configured; the pH of wastewater was adjusted at 7.3; and the temperature was 20 °C (room temperature). Twenty replicates were performed in a 250 ml grinding mouth flask with stopper for each experiment. The initial concentration

Table 2 Adsorption composite content of each experiment

No.	1	2	3	4	5	6	7	8	9	10	11	12
Composite contents (g)	0.1	0.2	0.3	0.4	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0

of AN was from 0.1 to 1,500 mg/l and 1 g composite was added into each flask.

Regeneration of composite

In the study, 25 % NaCl solution was the regeneration solution and 1:10 the ratio of solid to liquid. The 100 ml and 50 mg/l simulated AN wastewater was configured, The pH of wastewater was adjusted at 7.3, and the temperature was 20 °C (room temperature). Nine replicates were performed in a 250 ml grinding mouth flask with stopper for each experiment. After the regeneration was completed, different regeneration time of composite was added into flasks, and the adsorption capacity of composite was tested.

Instrumental techniques

Removal rate (E) of AN by composites adsorption

The equilibrium mass concentration of the AN in water is C_e (mg/l). The initial mass concentration of the AN in water is C_0.

$$E = \frac{C_0 - C_e}{C_0} \times 100\,\%. \tag{1}$$

XRD

X-ray diffraction measurements were conducted using the standard powder diffraction procedure. Adsorbents were ground with methanol in a small agate mortar. The mixture was smear mounted onto the zero-background quartz window of a Phillips specimen holder and allowed to air dry. Samples were analyzed by Cu Kα radiation generated in a Phillips XRG 300 X-ray diffractometer. A quartz standard slide was run to check for instrument wandering and to obtain accurate location of 2θ peaks.

SEM

The micrographs were obtained by a KYKY-2800B (China) scanning electron microscope. Scanning electron microscopy was performed on a DSM 940 cold field emission instrument. The accelerating voltage was 3,000 V. Scanning was performed in situ on the samples.

FTIR

Fourier transform infrared (FTIR) spectroscopy was carried out using an IR T11-IR200 spectrometer using the attenuated total reflectance method (ATR). The spectrum was collected 16 times and corrected for the background noise. The experiments were done on the samples, with KBr addition.

Results and discussion

Evidences for HVMT

FTIR measurement

Figure 1 showed the spectrum of pristine VMT (line a) and HVMT (line b). It was evidenced that acid treatment led to some changes in FTIR spectrum compared to original VMT: the peaks at 682 cm^{-1} can be ascribed to R–O–Si vibration in line a. The peak was weak in line b; the peaks at 463 cm^{-1} can be ascribed to Si–O–Si vibration in line a. The peak was sharp and strong in line b; the peak at 3,437 cm^{-1} is assigned to Si–OH vibration in the spectrum of pristine VMT. However, the peak was strong and clear by modified acid, and the peak moved to 3,422 cm^{-1}. In the attack of H$^+$, Si–O–Al/Fe/Mg bond was breaking, octahedral cation (R–O–Si) was dissolved and oxygen atoms in the top of the silicon–oxygen tetrahedron were transformed into a new Si–OH. The difference in chemical structure between HVMT and VMT was: the exchangeable cations in the interlayer decreased, and the number of Si–OH in HVMT surface increased.

