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Optimization and effect of powdered activated carbon addition on commingled chemical industrial wastewater treatment in a sequencing batch reactor

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ABSTRACT

In this study, commingled chemical industrial wastewater was treated using a sequencing batch reactor with powdered activated carbon (PAC–SBR). The performance of the PAC–SBR system was investigated under various dosages and feeding frequencies. Results showed that a PAC dosage of 2 g PAC/L-reactor and a feeding frequency of 0.25 g PAC/L-reactor to 0.5 g PAC/L-reactor per 3.5–7 d maintained a stable long-term performance during operation. The effects of PAC addition were also considered. Removal of organic matter in the PAC–SBR system demonstrated synergistic effects of biodegradation and PAC adsorption. The system with PAC had better sludge volume index values (100–200) than that without PAC (150–250). This result suggested that the sludge settling characteristic was improved. Hence, PAC addition was proven to increase biomass in the PAC–SBR system, which improved the removal of chemical oxygen demand through biodegradation. Furthermore, the excitation–emission matrix spectrum of organic matter showed that compounds in raw wastewater that contains aromatic rings, heterocyclic ring structures, and bicyclic aromatic structures were partially removed by biodegradation and PAC adsorption. The organic acid-like compounds, which were recalcitrant to biodegradation, were adsorbed via PAC. The results of organic matter fractionation showed that compounds in transphilic neutral and hydrophilic fractions were easily transformed through biodegradation, whereas compounds in hydrophobic fractions were adsorbed by PAC.

Keywords: Chemical industrial wastewater; Powdered activated carbon; Adsorption; Optimization; Organic matter fractionation

1. Introduction

Commingled chemical industrial wastewater is a challenging industrial effluent to treat and reuse because of its characteristics such as high pollutant concentration, complex component, toxicity or inhibition to biological treatment system, and refractoriness to biodegradation [1]. Biological treatment of industrial effluent through the use of suspended or attached growth is sufficient for organic pollutant removal. However, persistent refractory compounds limit removal performance in biological processes without

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other technology combinations for commingled chemical industrial wastewater treatment. Hence, biological treatment systems are combined with physical and chemical methods to treat this type of industrial wastewater [2].

Physical adsorption is a common water treatment method nowadays. Activated carbon, especially powdered activated carbon (PAC), is widely used in wastewater biological treatment and portable water treatment because of its large adsorption capacity [3]. The presence of PAC in a biological treatment system enhances the performance of the system because of the simultaneous biological removal and adsorption of pollutants. Biological inhibition caused by some organic compounds is reportedly decreased by PAC [4]. An activated sludge system with powdered activated carbon (PAC–AS) is thus considered an efficient strategy for treating commingled chemical industrial wastewater [5].

The PAC–AS system has been mostly used to treat organic pollutant-containing wastewaters [6]. The superior removal capabilities of the PAC–AS system in these studies were obtained at different PAC dosages. Jaafarzadeh et al. [7] employed a conventional AS process and batch decant reactor with PAC addition to treat landfill leachate. They found that the optimal PAC dosage was 3.5 g/L. Kargi and Pamukoglu [8] employed a biological process with PAC to treat landfill leachate and found 2 g/L PAC to be the optimal dosage. Li et al. [9] utilized a submerged membrane bioreactor (MBR) with 1.2 g/L PAC to treat synthetic wastewater. Liu et al. [10] employed MBR with 25 g/L PAC to treat reservoir water. Ma et al. [11] utilized an MBR with 50 g/L PAC for slightly polluted surface water treatment. Hence, different optimal PAC dosages were obtained to treat different types of wastewater. However, only a few studies have focused on treating commingled chemical industrial wastewater. Moreover, PAC feeding frequency should be considered to ensure that industrial applications achieve a stable long-term performance. Therefore, the effects of feeding frequency, which were rarely considered in previous research, on the PAC–AS system must be investigated and optimized.

