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Performance of a novel HABR–CFASR system for the biological treatment of mixed printing and dyeing wastewater (MPDW)

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ABSTRACT

Mixed printing and dyeing wastewater (MPDW) is characterized by a low-strength chemical oxygen demand (COD; average of around 1,000 mg/L), a high fraction of recalcitrant components, a low COD/SO_4^{2-} ratio (approximately 1.0), and a high pH (about 8–10 without adjustment). In this study, a biological system that combined a hybrid anaerobic baffled reactor (HABR) and a cross flow aerobic sludge reactor (CFASR) was successfully used to treat MPDW that was discharged from an industrial zone. The HABR–CFASR system was fed with real MPDW. In the start-up stage, the system had low organic loading rates of 0.66 and 0.45 kg COD/(m^3·d) for the HABR and CFASR, respectively. These increased to 2.03 and 1.00 kgCOD/(m^3·d), respectively, in the steady stage. The pH of the HABR influent was reduced by adding H_2SO_4 at a decreased dosage over the course of the experiment. Most of the final effluent from the combined treatment process was stable below 100 mg-COD/L and 20 mg-BOD/L after 12 h-HRT in the HABR followed by 20 h in the CFASR. The sulfate removal rate reached 17.59%, and the COD/SO_4^{2-} ratio varied slightly around 1.0, regardless of the Ns (sulfate loading rate), which varied between 1.5 and 2.5 kgSO_4^{2-}/(m^3·d). A GC/MS analysis demonstrated that the amount and types of organic compounds declined significantly after the HABR treatment.

Keywords: Mixed printing and dyeing wastewater (MPDW); Hybrid anaerobic baffled reactor (HABR); Cross flow aerobic sludge reactor (CFASR); Sulfate reduction

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1. Introduction

The textile industry is one of the most important industries in China and is responsible for the largest wastewater discharge [1]. More than 80% of textile industry wastewater is from the printing and dyeing industry [2–4]. In recent years, printing and dyeing enterprises have been concentrated in special industrial zones, and the vast majority of these industrial zones have their own centralized wastewater treatment plants

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(CWWTPs). Most of the factories in these industrial zones have sewage treatment facilities where the wastewater is pretreated before it is discharged to the CWWTPs. The wastewater generated by the factories is mainly characterized by a low strength (a chemical oxygen demand (COD) of around 1,000 mg/L) and weak biodegradability. In addition, the added chemical auxiliaries create biological toxicity [5,6].

The treatment of MPDW has been the focus of research for several decades, and many technologies have been developed to treat the contaminated wastewater. A fluidized bed reactor was developed for the anaerobic treatment of real textile wastewater [7]. Somasiri demonstrated how to remove the color and reduce the COD in real textile wastewater using an upflow anaerobic sludge blanket (USAB) reactor [8]. Firmino et al. removed the dyes from synthetic and real textile wastewaters using one- and two-stage anaerobic systems [9]. Kumar et al. compared the performance of different adsorbents in removing dye from textile industry effluent [10]. Gökkuş et al. used a Taguchi experimental design method to optimize chemical coagulation in the treatment of real textile wastewater [11]. Thanh et al. assessed the treatment efficiency and membrane fouling propensity of a submerged membrane bioreactor (MBR) for treating dyeing and textile wastewater by introducing powder-activated carbon and alum into the process [12]. Ellouze et al. treated synthetic textile wastewater with combined chemical coagulation/membrane processes [13]. However, the implementation of these methods has been seriously restricted by their uneven performance and unaffordable cost, particularly for industries in developing countries.

According to previous studies [14], many factors can inhibit the biological treatment process. One of the most significant is a high concentration of sulfate. Five main microbial groups are present in a sulfate-reducing reactor: acidogenic bacteria (AB), hydrogen-producing acetogens (HPA), hydrogen-utilizing SRB (HSRB), acetic acidutilizing SRB (ASRB), and fatty-acid-utilizing SRB (FSRB). The FSRB group includes propionic-acid-utilizing SRB (p-SRB), butyric-acid-utilizing SRB (b-SRB), and lactic-acid-utilizing SRB (l-SRB). It has been shown that anaerobic treatment processes do not effectively reduce sulfate in sulfate-rich wastewater such as MPDW [15].

