



Investigation of the anti-fouling performance of an aminated resin

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ARTICLE INFO

Article history:

Received 1 February 2012

Received in revised form 7 April 2012

Accepted 10 April 2012

Available online 19 April 2012

Keywords:

Aminated resin

Tannic acid

Anti-fouling

Pore blockage

Water reclamation

ABSTRACT

Accumulation of foulants on adsorbents surface was regarded as the key problem inhibiting the repeated use of adsorbents for bio-treated wastewater reclamation. In the present research, the adsorption performance of aminated resin NDA80 was investigated in terms of anti-fouling from the theoretical comparison with its precursor resin NDA79, and its repeated use in practical application. After preloading with tannic acid (TA) as model background contaminants in biologically treated wastewater, the pore volume and specific surface area of the resins decrease obviously due to partial micropore blockage. However, on NDA80, the adsorption affinities (K_a) of phenol and *p*-nitrophenol increase by 76% and 67% after loaded with 120 $\mu\text{mol/g}$ TA, respectively. Differently, the values of K_a on NDA79 remain almost constant before and after the same loading with TA. The special anti-fouling performance of aminated resin NDA80 was regarded as the contribution of functional groups containing nitrogen on the meso-macro-pore of the resin. The effluent from biologically treated coking wastewater was evaluated by NDA80 on a pilot-scale treatment. The removal of organic contaminants per specific surface area by NDA80 increased by about 35% due to its better anti-fouling property, which is corresponding to the theoretical experiment. The adsorption capacity of NDA80 was almost constant after 71 days of repeated use while the surface area and pore volume of NDA80 decreased by about 30%.

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1. Introduction

Water demand has increased dramatically as a result of increasing populations and developing economies. Fresh water supply is limited, so water shortage has become an important global issue. Wastewater reuse, therefore, is increasingly seen as an essential strategy for making better use of limited freshwater, and a means of preventing deterioration due to contaminants present in the aquatic environment from wastewater disposal [1,2]. However, as the use of various chemicals has increased, the pollutant component of effluent organic matters (EfOMs) has become the focus of great interest. The effluent from wastewater treatment plants contains significant concentrations of organics, such as soluble microbial products (SMPs), natural organic matter (NOM), and synthetic organic compounds (SOCs) [3]. The presence of these substances has complicated wastewater treatment because many of them cannot be or are slowly decomposed biologically. These compounds can cause colors, odors, and toxicity in the effluent, which may lead to aesthetic issues and possible disinfection by-product formation issues that pose risk to human health. Thus, improving the quality of secondary effluent through further treatment has become the subject of growing interest [4].

Some researchers have demonstrated processes in treating EfOMs, such as flocculation [5], adsorption [6], biofiltration [7], ion exchange [8], advanced oxidation processes [9], and membrane processes [10]. Abdessemed et al. [11] showed that adsorption is efficient in removing EfOMs from wastewater due to its simplicity in operation and availability of a wide range of adsorbents. Although activated carbon has been extensively utilized in treating and recovering wastewater, its reusability, and mechanic strength undermine its potential application in wastewater reclamation [12]. In recent years, polymeric resins have attracted increasing attention as an alternative to activated carbon in industrial effluent treatment, mainly due to their favorable physicochemical stability, large adsorption capacity, and structural diversity [13,14].

However, the fouling of resin was encountered during the repeated use of resin [15,16]. After longer run times, foulants in feed-water accumulate physically onto the resin surface and form a barrier in the resin pore, thereby inhibiting the penetration of adsorbates into the resin and blocking the adsorption site. Reductions in both adsorption capacity and adsorption rate are predominantly attributable to pore blockage or constriction [17]. On the other hand, literatures and our previous studies show that resins modified with aminated group could present superior adsorption capacity to SOCs (phenolic compounds [18,19], trihalomethane [20], aromatic sulfonates [21]) and NOM [22,23]. In the adsorption of SOCs on resins, the effect of foulants in multi-component

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Table 1
Physicochemical properties of selected SOCs and TA.