The organic matter in commingled chemical industrial wastewater is a heterogeneous mixture of hydrocarbons, aromatic compounds, carboxylic acids, and phenol among others. The removal of these organic pollutants, especially persistent compounds, is crucial in chemical industrial wastewater treatment. Microorganisms can effectively remove hydrophobic and readily biodegradable organics but are limited in removing hydrophilic and persistent compounds; PAC can complement biological processes and effectively remove hydrophilic and persistent compounds [12,13]. These results show that adding PAC to a biological treatment system changes the removal characteristics of organic matter because of the existence of both PAC adsorption and activated sludge biodegradation. Hence, the organic matter removal characteristics of the PAC–AS system are important.

In this study, a PAC–sequencing batch reactor (PAC–SBR) system was employed to treat commingled chemical industrial wastewater from a chemical industrial park in Tianjin, China. The PAC dosage and feeding frequency were optimized. The effects of PAC addition on the water quality and activated sludge characteristics were also determined. The results of this study could provide efficacious technical support to full-scale treatment of chemical industrial wastewater.

2. Material and methods

2.1. Wastewater source and qualities

Raw wastewater was obtained from the influent of a full-scale wastewater treatment plant in a chemical industrial park in Tianjin, China. More than 50 types of industrial plants are situated in this area, including pharmaceutical, manufacturing, petroleum chemical, electronics, chemical reagent, household, and personal care product plants. The detailed wastewater qualities are listed in Table 1.

2.2. Experimental setup and operating conditions

Three systems (PAC–SBR, PAC, and SBR systems) with a working volume of 2 L were employed to study the effects of PAC addition (Fig. 1). These systems were operated in the following sequence: filling (5 min), aerobic reaction (20 h), settling (1.5 h), decanting (0.5 h), and idling (1.91 h). The inoculated AS was obtained from a local municipal wastewater treatment plant (hydraulic retention time, 12.5 h; solid retention

Table 1
Characteristics of the commingled chemical industrial wastewater samples

Item	Value
CODcr	950–1,500 mg/L
BOD_5	200–800 mg/L
Ammonia nitrogen	15–20 mg/L
TN	25–30 mg/L
TP	2.0–2.5 mg/L
Suspended solids (SS)	100–256 mg/L
pH	7.8–8.3

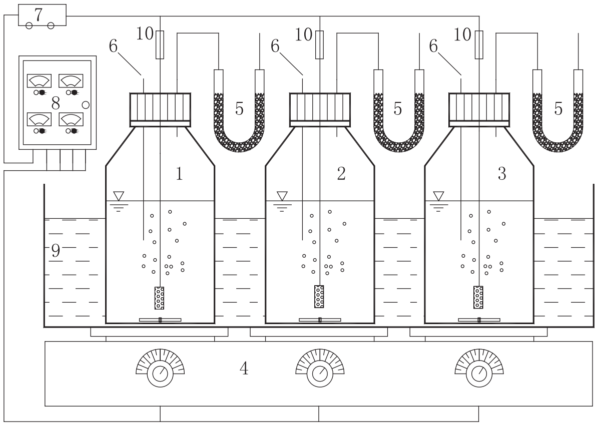

Fig. 1. Schematic layout of the reactor systems: (1) PAC–SBR, (2) SBR, (3) PAC, (4) magnetic stirrer, (5) air vent, (6) sampling port, (7) air pump, (8) time controller, (9) water bath, and (10) mass flow controller.

time, 20 d; TN and TP removal efficiency rates, 73% and 67%, respectively). During AS acclimation, the influent wastewater was diluted and mixed with glucose. Then, the ratio of industrial wastewater increased and glucose decreased gradually until the influent was the only industrial wastewater that remained at the end of AS acclimation. After 30 d of acclimation, the SBR systems were run continuously with 3.0 g/L mixed liquor suspended solids (MLSS). Commercial PAC (60 –120 μm) from the Tianjin Reagent Factory was pre-dried at 103˚C and initially injected to the reactors at 2 g PAC/L-reactor. The characteristics of PAC are shown in Table 2. Sodium azide (1 g/L) was added to inactivate the micro-organisms and prevent microbial biodegradation in the PAC system.