It is necessary to find a method to effectively decontaminate MPDW that involves upgrading and retrofitting existing facilities, rather that developing entirely new systems. Unfortunately, research is usually focused on developing new techniques that require new facilities and equipment. There is a little research on the feasibility of upgrading and retrofitting existing facilities. In this study, a practical application of a two-part treatment technique that includes a HABR and a CFASR was tested as a remedy for MPDW. The technique was designed to decontaminate MPDW to satisfy the new stricter discharge standards and to verify the feasibility of upgrading and retrofitting existing facilities with a HABR–CFASR system. The performance of the HABR in degrading and removing recalcitrant organic compounds was the major concern. A gas chromatography/mass spectrometry (GC/MS) analysis was used to evaluate and compare the organic compounds in the influent and effluent of the HABR.

2. Materials and methods

2.1. Wastewater source and characteristics

The wastewater used in this study was real MPDW, consisting mainly of printing and dyeing wastewater (about 80%), mixed with other industrial sewage (including brewery, chemical, pharmaceutical, tannery, food, and electroplating wastewater) and municipal sewage. The influent fed into the combined biological system was characterized by a COD range of 600–1,600 mg/L, a low COD/SO_4^{2-} ratio (approximately 1.0), and a high pH range (an average value of approximately 9.1). The MPDW was pretreated by coagulation/sedimentation (with Fe_2SO_4 as the coagulant) before it was input into the experimental installation.

2.2. Combined biological process

The experimental installation combined an HABR and a CFASR. The wastewater was put into the HABR first, after adjustment of the quality with a regulating pond. The effluent of the HABR flowed into a middle sedimentation tank and was then gathered into another tank and pumped into the CFASR. After processing, the CFASR effluent flowed into a secondary sedimentation tank where the sludge and wastewater were separated, and the effluent was directly discharged into natural waters.

2.2.1. HABR and sludge inoculation

A schematic representation of the experimental system is shown in Fig. 1. The HABR had a working volume of approximately 18 m^3 with a length of 3,500 mm, a width of 1,500 mm, and a height of 3,600 mm. The top half of the reactor was filled with wave-shaped epoxy glass cells (cell thickness, 1 mm; crest height of the wave, 45 mm; crest distance of the wave, 100 mm; slant distance of the wave, 130 mm; and specific surface area, 360 m^2/m^3). These patented

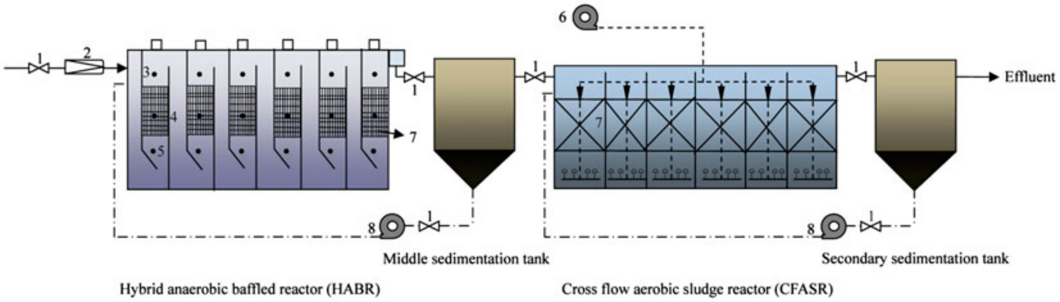

Fig. 1. Schematic diagram of the installation. 1. Valve; 2. Flow meter; 3. Wastewater port; 4. Online detector port; 5. Sludge port; 6. Air blower; 7. Fillings; 8. Return sludge pump.

fillings were used in our previous research and retained adequate sludge in the reactor. The HABR had six compartments, each of which had two sampling ports, one in the upper part and one in the lower part. The upper port was a wastewater sampling port and the lower port was a sludge sampling port. There was also a measuring and controlling port in the middle profile. Each compartment was divided into two portions by a baffle. One of the compartments was a downflow room and the other one an upflow room that was equivalent to an UASB. The width ratio of the two rooms was 1:7. A 30° angle at the bottom of the baffle ensured that the wastewater and sludge were adequately mixed.