XRD measurement

The XRD patterns for the original VMT and HVMT are shown in Fig. 2. After modification, the diffraction peaks of mica impurity were reduced and lowered significantly; however, the characteristic peak intensity changed little for VMT. According to the Bragg equation, it can be seen from Fig. 2 that the interlayer spacings (d001) of original VMT and HVMT were 1.506 and 1.372 nm. Based on this fact, it can be concluded that the basal spacing was reduced
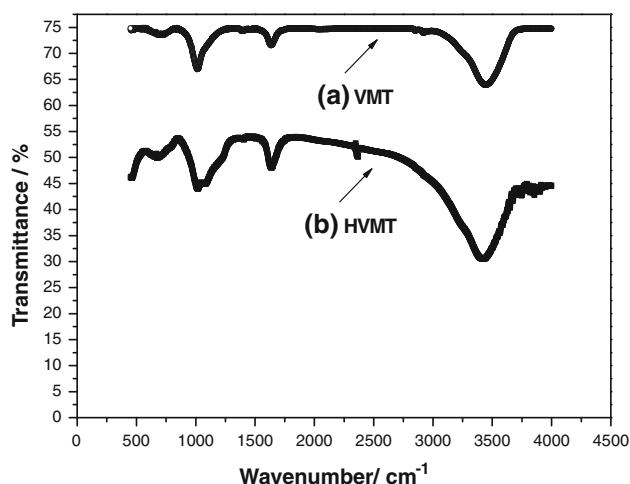
Fig. 1 FTIR spectra of **a** VMT and **b** HVMT
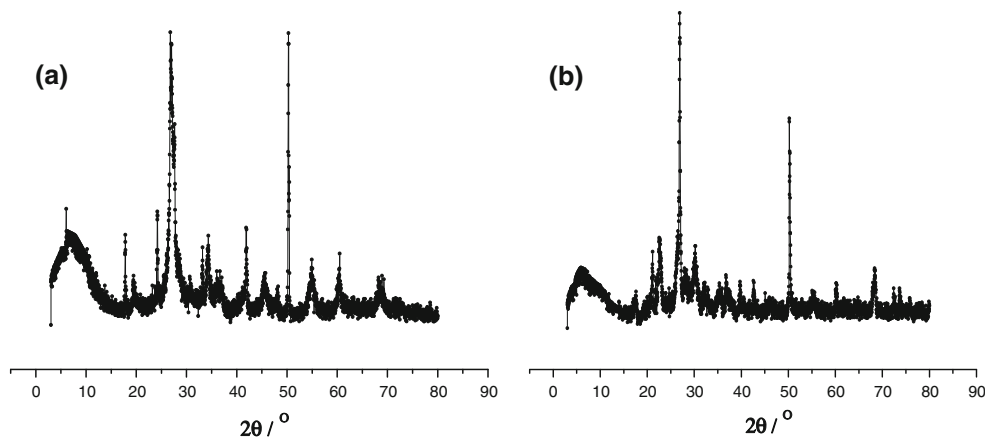
Fig. 2 XRD powder patters for **a** original VMT and **b** HVMT

Table 3 BET values of VMT and HVMT

| Material category	VMT	HVMT
BET values (m^2/g)	67. 07	83. 34

because a small amount of octahedral cations (R–O–Si, R = Al, Mg, Fe) are leaching, and there is a slight collapse in the layered structure. After the VMT was modified by hydrochloric acid, a small amount of aluminum–oxygen octahedron cation and oxygen–silicon tetrahedron cation was dissolved. Therefore, the aluminum–oxygen octahedron wafer and oxygen–silicon tetrahedron wafer were broken. VMT interior showed porous characteristics, and the impurities in the pore were dissolved. After hydrochloric acid treatment, the VMT and HVMT specific surface areas were measured by BET tester as shown in Table 3. The specific surface area of HVMT increased more than that of VMT.

Influence of pH on removal rate

To study the effect of pH on AN removal rate for composite, the simulated AN wastewater in different pH was configured. The AN concentration was measured after adsorption equilibrium and filtration, and the removal rate was calculated. The results were shown in Fig. 3; the removal rate of AN increased with increasing pH (in the range of 2.0–5.0). However, the removal rate of AN decreased with increasing pH (in the range of 5.0–9.0).

Influence of temperature on adsorption capacity

In the study of temperature, the simulated AN wastewater in different temperatures was configured; the AN concentration was measured after adsorption equilibrium and filtration; and the adsorption capacity was tested (Automatic
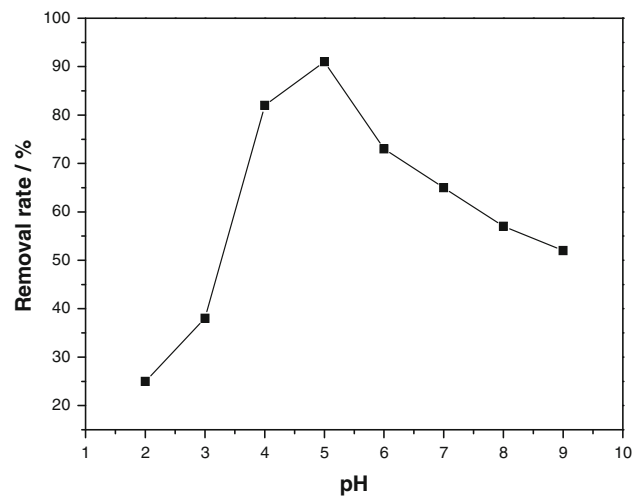

Fig. 3 Effect of pH on AN removal rate for composite

Smart Chemical Analyzer, Italy). The adsorption capacity of composite is influenced greatly by temperature. The results obtained by studying the effect of temperature on the AN adsorption capacity are shown in Fig. 4.

