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Study on the removal of dissolved organic matters in the raw water by a new magnetic anion-exchange resin

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ABSTRACT

The removal of dissolved organic matters (DOM) in the raw water of Tongyu River using a new magnetic anion-exchange resin (NDMP) was investigated. NDMP was more effective than a commercial magnetic ion-exchange resin (MIEX) for the purification of drinking water. NDMP can eliminate 49% of dissolved organic carbon (DOC) and 86% of UV absorbance at 254 nm (UV_{254}) in the raw water within 30 min. During multiple-loading jar tests, DOC of the effluent was 2.1 mg/L at 100 BV and increased to 3.0 mg/L after the loading of 1,200 BV by NDMP treatment. The saturated resin was renewed after regeneration with sodium chloride. NDMP displayed high removal ability for both hydrophobic acid (HPO) and transphilic acid (TPI) compared to MIEX resin, whereas the removal rate of hydrophilic (HPI) was relatively low. The removal rates of HPO, TPI, and HPI in the raw water at the NDMP amount of 10 mL/L were 62, 65, and 24%, respectively. Therefore, the NDMP resin can be used in the purification of drinking water at high DOC level.

Keywords: Ion exchange; Dissolved organic matter; Magnetic anion-exchange resin; Drinking water treatment

1. Introduction

Dissolved organic matter (DOM), consisting of both aromatic and aliphatic hydrocarbon structures, is ubiquitous in natural waters [1]. DOM can cause a series of problems in drinking water, such as color, taste, and odor [2,3]. Besides, DOM can form trihalomethanes and haloacetic acids with chlorine during the disinfection process, which renders it the major precursor of disinfection by-products (DBPs) [4–6]. Thus, advanced treatment processes are needed to remove DOM from the source water for drinking such as coagulation and oxidation [7]. In recent years, many researchers focus on improving current treatment processes and developing novel techniques to enhance the removal of DOM.

Although traditional treatment processes including coagulation, flocculation, and filtration have been widely adopted to remove DOM and turbidity from drinking water, effective methods for DOM removal remain to be developed [8,9]. It has been reported that coagulation can preferentially remove high

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molecular-weight substances and the hydrophobic fraction of organic carbon. The removal of low molecular-weight matter and hydrophilic (HPI) organic carbon fraction was less effective [10–13]. As a consequence, new technologies need to be developed to treat drinking source water with variable dissolved organic carbon (DOC) contents in different areas.

Compared with coagulation, ion-exchange resin can remove DOM more effectively from water. Thus, numerous studies lay an emphasis on the application of ion-exchange resin in drinking water treatment [14]. However, traditional ion-exchange resins can only be used in the fix-bed mode, which is not suitable for raw surface water containing high levels of suspended solids. Additionally, the adsorption rate of DOM is relatively low [15–17]. In recent years, a MIEX resin developed by Orica Watercare has been used in drinking water treatment [18,19]. MIEX is a strong base anion-exchange resin (AER) with macroporous polyacrylic matrix in chloride form. The beads are smaller than conventional AERs, which provides high specific surface area and thereby increases the adsorption capacity. Moreover, the MIEX process can be implemented in a high-rate configuration (fluidized bed process) or a dual-stage configuration consisting of a resin contactor and a resin separator. The magnetic properties of the resin beads facilitate rapid settling in the dual-stage configuration and enable fluidization in the high-rate configuration [20]. Because the backbone of MIEX contains a high proportion of magnetic iron oxide compounds, the fine resin beads can easily agglomerate into larger particles with fast-settling property. Thus, MIEX can remove DOM faster than traditional ion-exchange resins [20]. For instance, the majority of UV-absorbing organics was removed within the first 10–20 min by MIEX [15,21]. In previous studies, MIEX resin treatment is confirmed to be an effective process to remove DOM [22,23]. Depending on the water quality, the removal rates of UV absorbance at 254 nm (UV_{254}) and DOC obtained by the MIEX resin range from 50 to 85% and 40 to 75%, respectively [15,24]. The reduction of DOM after MIEX pretreatment accordingly decreases the consumption of chemicals in the following processes and restrains the formation of DBPs [15,21]. In previous studies, the MIEX resin has been used as a pretreatment method prior to coagulation to enhance the efficiency of coagulation and reduce the coagulant dosage (up to 60%), sludge formation, and turbidity [25–27]. There have been few reports about magnetic ion-exchange resins except MIEX resin. Recently, a novel magnetic ion-exchange resin (named NDMP in this paper) prepared by a new method was developed [28,29]. NDMP resin was prepared by polymerization of methyl acrylate (MA), while the MIEX resin was synthesized using glycidyl methacrylate (GMA) as the monomer [29]. Even though DOM removal by MIEX has been thoroughly studied in drinking water treatment, there is hardly any study to compare the removal performance of MIEX and NDMP.

