



# Accelerated removal of nitrate from aqueous solution by utilizing polyacrylic anion exchange resin with magnetic separation performance



Haiou Song<sup>a,b,c,d</sup>, Zhijian Yao<sup>a,b,d</sup>, Chendong Shuang<sup>a,b,c,d</sup>, Aimin Li<sup>a,b,c,d,\*</sup>

<sup>a</sup> State Key Laboratory of Pollution Control and Resource Reuse, School of the Environment, Nanjing University, Nanjing 210023, PR China

<sup>b</sup> National Engineering Research Center for Organic Pollution Control and Resources Reuse, Nanjing 210023, PR China

<sup>c</sup> Collaborative Innovation Center of Advanced Technology and Equipment for Water Pollution Control, Nanjing 210023, PR China

<sup>d</sup> Nanjing University & Yancheng Academy of Environmental Protection Technology and Engineering, Yancheng 210009, PR China

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## ABSTRACT

In order to quickly remove nitrate from aqueous solution, a magnetic strong base quaternary ammonium anion exchange resin (MAER) has been successfully designed and synthesized. The physicochemical properties of the MAER as well as its adsorption ones for nitrate removal were investigated in detail. A series of batch experiments were carried out to evaluate the effects of several factors on removal efficiency of nitrate, such as retention time, resin amount and initial nitrate concentration. Compared to the commercial Purolite A300 and D213, the equilibrium time for the adsorption process using MAER was only around 20 min, which is significantly less than that of two ones mentioned. Furthermore, the kinetic process of nitrate sorption on MAER could be well described by both pseudo-first-order and pseudo-second-order models. In addition, the results of batch experiments can be better fitted by the Langmuir and Freundlich adsorption isotherm models. Most importantly, the effects of competing ions on nitrate removal followed the order as:  $\text{SO}_4^{2-} > \text{Cl}^- > \text{HCO}_3^-$ . These results are of significance in guiding the development of novel resins with the rapid nitrate removal rate from aqueous solution, which would improve efficiency and save energy greatly.

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## 1. Introduction

Anion exchange resins have attracted extensive attention in recent years owing to their great potential in environmental pollutant treatment [1–3]. Until now, some commercial resins have been developed and applied in removing nitrate, involving Purolite A300, Amberlite IRA400, Indion NSSR, etc. [4–6]. In the recent reports, aiming to the nitrate removal from water, styrene and acrylic skeleton anion exchange resins have been considered more effective than non-ionic ones and activated carbon [7–9]. However, the lower adsorption efficiency attributed to the longer adsorption balance time limits their further practical application. There exist two main reasons to this phenomenon. One is the difficulty in mixed contactors between the larger size of resin bead

and the separation of smaller beads; the other is the relatively smaller BET surface areas of the resin.

Fortunately, the magnetic resins [10–12] anchoring magnetic iron oxide in the polymer matrix exhibit high settlement rates. Even some of them could be applied to raw water in a slurry form and mixed well with adsorbate by machine stirring [13,14]. So, the resins with magnetic separation performance could be considered as an efficient absorbent to purify polluted water in industry.

Recently, a magnetic anion exchange resins have been used to remove natural organic matter or heavy metals ions, which show the high adsorption capacities [15–17]. However, the corresponding studies on removal nitrite are seldom performed utilizing them.

In this investigation, a novel magnetic base ammonium anion exchange resin (MAER) has been synthesized successfully by optimizing the monomer ratios. Moreover, the physicochemical properties of the MAER have been confirmed by FT-IR, BET, EA, etc. At the same time, the adsorption properties of the MAER for nitrate were determined by the static and dynamic adsorption tests. And the significant effects on removal consequence have been discussed of competitive anions such as  $\text{SO}_4^{2-}$ ,  $\text{Cl}^-$  or  $\text{HCO}_3^-$  in

\* Corresponding author at: State Key Laboratory of Pollution Control and Resource Reuse, School of the Environment, Nanjing University, Nanjing 210023, PR China. Tel.: +86 25 8968 0378; fax: +86 25 8968 0377.

E-mail address: [songhaiou2011@126.com](mailto:songhaiou2011@126.com) (H. Song).

detail. The nitrate would be rapidly removed from the model solutions by using the magnetic anion exchange resin compared to the commercial resins. The favorable results obtained suggest that the magnetic adsorbent has the potential in drinking water supplies treatment.

## 2. Experimental method

### 2.1. Materials

The macroporous acrylic strong anion exchange resin D213 was purchased from Zhengguang Industrial Co. Ltd. (Hangzhou, china). Purolite A300 was provided by Purolite Int. Ltd. The magnetic strong anion exchange resin MAER was prepared by our group. They could be functionalized by reactions with  $-N^+(CH_3)_3$  groups to create quaternary ammonium exchange sites. Two resins were obtained as spherical beads with sizes ranging from 0.3 to 0.9 mm (D213) and 0.05 to 0.12 mm (MAER), respectively. The total anion-exchange capacities of the D213, A300 and MAERs are  $\sim 4.5$ ,  $\sim 3.5$  and  $\sim 4.8$  mmol/g, respectively. Ionic form of three resins is  $Cl^-$ .