Overall, from the experimental results reported in Fig. 4, adsorption capacity of AN decreased with increasing temperature. At a lower temperature range (10–20 °C), the adsorption capacity of AN decreased rapidly with increasing temperature; however, when the temperature increased to 20 °C, the adsorption capacity of AN decreased slowly with increasing temperature. According to the "Balance Motion" principle, increasing temperature was against the adsorption.

Influence of adsorption composite content on removal rate

The results obtained by studying the effects of composite content were shown in Fig. 5. In this study, the simulated
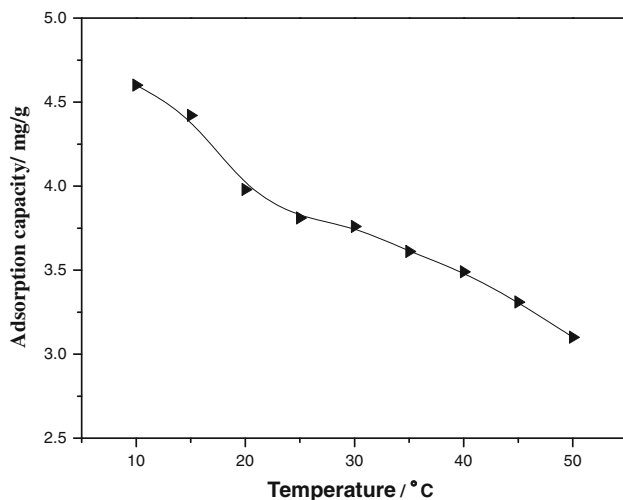
Fig. 4 Adsorption capacity of AN on composite as a function of temperature

AN wastewater was configured according to the foregoing requirements. The AN concentration was measured after adsorption equilibrium and filtration, and the AN removal rate was calculated. The removal rate of AN increased with increasing adsorption composite content. When the adsorption composite content was 2 g, the removal rate would tend toward equilibrium. Therefore, the removal rate did not increase with the adsorption composite content continuing to increase.

Influence of initial mass concentration on removal rate and adsorption capacity

In the study of initial mass concentration, the simulated AN wastewater was configured as the foregoing requirements; when the adsorption reached equilibrium, the removal rate
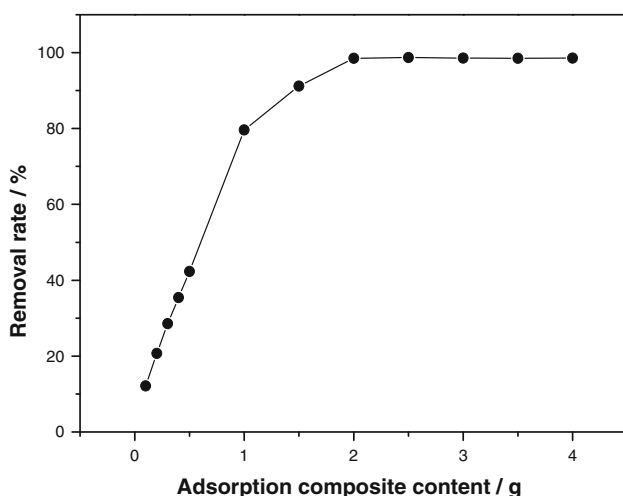
Fig. 5 Effect of adsorption composite content on removal rate

and adsorption capacity were calculated as shown in Table 4.

As can be seen from Table 4, in a variety of different initial concentrations of AN, the composite can effectively reduce the concentration of AN in the solution. The adsorption capacity of AN increased with increasing initial concentration; as for the removal rate of AN, the removal rate increased gradually with lower initial concentration of AN. When the initial concentration increased to a certain level, the removal rate started to decline. When the initial concentration and composite content showed adsorption equilibrium, the adsorption capacity remained unchanged.

For an analysis of the reasons, when the initial concentration was not high, the adsorption capacity of composite was far less than the amount of saturated adsorption reaching equilibrium. In this case, the composite adsorption potential (adsorption energy, adsorption sites and adsorption states) was not a limiting factor. Therefore, the removal rate increased with lower initial concentration of AN. On the contrary, the composite adsorption potential was a limiting factor with the initial concentration increasing; at this time, the removal rate decreased with increasing the initial concentration. Therefore, the initial concentration of AN should not be too much for this composite; it fits well with the actual initial concentration of AN.