The present work focused on three aspects: (1) the improvement of DOM removal by a NDMP; (2) comparison of the adsorption behaviors of the NDMP resin and commercial MIEX; (3) comparative studies on the removal of various organic acid fractions by NDMP and MIEX from raw water.

2. Materials and methods

2.1. Water samples and magnetic anion-exchange resins

The raw water sample was collected from Tongyu River, which is the drinking water source for almost 720,000 people of Dafeng City in China. The characteristics of the raw water were analyzed including TOC, DOC, UV_{254}, pH, turbidity, and alkalinity. A portion of the water sample was treated with XAD-series resin fractionation. The raw water samples were stored at 4℃ in the dark until the experiments were conducted. All of the experiments were completed within a four-week period.

Magnetic anion-exchange resins NDMP and MIEX were provided by Nanjing University and Orica Watercare, respectively. Their physical and chemical indexes were listed in Table 1. FI-IR spectra and scanning electron microscopy (SEM) were also used to characterize the resins. Both anion-exchange resins were turned into chloride form before use. The pretreatment procedure of both resins proceeded as follows. A total of 30 mL of resin was filled in a glass column (i.d. × length: 25 mm × 300 mm with a 400 mL reservoir). The resin was rinsed sequentially with 300 mL of 1 mol/L HCl aqueous solution, 300 mL of 1 mol/L NaOH aqueous solution, and 300 mL of distilled water at a flow rate of 5 mL/min. Subsequently, 300 mL of 1 mol/L HCl aqueous solution and 300 mL of NH_3-NH_4Cl aqueous solution (1 mol/L NH_4Cl solution with the pH adjusted to 9.25 ± 0.15 with NH_4OH solution) were successively pumped through the column at a flow rate of 5 mL/min. The resin was then washed with distilled water until the effluent was neutral [29].

The capacities of each resin were determined by experiments. The resin was soaked in $NaNO_3$ aqueous solution (1 mol/L) for 1 h, and then, the capacity was determined by titrating $AgNO_3$ against the released Cl^- in solution. The exchange capacity of resin was represented by the chlorine contents of resin particle

Table 1
Characteristics of the magnetic anion-exchange resins studied

Resin	Types	Pores	Structure	Particle size (μm)	Total exchange capacity (mmol/mL)	Water content (%)
NDMP	Strong base	Macropore	Acrylic	100–180	0.98	66
MIEX	Strong base	Macropore	Acrylic	50–180	0.58	65

per unit volume of the magnetic resin. The calculation formula was as follows: $Q = C/35.45$. The Q (mmol/mL) represents per unit volume of wet resin exchange capacity, and the C (mg/mL) represents the content of chloride ion on the surface of magnetic resin.

2.2. *Effect of time on the removal of DOC and UV_{254}*

A selected dose of either MIEX or NDMP resin was added to two liters of raw water in 2 L square jars. The resin and water were mixed at 150 rpm for 60 min and then allowed to settle for 20 min. The resin treatment procedures were based on a previous research [15]. 20 mL of samples were taken at 2, 5, 10, 20, 30, and 60 min for DOC and UV_{254} analysis. Each sample was filtered through 0.45 μm membrane prior to analysis.

2.3. *Effect of resin dose on the removal of DOC and UV_{254}*

The resin doses in the range of 2.5–15 mL/L were tested. Based on previous study, different resin volumes in the range of 2.5–15 mL were added into one liter of raw water in 1 L square jars, mixed for 30 min at 150 rpm, and then allowed to settle for 20 min. After settlement, 20 mL samples were taken for DOC and UV_{254} analysis. Each sample was filtered through 0.45 μm membrane, and then DOC and UV_{254} were determined.