To optimize PAC addition, different amounts of PAC (0 g/L-reactor to 10 g/L-reactor) and various PAC feeding frequencies (2 g/L-reactor per 28 d, 1 g/L-reactor per 14 d, 0.5 g/L-reactor per 7 d, 0.25 g/L-reactor per 3.5 d, and without PAC addition) were investigated in the PAC–SBR system.

2.3. Determining biomass in the PAC–AS system

Mixed liquor PAC suspended solid (MLCSS) and mixed liquor biomass suspended solid (MLBSS) concentrations were estimated using the differential heating method described by Arbuckle and Grigg [14]. The mixed liquor volatile suspended solids (MLVSS) contain both MLBSS and MLCSS. The biomass could be completely volatized and PAC loss could be neglected by heating at 400˚C. MLBSS was thus represented as weight loss by heating at 400˚C, whereas MLVSS was represented as weight loss by heating at 600˚C, which included the loss of PAC and biomass.

2.4. Fluorescence excitation–emission matrix (EEM) spectroscopic analysis

Fluorescence EEM spectroscopy was used as a preliminary means to identify the major contaminants present in the wastewater. Fluorescence spectroscopy was employed to measure the location and intensity of fluorophores in a 1 cm quartz cell using a spectrofluorometer (Jobin Yvon Fluorolog 3-21), with a xenon lamp as the excitation source at room temperature. The spectra were collected with subsequent scanning emission wavelengths from 280 to 480 nm at 5 nm increments by varying the excitation wavelength from 220 to 400 nm at 2 nm increments. The excitation and emission were scanned with a 5 nm slit width. The influent and effluent samples of the PAC–SBR, PAC, and SBR systems were collected on days 5, 15, and 20 (adjusted at pH 7) and diluted to a final dissolved organic carbon (DOC) of 1 mg/L or less than 1 mg/L (in the same dilution ratio) [15].

2.5. Organic matter fractionation of wastewater

Pretreatment for organic matter fractionation was conducted by following the method presented by Bu et al. [16]. The influent and effluent samples of the PAC–SBR, PAC, and SBR systems were collected on days 5, 15, and 20. The organic matter in the samples

Table 2
Characteristics of commercial PAC

Parameter	Unit	Value
Particle size	μm	60–120
Bet surface area	Sq. m g^{-1}	882
Langmuir surface area	Sq. m g^{-1}	1,104
Micropore area	Sq. m g^{-1}	416
Micropore volume	cc g^{-1}	0.19
Average pore diameter (4 v/a by Langmuir)	Å	17.4

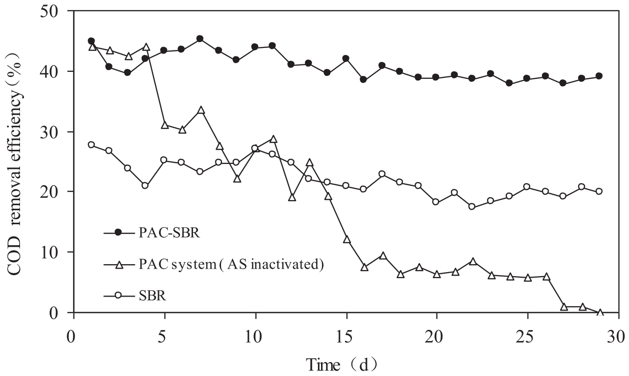

Fig. 2. Performance comparison of PAC–SBR, SBR, and PAC systems in COD removal efficiency.

was fractionated by two serial columns filled with Amberlite XAD-8 and XAD-4 resins. Before being added into the columns, the resins were cleaned with methanol and acetonitrile by Soxhlet extraction for 12 h, respectively. The wastewater samples were first filtered through a 0.45 μm glass fiber filter to remove suspended solids. The filtrate was then acidified with 6 M HCl to a pH of 2 and passed through the XAD-8 and XAD-4 resin columns in series. The materials retained in XAD-8 and XAD-4 were hydrophobic and transphilic fractions, respectively. The materials that were not retained on either resin comprised the hydrophilic fraction (HPI). The hydrophobic acid (HPO-A) and transphilic acid (TPI-A) were eluted by backwashing with 0.1 M NaOH from the XAD-8 and XAD-4 columns, respectively. The resins were washed with distilled water and extracted with a solution of 75% acetonitrile and 25% ultrapure water (Millipore Milli-Q). The extracts from the XAD-8 and XAD-4 columns were condensed using a rotary evaporator and dried using a freeze dryer. These extracts were separately redissolved in Milli-Q water with pH 7, which comprised the hydrophobic neutral (HPO-N) and transphilic neutral (TPI-N), respectively.