Compound	Molecular weight	log K_{ow}	Water solubility (mmol L ⁻¹ 20 °C)	pKa	Diameter (nm) ^a
TA	1702	13.33	Soluble	–	2.84
Phenol	94	2.17	880	9.8	0.772
<i>p</i> -Nitrophenol	139	1.91	83	7.2	0.932
Aniline	93	0.94	387	4.6	0.810

Data from Refs. [29,30];

^a Calculated at chemicalize.org.

wastewaters on the adsorption capacities of aminated resin and the anti-fouling performance of resin are still lack of systematic research.

The objective of the current study is to investigate the anti-fouling performance of a novel adsorbent modified with amino group in the reclamation of wastewater. Phenol, *p*-nitrophenol, and aniline were employed as candidates of SOCs in industrial wastewater. Tannic acid (TA), chosen as the representative SMP and NOM in biologically treated sewage effluent [24–26], was preloaded as a background contaminant on the resins to examine the effectiveness of fouling on adsorption capacity and adsorption mechanism. Real wastewater from the coking plant after biological treatment was evaluated through a pilot experiment in the current study to simulate the potential application of the new resins in wastewater reclamation. Understanding the adsorption mechanisms and fouling characteristics of resins provides basis for its application in new resin process designs.

2. Materials and methods

2.1. Adsorbents

NDA79 is a chloromethylated and hypercrosslinked macroporous polystyrene adsorbent from Jiangsu N&G Environmental Sci. & Technol. Co., Ltd. (Nanjing, PRC). To aminated NDA79 [21], the resin particles were washed by solvent for 12 h under mechanical stirring. Dimethylamine solution, as the amination reagent, was then gradually added to the flask with the adsorbent. The temperature was increased to 318 K and maintained for 12 h until the reaction was complete. The modified adsorbent was filtered and washed by distilled water. After drying under vacuum at 333 K for 4 h, NDA80 was synthesized.

2.2. Adsorbates

TA (purity > 95%, chemical formula listed in Fig. S1) was purchased from Sigma Aldrich Co. (St. Louis, MO, USA). Phenol, *p*-nitrophenol, and aniline were listed as priority pollutants by the US EPA and/or the M.E.P. of the PRC [27,28]. These reagents were obtained at A.R. grade or higher from Nanjing Chemical Reagent Co., Ltd. (Nanjing, PRC). The physicochemical properties of selected SOCs and TA are listed in Table 1.

2.3. TA Preloading and adsorbent characterization

First, the adsorption isotherms of TA on the resins were determined. Solutions with a range of initial TA concentrations (10–2500 μmol/L) were prepared in 0.01 mol/L NaCl (to maintain a constant ionic strength) and 1.5 mmol/L NaN₃ (a biocide). The 100 mL aliquots were added to a series of 250 mL flasks containing 0.05 g of the adsorbent. The flasks were stoppered and sealed with PTFE sealing tape, and then agitated in a QHZ-98A model shaker with a thermostat (Taicang Huamei Biochemistry Instrument Factory, Suzhou, China) for 2 weeks at 293 K. The amounts of TA

adsorbed onto each resin sample were calculated from the concentration of TA in the solution, which was then analyzed on a Waters 1525 Binary HPLC with a Waters 2487 Dual λ UV Absorbance Detector (Waters Corp., Milford, MA, USA). The isotherms were plotted as the amount of TA adsorbed versus the corresponding equilibrium concentration and were well-fitted by the Freundlich isotherm.

The obtained Freundlich parameters were used to calculate the initial concentrations of TA required to load TA onto the resin at the desired four loading densities: approximately 30, 60, 90, and 120 μmol/g. Then, batch adsorption experiments were conducted at the calculated initial concentrations and under the same conditions as above, except that the volume of solution and weight of adsorbent were increased to 4000 mL and 2.0 g, respectively. The sorption capacity was then calculated by analyzing the equilibrated solution. Initial concentrations were readjusted until the errors of loading quantities were less than 5%. Preloaded adsorbents were filtered, washed with 100 mL distilled water three times and vacuum-dried for 1 week at 313 K. Losses of TA during rinsing were negligible. The NDA79 samples loaded with 30, 60, 90, and 120 μmol/g TA were labeled NDA79a, NDA79b, NDA79c, and NDA79d, respectively. In the same manner, the ND80 samples were labeled NDA80a, NDA80b, NDA80c, and NDA80d.