2.4 *DOM fractionation*

In order to investigate the effect of DOM compositions on its removal rate, DOM in the raw and treated water samples was fractionated into hydrophobic acid (HPO), transphilic acid (TPI), and HPI fractions by XAD-series resins including Amberlite XAD-8 and XAD-4 resins (Rohm and Haas, Philadelphia, PA) [30–32]. The resins were cleaned according to the method reported by Thurman and Malcolm [30]. The charge density (mmol/g) was determined by direct potentiometric titration following published procedures [33]. And the carboxyl acidity is defined as the charge density at pH 8. Firstly, a solution containing different fractions of DOM was purged with nitrogen gas for 30 min to eliminate carbon dioxide and then titrated with 0.04 N NaOH solution under a nitrogen atmosphere. The NaOH solution was added in 0.1 mL increments, and the pH value was recorded after each addition of titrant. All samples were titrated up to approximately pH 11. The charge density of three isolations of DOM was calculated based on the pH measurements and a charge balance of the solution as follows: Charge density (mmol/g) = $([H^+]+[Na^+]-[OH^-])/C(DOM)$(1), where the concentrations of H^+, Na^+, and OH^- are in mmol/L and C (DOM) is the DOC concentration of the solution (g/L) [34,35].

2.5. *Multiple-loading jar tests*

In order to evaluate the performance of magnetic anion-exchange resins in continuously operated processes, multiple-loading tests were performed in a series of successive jar tests with the same resin used for repeated cycles. After the kinetic tests, the resin dose of 10 mL/L, and the mixing time of 30 min were selected for all multiple-loading jar tests. The dose and the contact time were determined based on the minimum mixing time after which the increment of DOC removal rate was lower than 5%. The first step represented the load of 100 BV, that is, the treated water volume is 100 times of the resin volume. After 30 min of mixing and 20 min of settling, samples were taken from the supernatant and filtered for DOC and UV_{254} measurements. For all subsequent loading steps, the beaker containing 10 mL of settled NDMP resin was mixed again with 1L of raw water for 30 min. The same procedure was repeated until a load of 2,000 BV was achieved after 20 loading steps.

2.6. *Regeneration of the magnetic anion-exchange resins*

In order to investigate the regeneration performance of magnetic anion-exchange resins in continuous operating processes, the same magnetic resins used during batch experiments were regenerated each time after each loading step. The resins were regenerated according to a previous work [7]. After jar tests, the resins were rinsed with deionized (DI) water. Sodium chloride solution at a concentration of 15 wt%

(≈3 M) was used as the regeneration agent. The amount of the regeneration solution was determined to be the volume of sodium chloride solution that contained 25 times more chloride than was theoretically available on the resin. For instance, 10 mL/L of NDMP-Cl resin has an ion capacity of 9.8 mmol/L; therefore, the regeneration solution should contain 245 mmol/L Cl^-. The regeneration procedure was conducted by regulating the ratio of the volume of regeneration solution to the volume of resin. The resin was mixed with the calculated amount of regeneration solution on a stir plate for 20 min and allowed to settle for 20 min before decanting the brine. Afterward, the resins were rinsed with DI water, mixed for 10 min, settled for 10 min, and decanted. The operation mentioned above was repeated for two times. The first step represented the load of 100 BV. For all subsequent loading steps, the beaker containing 10 mL of settled resins after regeneration was mixed again with 1 L of raw water for 30 min. The same procedure was repeated until a load of 2,000 BV was achieved after 20 loading steps.

2.7. Analytical methods

TOC was measured using a TOC analyzer (TOC-V CSH, Shimadzu, Japan). DOC was defined as the organic carbon concentration of a sample after filtering through a 0.45-μm membrane filter (Millipore, USA). UV_{254} was measured using a UV spectrophotometry (UV-1800, Shimadzu, Japan). The reported value was the average of triple values with relative percent differences of <5% between triple samples and calibration check standards. Turbidity was determined on a turbidity meter (AQ3010, Thermo Fisher Scientific, USA). The DOC concentration and UV absorbance of raw water were measured as soon as the samples were taken.

3. Results and discussion

3.1. Characteristics of raw water and magnetic anion-exchange resins

The concentration and characteristics of organic compounds in the raw water were determined by TOC, DOC, UV_{254}, and specific UV absorbance ($SUVA_{254}$ [L/mg m] = UV_{254} [cm^{-1}] /DOC [mg/L] × 100). The raw water had high DOC content ranging from 3.8 to 4.2 mg/L and high $SUVA_{254}$ ranging from 3.2 to 3.7 L/(mg m). UV_{254} ranged from 0.10 to 0.14 cm^{-1}, which reflects the presence of unsaturated double bonds and aromatic compounds. Moreover, the turbidity and alkalinity of the raw water were as high as 20–40 NTU and 100–150 mg/L as $CaCO_3$, respectively.