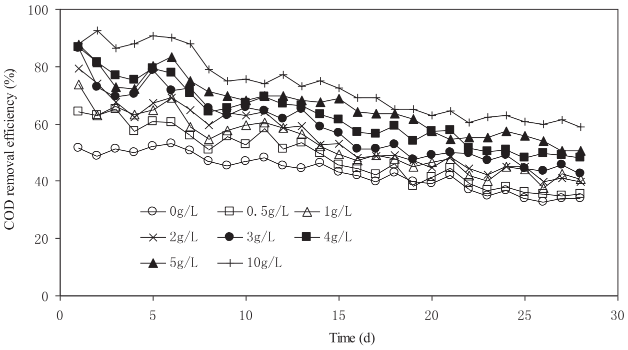

Fig. 3. COD removal efficiency with different PAC dosage additions.

2.6. Analytical methods for activated sludge and wastewater quality

Aqueous samples were collected daily, and sludge samples were collected every 7 d. The MLSS and MLVSS concentrations and sludge volume index (SVI) in the PAC–SBR and SBR systems were determined. The other water quality parameters were analyzed by following standard methods [17]. COD was measured using the potassium dichromate oxidation method. BOD_5 was determined using fast BOD measuring devices (OxiTOP IS6, Germany).

3. Results and discussion

3.1. COD removal in the three systems

The COD removal performance of the PAC–SBR, PAC, and SBR systems is compared in Fig. 2. PAC was initially added at 2 g PAC/L-reactor to the systems and supplied continuously to maintain the PAC concentration at 2 g/L. The results showed that the COD removal efficiency rate in the PAC system was achieved as high as 45% in the first 5 d of operation but sharply decreased with time (<10% beyond 15 d of operation). This result was due to

Table 3
Estimated mass balances (%) of COD in various treatment systems*

	SBR		PAC system		PAC–SBR		
Stage	Mass biodegraded	Mass in effluent	Mass adsorbed	Mass in effluent	Mass biodegraded	Mass adsorbed	Mass in effluent
Days 1–4	24.7	75.3	43.5	56.5	–	42.7	58.3
Days 5–14	24	76	25.1	74.9	17.5	25.1	57.4
Days 15–30	19.9	80.1	5.6	94.4	33.3	5.6	61.1

*The numbers were average value of COD percentage and were obtained according to the results in Fig. 2. The mass balance of COD in each SBR was determined by Equation: $COD_{in} = COD_b + COD_{ad} + COD_{out}$.

PAC adsorption capacity exhaustion. However, the long-term performance of PAC–SBR was better (>40% removal efficiency) than the SBR (20% removal efficiency). This result indicated that COD removal performance demonstrated synergistic effects with the combination of biodegradation and PAC adsorption (days 15–30).

The mass balance of COD in the systems was determined to investigate the contribution of sorption and biodegradation on COD removal in the PAC–SBR system (Table 3). The average values of COD percentage in Table 3 are obtained according to the results in Fig. 2. The sorption of organic matter in the SBR system could be neglected because of long-term acclimation and a spot of excess sludge removal (solid retention time, 20–30 d). The minor importance of sorption in the AS system was also reported by Nesseris and Stasinakis [18]. In the PAC–SBR system, 42.7% of COD was removed via PAC on days 1–4, whereas AS biodegradation was not significant. On days 5–14, COD removal via adsorption decreased because of adsorption capacity exhaustion, whereas COD removal by biodegradation increased. On days 15–30, PAC adsorption function was not significant, whereas biodegradation was the principal approach of removing COD. Hence, in the PAC-SBR system, most organic matter was initially removed by PAC via adsorption and then, gradually by AS via biodegradation.