### 2.2. Chemicals

Methyl acrylate (MA) ( $\geq 98.5\%$ ) and benzoyl peroxide (BPO) ( $\geq 98.0\%$ ) were purchased from Sinopharm Chemical Reagent Company in China. Iron (III) chloride, iron (II) sulfate, ammonia solution (25 wt%) were purchased from Sinopharm Chemical Reagent Company in China. 200# solvent naphtha (SN200) was obtained from Zhongchao Chemical Co. Ltd. in China. Industrial products of divinylbenzene (DVB) (63.3%),  $\gamma$ -methacryloxypropyl-trimethoxysilane ( $\gamma$ -MPS) ( $\geq 98.0\%$ ), polyvinyl alcohol (PVA) (1788), polyvinyl pyrrolidone (PVP) (K90, BASF company), *N,N*-dimethyl-1,3-propanediamine (DMPDA) ( $\geq 99.0\%$ ), and monochloromethane (MCM) ( $\geq 99.0\%$ ) were all purchased from commercial approach.

All the inorganic chemicals of analytical grade were purchased from Chemical Company in Beijing. Different solutions of  $NO_3^-$ ,  $HCO_3^-$ ,  $Cl^-$  and  $SO_4^{2-}$  were prepared by dissolving the proper amounts of their sodium into de-ionized (DI) water.

### 2.3. Preparation of MAER

In brief, the preparation procedure of MAER includes two steps mainly and illustrated in Scheme 1 according to Ref. [18]. (1)

Polymerization, ammonolysis and alkylation using DVB, MA,  $\gamma$ -MPS modified  $Fe_3O_4$  and SN200; (2) quaternarization with DMPDA and MCM.

Prior to use, all the resins were extracted with ethanol to remove the residual impurities. After filtered, they were rinsed successively with 1 M NaOH and 0.1 M HCl for 3 times and dried under vacuum at 333 K for 8–10 h for the further use.

### 2.4. Batch adsorption experiments

Equilibrium adsorption experiments were carried out in 150 mL glass bottles. To start the experiment, 0.1 g of the given resin was introduced into a 50 mL of solution containing the known nitrate concentration (0.805, 1.61, 3.22, 6.44 and 9.66 mmol/L, respectively). The amount of nitrate uptake was calculated based on the mass balance before and after the tests. The flasks were then transferred to a shaker with a thermostat and shaken under 140 rpm until the adsorption process reached equilibrium. Additional co-ions including  $HCO_3^-$ ,  $Cl^-$  or  $SO_4^{2-}$  were added when by dissolving their sodium into the test solution while necessary. All batch experiments were performed in triplicates under conditions.

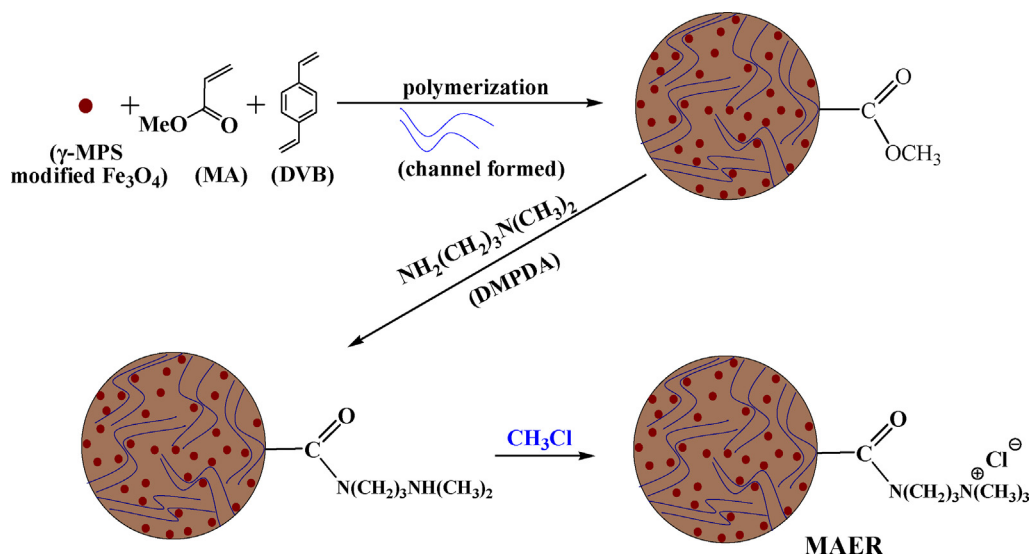
For determination of adsorption kinetics, 1.0 g of resin was introduced to mix with 500 mL of 1.61 mmol/L nitrate in 1.0 L flask and stirred. At various time intervals 1.0 mL solution were sampled for nitrate analysis [19].

### 2.5. Fixed-bed column runs

Fixed-bed adsorption experiments were conducted in a glass column (internal diameter 1.0 cm and length 12 cm) equipped with a water bath to maintain a constant temperature of 293 K. A peristaltic pump was used to ensure a constant flow rate and an automated fraction collector was chosen to collect the effluent sample. The influent nitrate concentration was 1.61 mmol/L. After adsorption, the exhausted resin bed was subjected to regeneration by NaCl [20].

### 2.6. Characterization and analyses

The content of anions in aqueous solution was determined using ion chromatography (Dionex ICS-1000). Vacuum dried MAER was used to measure its carbon, hydrogen and nitrogen content on elemental analyzer (EA, Vario Micro, Elementar, Germany). The



Scheme 1. Preparation of MAER.

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