Both MIEX and NDMP resins are strong base anion-exchange resin with a macroporous polyacrylic matrix in chloride form. The diameters of both resin beads were around 100 μm, which are about 3–8 times smaller than conventional resins. Meanwhile, the NDMP resin had higher exchange capacity of 0.98 mmol/mL than the MIEX resin of 0.58 mmol/mL. FT-IR spectra of NDMP and MIEX resins were shown in Fig. 1. The peak at 1,653 cm^{-1} and peak at 1,550 cm^{-1} were corresponding to a stretching vibration of the "C=O" bond and a bending vibration of the "N–H" bond, respectively, which demonstrated the structure of the secondary amides of NDMP. The absorption band at 1,722 cm^{-1} was attributed to a stretching vibration of aliphatic group of MIEX. The quaternary amine groups of NDMP and MIEX resins were verified by the absorption bands at 3,026 and 3,017 cm^{-1}, respectively. The absorption band at 3,380 cm^{-1} may be related to –OH group of MIEX, while the absorption band at 3,425 cm^{-1} might be attributed to a stretching vibration of the N–H bond of the secondary amine group of NDMP [36].

The surface of the beads of NDMP resin appears to be rougher than that of MIEX resin which characterized by SEM photos (Fig. 2). This could be explained by the presence of irregular nano-sized Fe_3O_4 and needle-shaped γ-Fe_2O_3 used in the process of preparation of NDMP and MIEX resin, respectively [36].

3.2. Effect of time on the removal of DOM

For MIEX and NDMP resins, both DOC and UV absorbance in the aqueous phase witnessed a rapid decrease with time, and almost reached the lowest

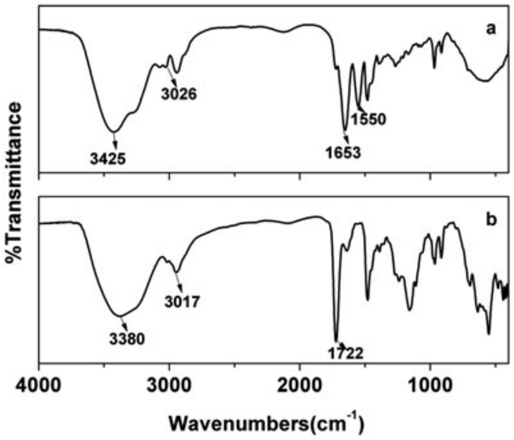

Fig. 1. FT-IR spectra of (a) NDMP and (b) MIEX resins.

level at about 30 min. The fast kinetics may be ascribed to the smaller bead size of the two resins. These results are consistent with the observation of Singer and Bilyk who reported that the majority of UV_{254} was removed with magnetic anion-exchange resin within the first 20–30 min [15]. The NDMP resin exhibited better affinity to DOC and UV-absorbing compounds than MIEX resin as shown in Fig. 3. The removal rates of DOC and UV-absorbing substance ranged from 34 to 49% and from 67 to 86%, respectively, at the NDMP amount of 10 mL/L and the contact time of 10–30 min. After 30 min of reaction, NDMP could eliminate 49% of DOC and 86% of UV absorbance, whereas MIEX removed 36% of DOC and 78% of UV absorbance. This could be explained by the higher exchange capacity of NDMP (0.98 mmol/mL) than that of MIEX (0.58 mmol/mL). As a result, the NDMP resin is more effective than the MIEX resin for the removal of DOM in the raw water.

3.3. Effect of resin dose on the removal of DOM

The effect of NDMP and MIEX dose on the removal of DOM (as measured by DOC and UV-absorbing organics) in raw water is shown in Fig. 4. When the resin amount was elevated from 2.5 to 15 mL (settled resin)/L with the contact time of 30 min, the removal rates for the NDMP resin increased from 25 to 50% for DOC and improved from 54 to 87% for UV absorbance. The DOC content of raw water decreased from 4 to 2 mg/L after 30 min of contact time at the NDMP resin dose of 10 mL/L. By comparison, the MIEX resin demonstrated weaker affinity to DOM with the reduction of DOC and UV absorbance ranged from 19 to 36% and 52 to 79%, separately. These results indicated that the NDMP resin could effectively remove NOM and was more effective than the MIEX resin in water treatment. The highest removal rate of DOM was obtained at the resin dose of 10 mL/L for both resins. The curves revealed that the removal rates of UV absorbance and DOC were positively correlated. This suggests that NDMP can remove a wide range of dissolved organic compounds including UV-absorbing organic matters [10].