The Brunauer–Emmett–Teller surface areas (S_{BET}) and pore size distributions (PSDs) of the adsorbents were measured by N₂ adsorption at 77 K using a Micromeritics ASAP-2010 surface area analyzer (Micromeritics Instrument Corp., Norcross, GA, USA). Strong and weak base exchange capacities were determined by chemical titration.

2.4. Batch adsorption experiments

The solution was prepared from deionized water stripped with nitrogen prior to the addition of the adsorbate. The solution pH was circumneutral. The prepared adsorbents (100 ± 0.2 mg) were introduced into 250 mL glass flasks containing 100 mL solution with known concentrations of solute: 1.5 mmol/L NaN₃ and 0.01 mol/L NaCl. The flasks were capped and sealed with PTFE tape before their headspaces were purged with nitrogen gas and shaken for 24 h at 293 K to ensure adsorption equilibrium. The amount of solute loaded onto the adsorbent particles was calculated from the solute concentration in the sampled solution. In these experiments, desorption of TA from the preloaded adsorbents was tested and found to be negligible. Blank samples without adsorbents were also prepared, and losses of adsorbates from solution phases via mechanisms other than adsorption were insignificant.

2.5. Field experiment

The study was conducted using an experimental pilot plant (2.4 m³/day) located within the 6000 m³/day industrial wastewater treatment plant of the coking plant of Liuzhou Iron & Steel Corporation, China. The purpose of this pilot plant study was to define the optimum conditions necessary for actual company application.

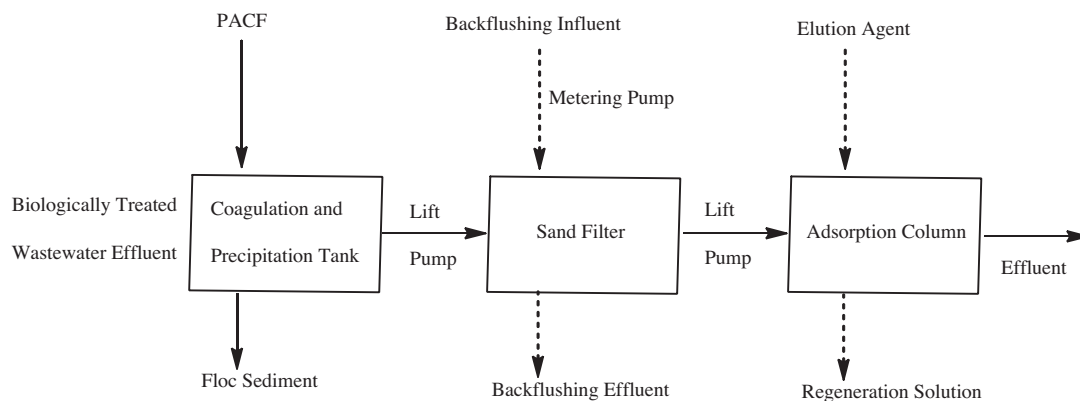


Fig. 1. Flow diagram of the treatment employed.

The plant is completely automated and has three interconnecting modules: settling, filtration, and adsorption. Fig. 1 shows a schematic diagram of the pilot treatment plant. Based on the bench-scale results, the addition of 0.4% PACF with a residence time of about 2 h in the coagulation tank and flocculant produced an excellent and well-formed floc inside the settler. The sand filter with an inner diameter of 0.24 m consisted of 70 cm layer of coarse sand (0.4–0.8 mm). Average filtration velocity was $0.147 \text{ m}^3 / (\text{m}^2 \text{ min})$. During the experiment, there were two filters in operation. Once silted, it was replaced with the other one for the cleaning process. General practices of sand cleaning with air and filtered water were employed. Adsorption was carried out with an iron stainless column filled with 25 L resin. The adsorption velocity was 4 bed volumes (BVs) per hour. The regeneration of saturated resin can be achieved with 1 BV 8% NaOH, 0.6 BV 50% methanol, and 2 BV water at 313 K. The velocity flow was 0.5 BV/h.