The PAC–SBR system was proven effective in this industrial wastewater treatment. Therefore, the removal performance of the PAC–SBR system under various operating conditions and the influence of PAC addition on both the water quality and AS characteristics were determined in the following section.

3.2. Optimization of the performance of the PAC–SBR system

3.2.1. PAC dosage

PAC, which varied from 0 g PAC/L-reactor to 10 g PAC/L-reactor, was initially added to determine the effects of PAC dosage on COD removal performance in the PAC–SBR system. As shown in Fig. 3, organic matter removal was enhanced with increasing PAC dosage. The average COD removal efficiency increased from 43% without PAC addition to 73% with 10 g PAC/L-reactor addition. Nevertheless, COD removal performance continuously decreased during the 28-d operation because of PAC adsorption saturation.

The increase in COD removal efficiency with increasing PAC dosage has been attributed to PAC adsorption [19]. However, the increase in organic matter removal with increasing PAC dosage from 2 g PAC/L-reactor to 10 g PAC/L-reactor was initially not significant. Furthermore, the PAC adsorption capacity

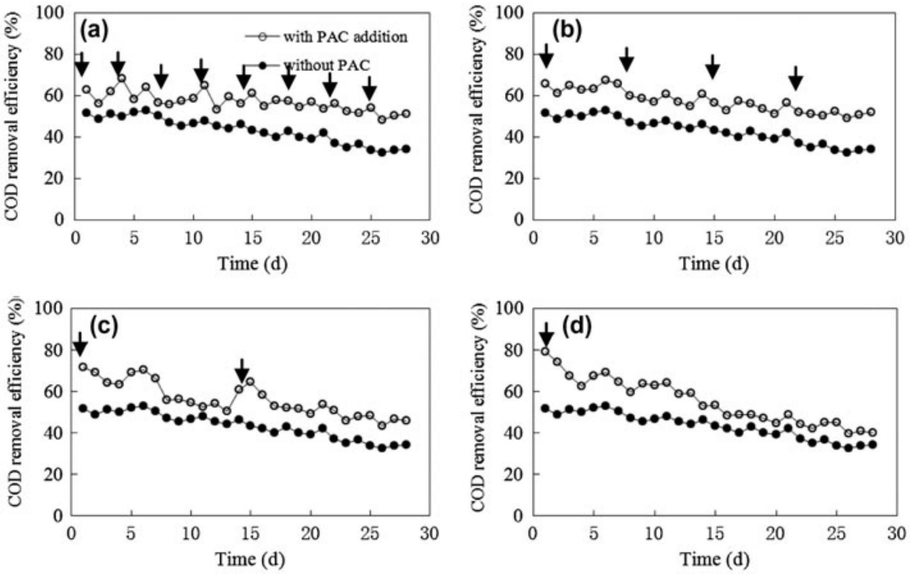

Fig. 4. COD removal performance with different PAC frequency additions: (a) adding 0.25 g-PAC/L-reactor per 3.5 d; (b) adding 0.5 g-PAC/L-reactor per 7 d; (c) adding 1 g-PAC/L-reactor per 14 d; and (d) adding 2 g-PAC/L-reactor per 28 d. The arrows show the time point of PAC addition.

could not be fully utilized, and PAC runoff could occur at a high dosage of PAC addition. Therefore, the optimal PAC dosage addition was chosen to be 2 g PAC/L-reactor. Moreover, PAC should be fed continuously to maintain high removal performance during long-term operation. The PAC feeding frequency is thus considered in the next section.

3.2.2. Frequency of PAC addition

Fig. 4 shows COD removal under different PAC feeding frequencies. PAC addition with low dosage but high feeding frequency (such as 0.25 g PAC/L-reactor per 3.5 d) exhibited relatively lower performance at the initial stage but maintained a more stable and long-term performance than those with high dosage but low feeding frequency (2 g PAC/L-reactor per 28 d). Hence, the optimal feeding frequency of PAC was determined to be 0.25 g PAC/L-reactor to 0.5 g PAC/L-reactor per 3.5–7 d.