3.4. Multiple-loading jar tests

According to the results of multiple-loading jar tests shown in Fig. 5, the DOC level of treated water was 2.1 mg/L at 100 BV loading for the NDMP resin while the value was 2.6 mg/L for MIEX resin under the same condition. DOC concentrations gradually increased to 3.0 mg/L and 3.6 mg/L for NDMP and MIEX at 1,200 BV loading and remained constant in further loading to 2,000 BV. The removal of DOC by the two resins both decreased by about 1.0 mg/L at the load of 2,000 BV compared to that of fresh resins. The UV absorbance removal by NDMP and MIEX also presented decreases of 0.028 and 0.032 cm^{-1} at the load of 2,000 BV as compared to the fresh resin. The results demonstrated that the NDMP resin is slightly better than MIEX for DOM removal in continuous operation processes.

3.5. Regeneration of ion-exchange resins

In order to investigate the regeneration performance of magnetic anion-exchange resins in continuously operating processes, the same resin was reused many times with regeneration. Fig. 6 illustrates the performance of NDMP and MIEX resins over multiple regeneration cycles for the removal of DOC and UV_{254}. The saturation of the two resins was both relieved when regenerated with sodium chloride solution. At a load of 2,000 BV, DOC concentrations of treated water were 2.7 mg/L and 3.3 mg/L for NDMP

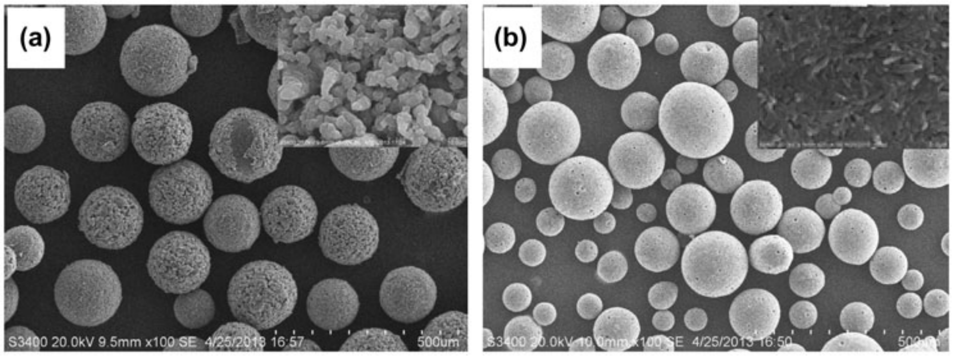

Fig. 2. SEM photos of (a) NDMP and (b) MIEX resins.

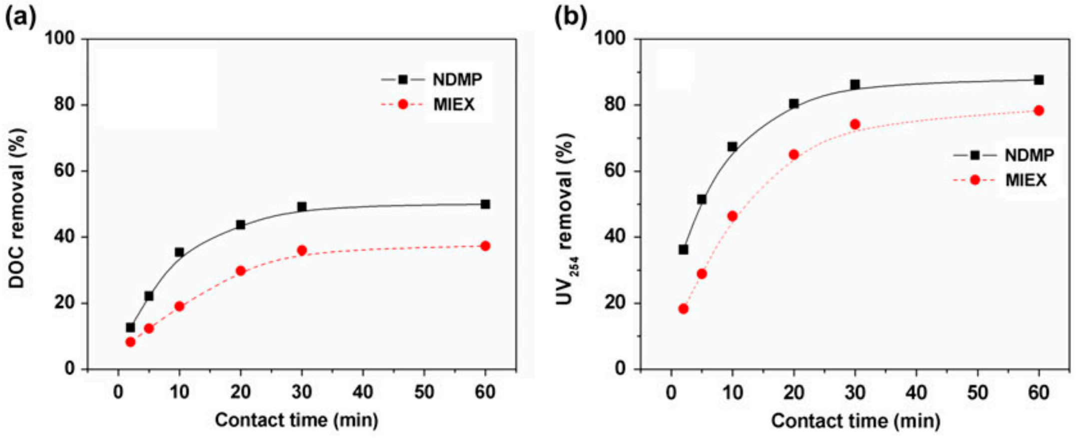

Fig. 3. (a) DOC and (b) UV_{254} absorbance removal by NDMP and MIEX resins with a dosage of 10 mL/L in raw water within 60 min of contact time.