Table 4 Removal rate and adsorption capacity with different initial concentration of AN

Initial concentration of AN (mg/l)	Removal rate (%)	Adsorption capacity (mg/g)
1.0	40.00	0.04
2.0	50.00	0.10
3.0	53.33	0.16
4.0	57.50	0.23
5.0	60.00	0.30
6.0	63.33	0.38
8.0	66.25	0.53
10	73.00	0.73
30	77.67	2.33
50	79.60	3.98
100	83.80	8.38
200	89.00	17.80
300	91.33	27.40
400	93.25	37.90
500	90.66	45.33
600	88.33	52.98
800	76.55	61.24
1,000	68.96	68.96
1,200	61.47	73.76
1,500	52.43	78.64
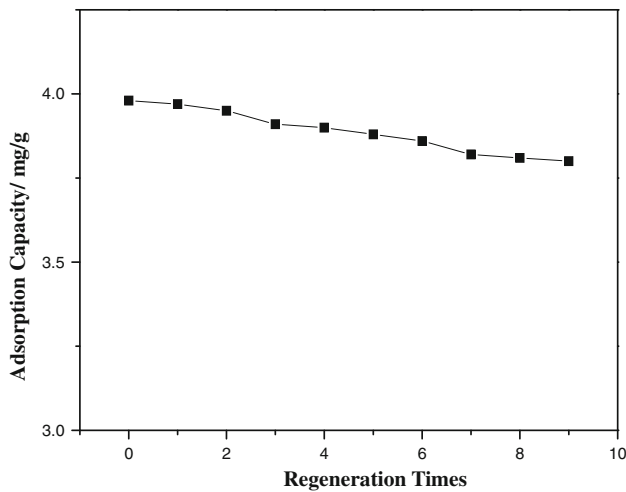
Fig. 6 The relationship between adsorption capacity and regeneration times

Regeneration and cost accounting of composite

There were a large number of Na^+ in the NaCl solution (25 %), and the ion exchange capacity of NH_4^+ was stronger than that of Na^+. Therefore, the NH_4^+ can be easily exchanged by Na^+. In this way, the composite can be regenerated. As shown in Fig. 6, after regeneration for nine cycles, the ammonia nitrogen adsorption capacity of composite decreased by only 4.5 %. This is because Na^+ can exchange all the cation in the composite (including the ion of strong ion exchange capacity, such as K^+); the regeneration process is actually an activation process. After several times regeneration, the adsorption capacity of the composite can be restored, and composite can be recycled to be reused.

To promote the use of HVMT/PC/EPDM porous composite, one of the determining factors was price. In accordance with the best formula of composite, in the laboratory and small-scale preparation of composite, the cost estimates of HVMT/PC/EPDM porous composite material was USD 116/kg. Because the preparation of composite was in the laboratory, the cost of the raw materials are more expensive in small quantities not in bulk; accordingly, there is also an increase in the cost of equipment, power and energy. If this composite can be mass-produced in the factory, the raw material costs can be greatly reduced, and taking into account that the composite can be nine times regenerated and reused. Therefore, the cost of HVMT/PC/EPDM porous composite can be controlled at USD 16/kg.

Conclusions

1. A new kind of AN sewage water treatment composite was prepared by melt blending, with synthetic rubber EPDM and polycarbonate as the matrixes and HVMT powder as the filler.
2. By FTIR and XRD measurements, it was confirmed that the HVMT was more suitable as a filler than the original VMT.
3. The influence of pH on the removal rate showed that the removal rate of AN increased with increasing pH (in the range of 2.0–5.0); the removal rate of AN decreased with increasing pH (in the range of 5.0–9.0).
4. The influence of temperature on the adsorption capacity showed that increasing temperatures were against the adsorption. "Room temperature" is the optimum temperature.
5. The influence of composite content on the removal rate showed that the removal rate of AN increased with increasing composite content. When the adsorption reached equilibrium, the removal rate remained unchanged.
6. By considering the influence of initial mass concentration on removal rate and adsorption capacity, it can be concluded that the adsorption capacity of AN increased with increasing initial concentration; the removal rate increased gradually with lower initial concentration of AN; when the initial concentration was increased to a certain level, the removal rate started to decline. When the initial concentration and composite content showed adsorption equilibrium, the adsorption capacity remained unchanged.
7. After regeneration for nine cycles, the ammonia nitrogen adsorption capacity of composite decreased by only 4.5 %. If the composite was prepared in large-scale production lines, the costs of HVMT/PC/EPDM porous composite could be controlled at less than USD 16/kg.

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