3. Results and discussion

3.1. Characterization of resins modified and preloaded

As shown in Fig. 2, Fig. S2 and Table S1, NDA80 has been prepared through amination of NDA79 with dimethylamine, and its pore volume and S_{BET} were reduced by 6.66% and 18.19%, respectively. In the modification process, the S_{BET} of the modified resin decreases more drastically than its pore volume (2.73 times). Through titration, the strong and weak base exchange capacities of NDA80 were determined to be 0.12 and 2.1 mmol/g. The elemental analysis experiment showed that the percentage of nitrogen element in NDA80 was 3.3%. The presence of amino group

on NDA80 was further supported by the absorbance bands at 2764 cm^{-1} (amido group) and 3430 cm^{-1} (N–H bond) in its IR spectra (Fig. S3).

After modification, the average pore width of NDA80 increases from 2.74 nm to 3.13 nm, whereas the pore size of the micropore changed insignificantly (from 1.75 nm to 1.83 nm) compared with that of the mesomacropore (from 4.00 nm to 4.83 nm). These findings indicate that chemical modification by amination might have taken place at the mesomacropore region. Pan et al. [21] reported that the amination reaction may occur mainly in the macroporous region of the resin particle. However, in that study, there was no evidence to support this assumption. In the following section, detailed data will be presented to certify a conclusion.

After preloading with TA, the pore volume and S_{BET} of the resins obviously decrease (Fig. 2 and Table S1). The pore volume and S_{BET} in the micropore field decrease more drastically than that of the mesomacropore, which indicates that TA preloading has a marked influence on the micropore. Moreover, the reduction of S_{BET} is clearly greater than that of the pore volume. For instance, the S_{BET} of NDA79d and NDA80d decrease by 49% and 62% compared with that of NDA79 and NDA80 whose pore volumes decrease by only 35% and 51% at the same loading amounts of TA. Based from these findings, TA preloading leads to partial micropore blockage, and this blocked site could not interact with the adsorbate molecule.

3.2. Effect of amination

Adsorption isotherms of phenol, *p*-nitrophenol, and aniline onto resins with and without TA-loading are presented in Fig. 3, plotted as adsorbed concentration (q , $\mu\text{mol g}^{-1}$ resin) versus aqueous-phase

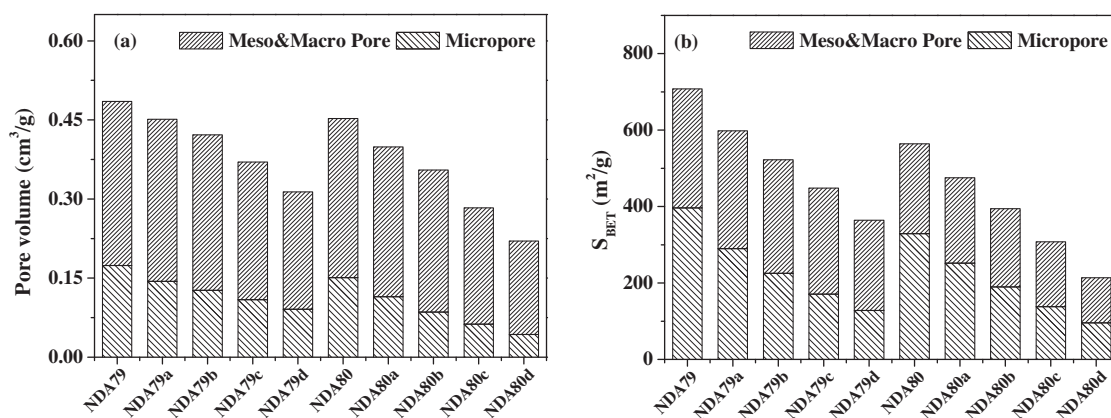


Fig. 2. Influence of modification and TA preloading on the pore volume (a) and S_{BET} (b) of NDA79 and NDA80.