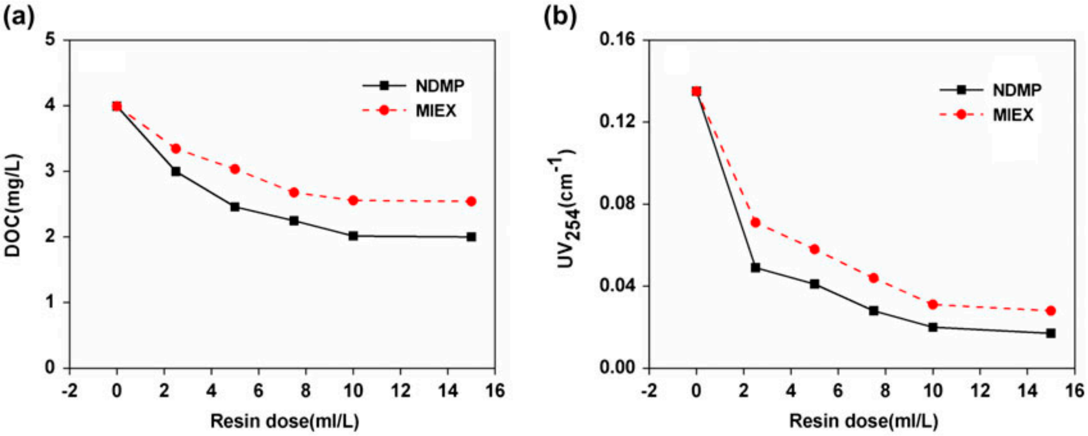

Fig. 4. Illustrative impact of NDMP and MIEX treatment on (a) DOC and (b) UV_{254}.

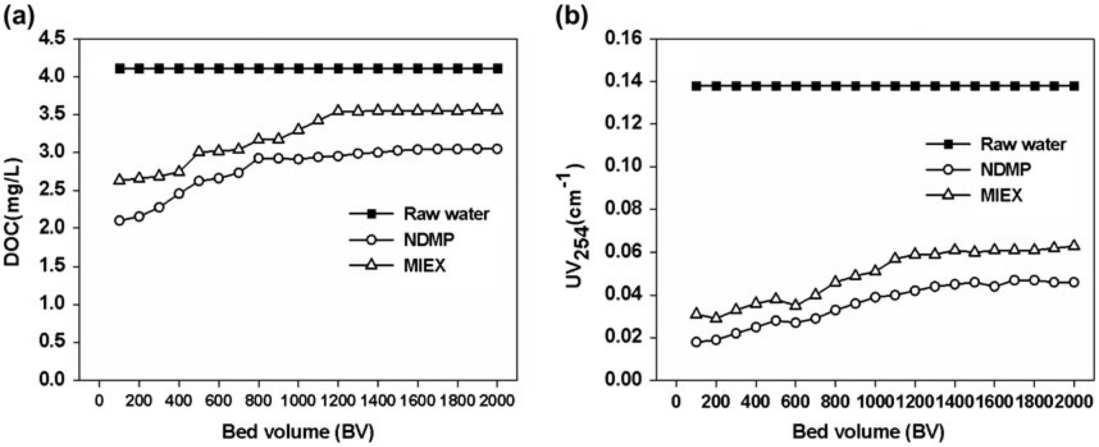

Fig. 5. Multiple-loading jar tests of NDMP and MIEX for (a) DOC and (b) UV_{254} removal for the raw water.