3.3. Effects of PAC addition on activated sludge characteristics

SVI and MLBSS were considered to reveal the effects of PAC addition on activated sludge characteristics. As shown in Fig. 5, the SVI in the SBR system increased while the biomass decreased during the operation, which suggests a tendency to decay and disintegrate because of the toxicity of compounds and insufficient biodegradable substrate [20,21]. Moreover, deterioration of the sludge settling capability and compressibility decreased MLBSS concentration [22]. However, after the initial addition of 2 g/L-reactor PAC, the value of SVI in the PAC–SBR system gradually decreased and the biomass (excluding PAC) increased slightly. This result agreed with the findings of Uygur and Kargi [22] that PAC aids in the formation of large microbial flocs, which improve settling properties and increase biomass in the reactor.

Changes in effluent SS concentration during the operation also supported the results. As shown in Fig. 5(a), the SS concentration in the SBR system effluents increased with system operation because of the change in sludge settling capability. However, the SS concentration in the PAC–SBR system effluents increased during the initial operation because of PAC runoff. Then, AS settleability improved because of PAC addition, resulting in the decrease of SS concentration.

3.4. Fluorescence analysis of wastewater in different treatment systems

Fluorescent analysis of organic matter reflects the removal characteristics of organic matter in the wastewater [23]. The EEM spectra of organic matter in the raw wastewater and treated effluents are shown in Fig. 6. Three principal EEM peaks of organic matter in the raw wastewater were observed. The fluorescence peak at $\lambda_{ex}/\lambda_{em} = 270–300$ nm/320–380 nm (A) is labeled as the compounds that contain aromatic and heterocyclic ring structures [24,25]. The peak with a slight shoulder at $\lambda_{ex}/\lambda_{em} = 310–330$ nm/330–370 nm (B) is characterized as the compounds that contain nitrogen [24]. The peak at $\lambda_{ex}/\lambda_{em} = 280–360$ nm/380–440 nm (C) is labeled as organic acid-like compounds [24,26] or compounds that contain three to five aromatic rings [27]. Multiple peaks in the EEM spectrum of organic matter in raw wastewater showed that the sample was composed of complex components associated with compounds that contain aromatic rings, heterocyclic ring structures, bicyclic aromatic structures, and organic acid-like compounds.

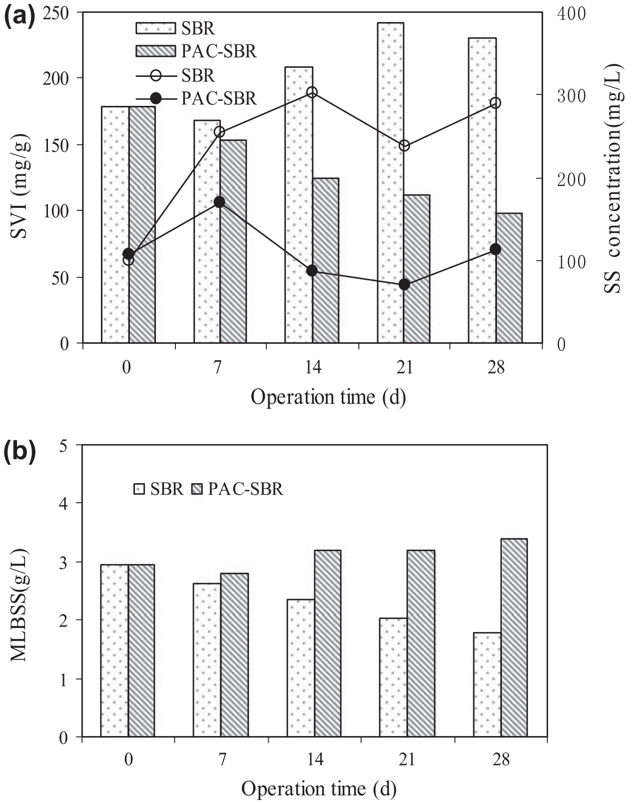

Fig. 5. Changes in activated sludge characteristics and SS concentrations of treated effluents in PAC–SBR and SBR systems: (a) SVI of activated sludge and SS concentration in treated wastewater and (b) MLBSS concentration in the systems.