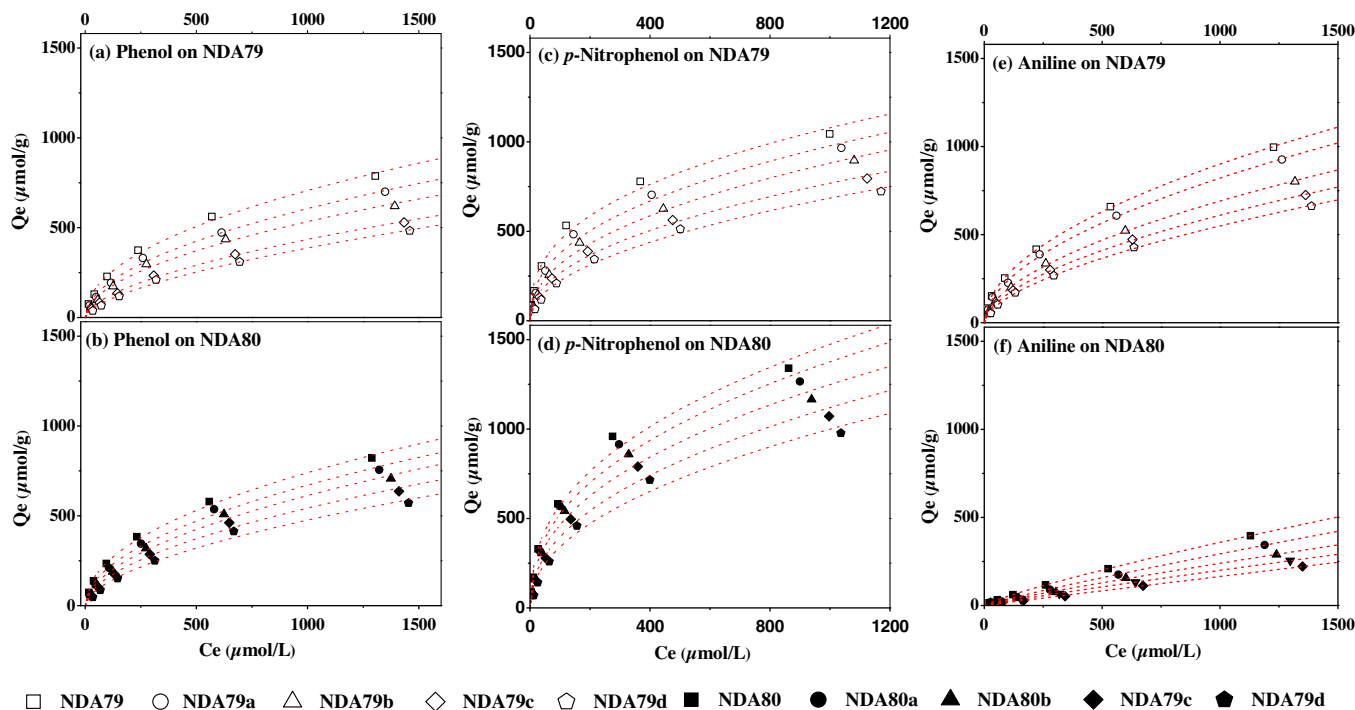


Fig. 3. Isotherms of SOCs onto NDA80 with and without TA-loading at 293 K (Empty marks correspond to the resins before modification (NDA79s) and full marks correspond to the resins after modification (NDA80s); NDA79 and NDA80 are the resins without TA-loading, and the others are resins with TA-loading; Dotted lines are the fitted results with Freundlich equation).

concentration (C_e , $\mu\text{mol L}^{-1}$) at 293 K after equilibrium. As depicted, the adsorption amounts of adsorbates onto NDA79 can be ranked as follows: *p*-nitrophenol > aniline > phenol. Those of NDA80 can be ranked as follows: *p*-nitrophenol > phenol > aniline.

To analyze the adsorption interactions between the adsorbates and the surface of the resins, the adsorption data were fitted to the Freundlich model, $q = K_F \cdot C_e^n$, by nonlinear regression (Table S2), where K_F is a system constant related to the bonding energy and the slope n is a measure of the adsorption intensity or surface heterogeneity. The Freundlich model fits most of the adsorption data well ($R^2 > 0.97$) and the Freundlich n values are all smaller than 1, reflecting adsorption nonlinearity.