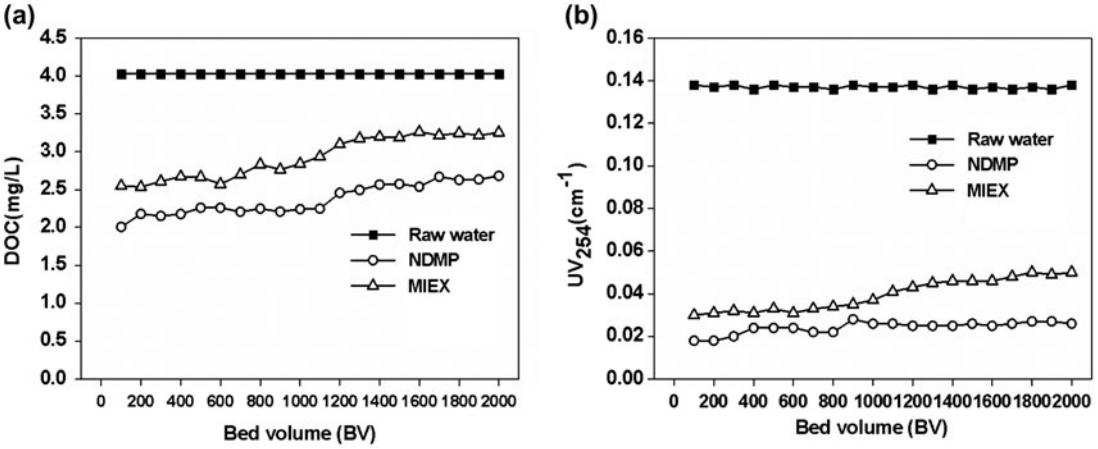

Fig. 6. Effect of regeneration on the removal efficiency of (a) DOC and (b) UV_{254} on NDMP and MIEX resins during batch experiments. The resins were regenerated by sodium chloride (15 wt%) for the raw water.

and MIEX resin treatment, respectively. The removal rates of DOC by the two resins decreased by 13–14% (0.7–0.8 mg/L) at the load of 2,000 BV compared to that of the fresh resin. The removal levels of UV absorbance by NDMP and MIEX resin were also reduced by 9% (0.008 cm^{-1}) and 17% (0.02 cm^{-1}), respectively. The results of UV_{254} removal showed that the regeneration performance of NDMP was better than that of MIEX in multiple-loading procedures (Fig. 5(b)). Hence, the NDMP resin displayed better performance for the removal of aromatic substances after the regeneration with sodium chloride. These results indicate that the NDMP resin could be loaded up to 2,000 BV or more (not tested) after regeneration. Therefore, the NDMP resin is suited to be applied in drinking water treatment.

3.6. Effect of DOM characteristics on its removal

Raw water and treated water after NDMP and MIEX treatment were separated into HPO, TPI, and HPI fractions using the XAD-8/XAD-4 resin fractionation technique [32]. The DOC of the raw water was composed of 48% HPO, 21% TPI, and 31% HPI, which was consistent with its relatively high SUVA value. As the $SUVA_{254}$ is directly correlated with the content of aromatic carbon and the molecular weight of DOM [37,38], high SUVA values correspond to high proportions of hydrophobic compounds. Fig. 7 demonstrated that both NDMP and MIEX could remove HPO and HPI fractions to a certain extent [15,38]. For any given resin dose, the removal rates of all three organic acid fractions increased with the increasing amount of the

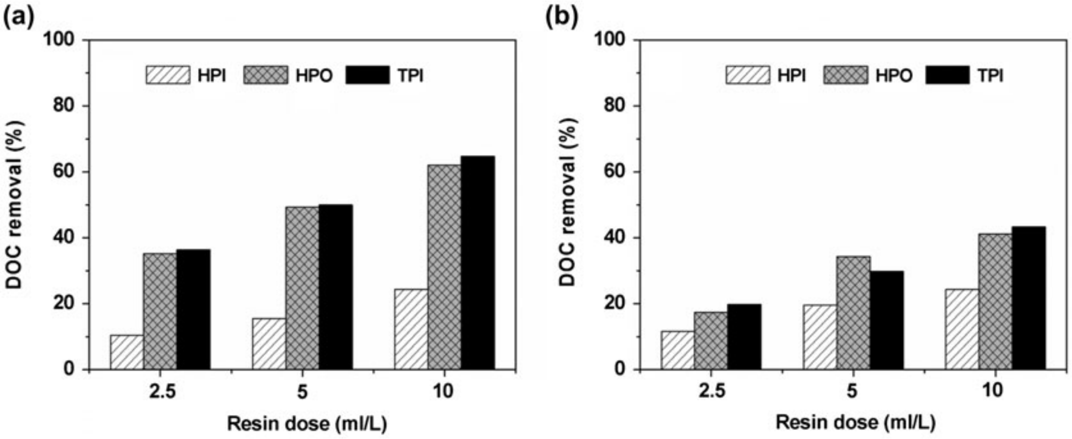

Fig. 7. Impact of (a) NDMP and (b) MIEX resins dose on the removal of various organic acid fractions for raw water.