In the effluent of the SBR system, the three EEM peaks (peaks A, B, and C) still existed. The fluores-

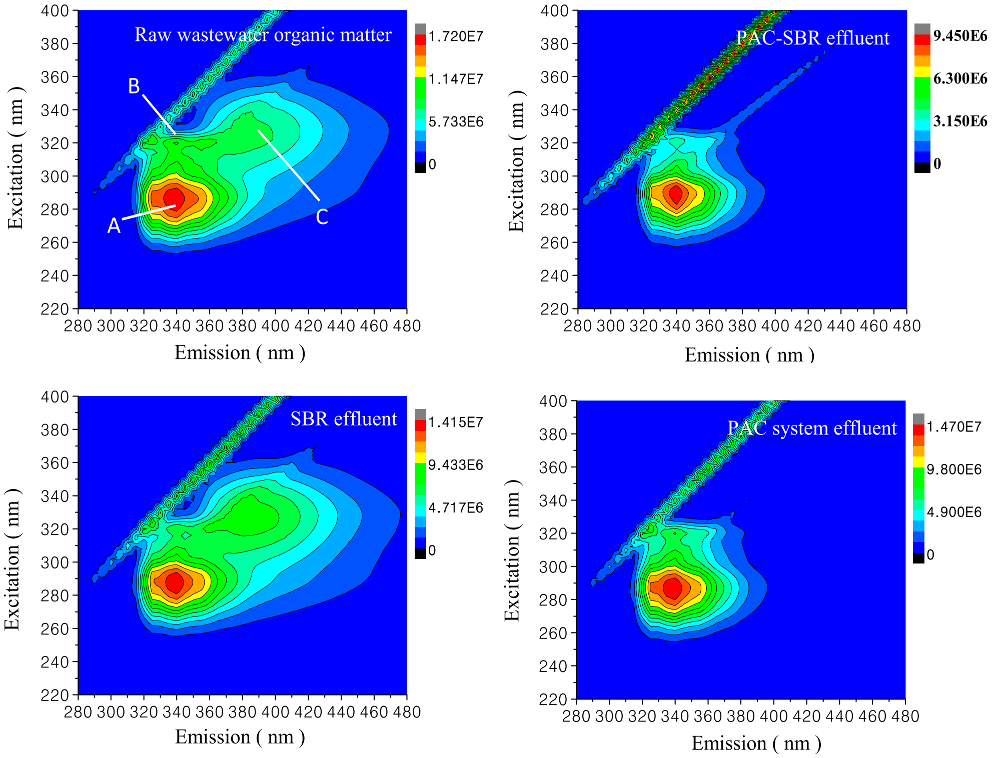

Fig. 6. Fluorescence EEMs for organic matter in the raw wastewater and treated effluents by various treatment systems.

cence peaks A and B were partially reduced, whereas peak C was not reduced significantly after the treatment. This observation indicated that the organic matter in peak C was persistent and difficult to biodegrade. In the effluent of the PAC and PAC–SBR systems, the fluorescence peak C disappeared after the treatment. This result suggested that these organic compounds were easily adsorbed by PAC, which was also illustrated in previous researches [28,29]. Therefore, PAC helped to remove the compounds that were difficult for micro-organisms to biodegrade.

3.5. Effect of PAC addition on organic matter fractionation in wastewater

The polarity distribution analysis in the PAC–SBR, PAC, and SBR systems was employed to reveal the removal characteristics of organic matter (Fig. 7). The organic matter in the hydrophobic fractions (HPO-A and HPO-N) was dominant in the raw wastewater, accounting for more than 48% of the total organic carbon (TOC). The compounds in the transphilic (TPI-A and TPI-N) and hydrophilic fractions accounted for 29 and 22% of TOC, respectively.