Hydrophobic effect, π - π bonds, and hydrogen bonding are the most representative interactions in the adsorption, and the exact strength of these forces depends on the characteristics of the species involved, typically for the adsorbates with certain functional groups [31]. Hydrophobic effect has been emphasized in several studies that involved humic acid [23] and naphthylamine [32] adsorption on polymeric resins. For the three adsorbates, the adsorption capacities onto NDA79 are consistent with their water solubility (Table 1), indicating that hydrophobic interactions related to water solubility between compounds and the surface of resins may be a predominant factor to their adsorption capacities. In addition to the hydrophobic effect, the existence of π - π bonds is reasonable. The tested compounds each possess one benzene ring and one or two functional groups containing oxygen/nitrogen. The electron density on the benzene ring is altered with the existence of an electron-donating (hydroxyl and amino) and an electron-withdrawing (nitro) group, causing the aromatic rings to be effective π -electron acceptors/donors.

Hydrogen bonding is a mechanism for the adsorption of organic compounds on carbon-based resins [33]. The -OH and/or NO_2 on the phenolics and the nitrogen-containing groups on the resin surface of NDA80 may exhibit hydrogen bonding. The adsorption enhancement of phenolic compounds on NDA80 indicates that

the functional groups on the aminated resins surface may play a prominent role. In particular, the amounts of *p*-nitrophenol adsorbed by NDA80 are obviously higher than the amount of phenol adsorbed, demonstrating that the contribution of functional groups from *p*-nitrophenol (-OH and NO_2) is much stronger than that from phenol (-OH). However, the adsorption capacities of aniline decrease drastically onto NDA80 compared with NDA79, which should be attributed to the repulsive interactions between the functional groups containing nitrogen attached to NDA80 and aniline under the same condition in an aqueous solution. Considering the reduction of the surface area of NDA80 after modification, it can be concluded that the surface area of NDA79 and the functional groups on NDA80 may be the main factor and the determinant, respectively, in an adsorption process.

3.3. Effect of preloading

Single-concentration point adsorption coefficients (K_d) of the adsorbates from the fitted Freundlich equations at equilibrium concentrations of $800 \mu\text{mol/L}$ were calculated to clearly explain the relationship between adsorption capacity and interactions (Table 2).

As shown in Table 2, the K_d values of the adsorbates decrease with an increase in the preloading, which should be attributed to the decrease of the surface area of the resin. The values of K_d for *p*-nitrophenol are much greater than those for the other two adsorbates due to the contribution of multi-interactions. Thus, the aminated resin is advantageous in adsorbing SOCs with hydrogen bonding acceptors/donors, such as -OH and $-\text{NO}_2$, which corresponds to the typical character of organic compounds in effluent organic matters (EfOMs) [1,2].

The significant differences among their adsorption behavior and mechanism can be further explained by the preloading of TA on their surfaces. An attempt to evaluate the adsorption affinity (K_a) per unit specific surface area ($K_a = K_d/S_{\text{BET}}$) has been made.

Table 2
Adsorption coefficients (K_a) of the three adsorbates on the resins.

Resin	K_a		
	Phenol	<i>p</i> -Nitrophenol	Aniline
NDA79	629	990	800
NDA79a	543	896	738
NDA79b	473	795	609
NDA79c	363	695	542
NDA79d	328	611	481
NDA80	672	1345	288
NDA80a	600	1250	275
NDA80b	548	1125	197
NDA80c	476	1010	160
NDA80d	424	895	130

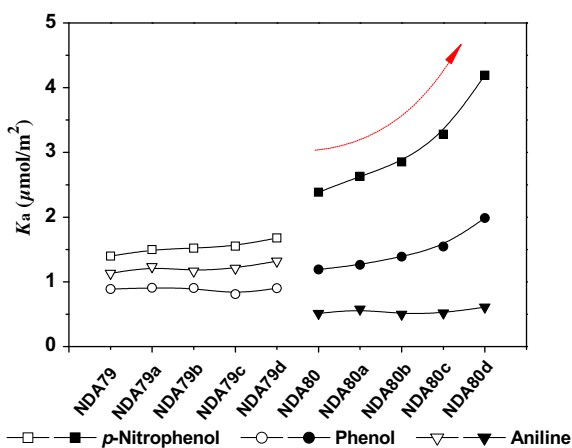


Fig. 4. Adsorption coefficients (K_a) of adsorbates on the resins with and without TA-loading. (Empty marks correspond to the resins before modification (NDA79s) and full marks correspond to the resins after modification (NDA80s); NDA79 and NDA80 are the resins without TA-loading, and the others are resins with TA-loading.)