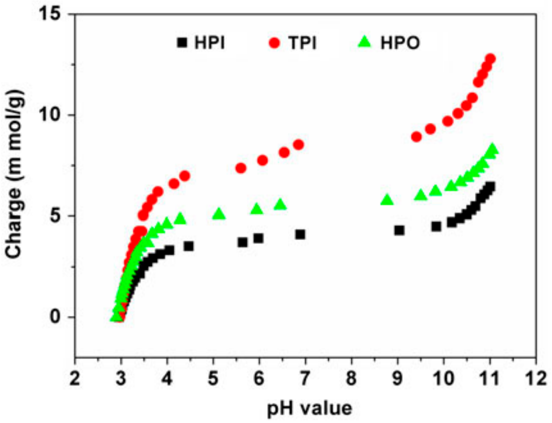

Fig. 8. Titration curve of three hydrophilic and hydrophobic distribution of NOM.

Table 2
Acidity of three fractions of DOM

	Carboxylic groups (mmol/g)	Weakly acidic groups(mmol/g)	Total acidity (mmol/g)
HPI	4.086	2.169	6.255
TPI	8.244	4.159	12.403
HPO	4.873	2.246	7.119

two resins. With the NDMP resin dose of 10 mL/L and the mixing time of 30 min, the removal rates of HPO and TPI were 62 and 65%, respectively, which were higher than that of HPI (24%). It is indicated that the NDMP resin has much higher affinity to more hydrophobic components in DOM. Meanwhile, the removal of HPO and TPI fraction by MIEX was 41 and 43% at the resin dose of 10 mL/L, while the HPI fraction reduction was 24% as shown in Fig. 7(b). This could be explained by the lower charge density of the –NMe_3^+ sites of both resins, rendering the greater affinity for hydrophobic counterions [39]. As compared with MIEX resin, the NDMP resin exhibited similar removal capacity for HPI components and higher elimination rate for the hydrophobic fraction of DOM. According to the potentiometric titration results as shown in Fig. 8 and Table 2, the TPI isolates exhibited the greatest charge density among the three fractions of DOM. The general trend in the carboxyl acidity of the DOM isolates follows the order of TPI > HPO > HPI. Although the main mechanism for the removal of DOM isolates was anion exchange, the charge density of DOM isolates would also influence the extent of DOM removal by magnetic resins [39]. Due to the higher exchange capacity, the NDMP resin showed higher removal efficiency of TPI and HPO isolates with more carboxyl acidity groups. The results eventually led to the higher removal ability of NDMP than MIEX for the removal of DOM.

4. Conclusions

This work investigated the efficiency of a NDMP for the removal of DOM in drinking water treatment as compared to a commercial magnetic resin MIEX. The major conclusions were summarized as follows.

(1) The removal rates of DOC and UV absorbance increased from 25 to 50% and from 54 to 87%, respectively, when the NDMP dosage was elevated from 2.5 to 15 mL (settled resin)/L raw water at 30 min of contact time. The dose of 10 mL settled resin/L and the contact time of 30 min were found to be optimal for DOM removal in the raw water.

(2) Because of the higher exchange capacity, the NDMP resin could remove 11% more DOC and 12% more UV absorbance than MIEX resin with the resin dose of 10 mL/L and the contact time of 30 min. In the multiple-loading jar tests, the DOC level of the treated water was 2.1 mg/L at 100 BV load after treatment with NDMP compared with 2.6 mg/L with MIEX under the same condition. DOC levels gradually increased and reached 3.0 mg/L and 3.6 mg/L after treatment with NDMP and MIEX, respectively. The saturation of the two resins could be relieved after regeneration with sodium chloride. The results confirm that the NDMP resin exhibits better performance for the removal of DOM in the bench-scale experiments.

(3) Magnetic ion-exchange treatment can remove both hydrophobic and HPI organic acid fractions to a certain extent. The NDMP resin could remove more hydrophobic fractions than HPI fractions of DOM, while MIEX resin removes both humic and non-humic substances to a similar extent. As compared with MIEX resin, NDMP resin could remove the hydrophobic fraction of DOM more efficiently.

NDMP process appears to be an advantageous method to remove both hydrophobic and HPI fractions of DOM at high DOC levels in the drinking water treatment even at a relatively low dose and a short contact time.

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