In the effluent of the SBR system, the compounds in these fractions could be transformed with the removal efficiency in the following order: TPI-N (71%) > HPI (59%) > HPO-N (53%) > HPO-A (48%) > TPI-A (<1%). The results showed that the organic matter in the hydrophobic and TPI-A fractions was recalcitrant to biodegradation, whereas the compounds in the TPI-N and HPI fractions were readily biodegradable. In contrast, the compounds in these fractions could be adsorbed by PAC with the removal efficiency in the following order: HPO-A (70%) > HPO-N (63%) > TPI-N (44%) > TPI-A (27%) > HPI (21%) in the PAC system. The results showed that the organic matter in the hydrophobic and transphilic fractions was easily adsorbed by PAC, whereas the compounds in the HPI fraction were difficult to be adsorbed. In the effluent of the PAC–SBR system, the compounds in these fractions were significantly removed because of biodegradation and PAC adsorption. PAC adsorbed the hydrophobic compounds that often inhibited micro-organisms. Thus, the removal performance could be enhanced in the PAC–SBR system.

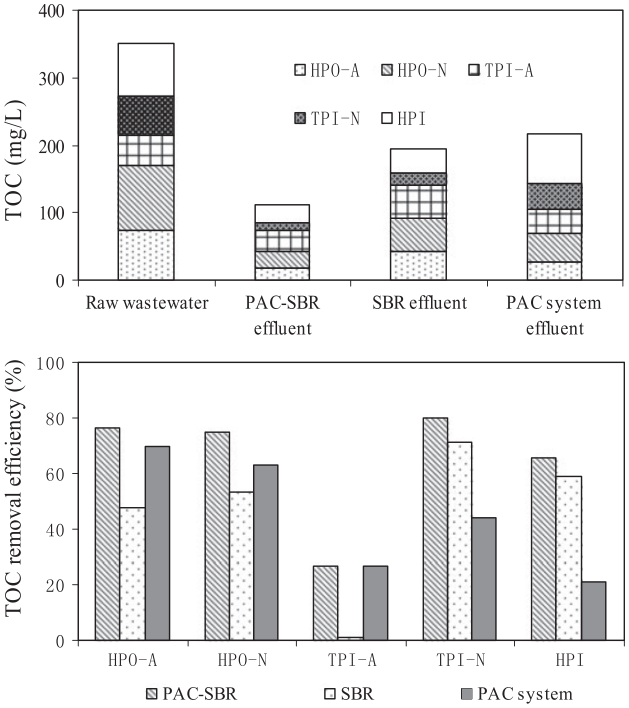

Fig. 7. Organic matter fractions of the raw wastewater and the effluent samples from the three systems (upper) and the removal efficiencies of the organic matter polarity distribution for these systems (lower). Values are given as average values with $n = 3$.

4. Conclusions

The PAC–SBR system was shown to effectively treat commingled chemical industrial wastewater. The use of PAC dosage and feeding frequency of 2 g PAC/L-reactor and 0.25 g PAC/L-reactor to 0.5 g PAC/L-reactor per 3–7 d, respectively, maintained a stable and long-term performance in the PAC–SBR system. The improvement of settling characteristics of AS by PAC addition prevented AS runoff caused by the toxic and refractory characteristics of organic matter in the industrial wastewater. Organic matter removal was also enhanced in the PAC–SBR system. The EEM spectrum of organic matter in the influents and treated effluents revealed that the compounds in raw wastewater that contain aromatic rings, heterocyclic ring structures, and bicyclic aromatic structures can be partially removed through biodegradation and PAC adsorption. In the PAC–SBR system, the removal of these compounds was enhanced by the combination of biodegradation and PAC adsorption. The organic acid-like compounds recalcitrant to biodegradation were adsorbed via PAC in the PAC–SBR system. Moreover, the results of organic matter fractionation showed that the compounds in the TPI-N and HPI fractions were easily transformed by biodegradation, whereas the compounds in the HPO-A and HPO-N fractions were adsorbed by PAC.

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