The obtained values of K_a are depicted in Fig. 4. The values of K_a on NDA79s at different preloading are almost constant, demonstrating that hydrophobic interactions are dependent on the specific surface area of the resin and are the main factors in the adsorption process on NDA79s without functional groups containing nitrogen. On the other hand, the adsorption affinity K_a of all adsorbates has not been influenced by the increase of preloading on NDA79s, showing that the disparity of the substituent groups attached to benzene ring is negligible.

The K_a curves of NDA80s in Fig. 4 are different from those on NDA79s. Moreover, the K_a values of NDA80s for aniline with different preloadings remain constant and are slightly less than those on NDA79s. This finding is attributed to the repulsive interactions abovementioned. Thus, the amino group of aniline has a negative contribution to its adsorption on NDA80. Interestingly, after the preloading of TA, the adsorption affinities of *p*-nitrophenol and phenol on the aminated resins of NDA80 increase with the increase in preloading. For example, on NDA80d, the adsorption affinities (K_a) of phenol and *p*-nitrophenol increase by 76% and 67% compared to NDA80 after loaded with 120 $\mu\text{mol/g}$ TA, indicating that NDA80 may be a much better anti-fouling porous adsorbent.

According to the characterization of the NDA80s, by increasing the preloading of TA, partial micropore blockage leads to the obvious reduction of the micropore area. However, the increasing adsorption affinities of *p*-nitrophenol and phenol on the NDA80s certify that the interaction sites of the two phenolic compounds should lie in the mesomacropore of the resin rather than in the micropore region. Hydrogen bonding is the predominant interaction in phenolic compound adsorption on NDA80s. Therefore, the amination reaction occurs mainly in the mesomacropore region of the resin, which confirmed the assumption in Section 3.1. Furthermore, the existence of functional groups containing nitrogen on these regions of the resin which are difficult to shield, is regarded as the main driving force to their anti-fouling performance.

3.4. Practical application

Coking wastewater is a complex industrial wastewater generated from coal coking plants. Traditionally, biological treatment is the main coking wastewater treatment technology [34]. However, the effluent of common biological treatments contain certain amount of organic pollutants, such as phenols, polycyclic aromatic hydrocarbons (PAHs), SMPs, NOMs, and other toxic compounds, which makes coking wastewater more recalcitrant for further biodegradation ($\text{BOD}_5/\text{COD} < 0.1$), resulting in the failure to meet water reclamation standards [35,36].

In this section, the application of the adsorption process using the aminated resin NDA80 was investigated by reclaiming biologically treated coking wastewater and optimizing the process parameters. The treatment processes consisted of three parts, as shown in Fig. 1. After the pretreatment (flocculation and filtration), the COD values of water are still high (80–157 mg/L, mean value is 103 mg/L). Six batches (71 days) of adsorption recycles were applied to the process to examine the performance of the adsorption capacity of NDA 80 after regeneration. Fig. 5 shows the operating

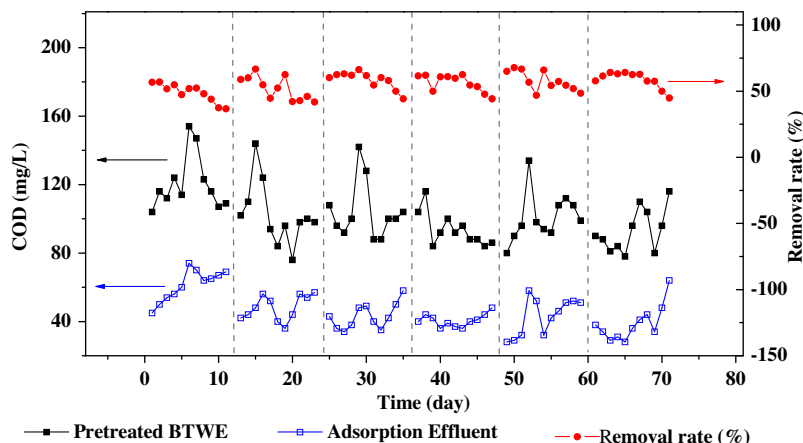


Fig. 5. Removal rates and COD values corresponding to batches and days.

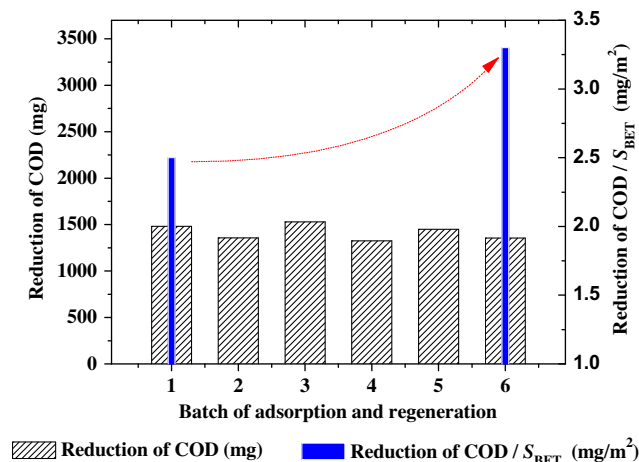


Fig. 6. COD reduction and tendency per surface area corresponding to the batches.

parameters in terms of removal rates and COD values in the adsorption unit corresponding to the days. As shown in Fig. 5, the COD values of the influent fluctuate largely and the removal rates almost remained constant (about 50%).

After six batches and adsorption and regeneration with batch processing volume of 1000 BV, the pore volume and S_{BET} of resin decrease largely, while the sphericity after attrition of resin NDA80 change insignificantly (Table S3). Compared with the drop of surface area of resin, the quality of effluent and the total COD reduction in each batch was steady. As shown in Fig. 6, the total COD reduction per specific surface area of resin presents a 135% increase from batch 1 to batch 6, which corresponds to the change of adsorbate adsorption affinity on the resins with TA-loading (Fig. 4). This result demonstrates that the blockage of the micropore does not influence the adsorption capacity of the aminated resin NDA80, which is in agreement with the observations on the static experiment. The superior properties of NDA80, namely, sorption capacity and anti-contamination, as manifested by the successful performance on an industrial scale, make aminated hypercross-linked resin NDA80 a highly feasible adsorbent for reclaiming water in the advanced treatment of coking wastewater.

4. Conclusions

The present work investigated the main factors supposedly controlling the special anti-fouling performance of aminated resin, such as the water solubility of adsorbates, S_{BET} of resin, functional group, and preloading of TA onto resins as model background contaminants in biologically treated wastewater. The results demonstrated that (1) the pore volume and S_{BET} of the resins clearly decrease due to partial micropore blockage, whereas the adsorption affinities of the adsorbates slightly increase by increasing the preloading of TA; (2) the amination reaction occurs mainly in the mesomacropore of the resin, which contributes positively to the adsorption of *p*-nitrophenol and phenol and restrains the adsorption of aniline due to the existence of functional groups on the surface of NDA80; (3) the hydrophobic effect plays an important role in the adsorption process on hypercrosslinked resin NDA79, whereas the hydrogen bonding is the determinant factor on aminated resin NDA80; and (4) the blockage of micropore does not clearly influence the adsorption capacity of the aminated resin NDA80 due to the existence of functional groups on the mesomacropore, leading to the superior properties of anti-contamination on pilot scale treating the effluent from biologically treated coking wastewater. The current work presents data which are beneficial in exploring the potential application of aminated resin NDA80 in associated wastewater reclamation.

Acknowledgments

We gratefully thank Dr. Weiben Yang (Nanjing Normal University) for his kind assistance. This research was funded by the Program for Changjiang Scholars Innovative Research Team in University, NSFC (50825802, 50938004 and 51178215) and Jiangsu Nature Science Fund for Distinguished Scientists (BK2010006 and BK2011032) PR China.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.ccej.2012.04.038>.

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