The HABR was inoculated with sludge from the sludge storage tanks, which was made up of mixed liquor volatile suspended solids (MLVSS) at 35 g/L. The inoculation quantity was approximately 50% of the working volume of the HABR.

2.2.2. CFASR and sludge inoculation

The CFASR had a working volume of 21 m^3 with a length of 3,000 mm, a width of 2,000 mm, and a height of 4,000 mm. The submerged fillings were the same as those used in the HABR. The core component of the CFASR is the patented cross flow fillings (Patent No. 96251960.X) invented by Ren et al. The special formation of the fillings causes wastewater to cross flow through them. This turbulent flow ensures that there is sufficient contact between the wastewater and the sludge. The special structure and fillings give the CFASR many advantages in remedying recalcitrant industrial wastewater. The CFSAR's resistance to shock loading is superior and it is more efficient than other aerobic systems.

The seeding sludge obtained from the return sludge from the secondary clarifier pool was used for micro-organism inoculation and acclimation. The seeding quantity was approximately one-sixth of the working volume of the CFASR. The mixed liquor suspended solids (MLSS) and the MLVSS of the seeding sludge were 1,358 mg/L and 737 mg/L, respectively. Wastewater and compound fertilizer were then input to adjust the biological oxygen demand (BOD)/N/P ratio to approximately 100:5:1.

2.3. Operational conditions

A 213-day test was conducted in three stages for the HABR and two stages for the CFASR. The HABR stages (I - startup stage, II - steady stage, and III - high-pH stage) were defined according to the influent pH range and the organic loading rate (OLR). In the first 20 days, the HABR had a constant HRT value of around 60 h, which was reduced to 40 h in the following 14 days and stabilized at approximately 24 h from day 35. The pH was adjusted to 7–8 by adding H_2SO_4 for the first 150 days. The OLR was gradually increased by raising the influent flux in the steady stage and the corresponding HRT operated at around 22, 20, 18, 16, 14, and 12 h . To explore how well the HABR performed when processing an influent with a high pH (simulating the actual situation), the amount of H_2SO_4 added was reduced or abolished after day 151, and the pH was allowed to vary from 8 to10.

The test period for the CFASR was divided into two stages: (1) start-up stage and (2) steady stage. The CFASR was fed with dilute wastewater for the first 10 days; the HRT was approximately 40 h throughout this stage. To determine feasible parameters for the system, six HRTs (40 h, 35 h, 30 h, 25 h, 20 h, and 15 h) were tested in stage II, with each test lasting 30 days. The HABR and CFASR were coupled from day 151. The operating conditions for the HABR–CFASR system are summarized in Table 1.

Table 1
Operational conditions of the HABR–CFASR system

HABR

Stage	Time (d)	HRT (h)	SRT (d)	pH
I	1–20	60		
	21–34	40	50	7–8
	35–47	24		
II	48–56	22	100	7–8
	57–65	20		
	66–72	18		
	73–82	16		
	83–108	14		
	109–150	12		
III	151–213	12	100	8–10

CFASR

Stage	Time (d)	HRT (h)	SRT (d)	DO (mg/L)	Sludge recycle ratio (%)
I	1–30	40	10	2–4	40
II	31–60	40			
	61–90	35			
	91–120	30	5–10	2–4	20–40
	121–150	25			
	151–180	20			
	151–213	15			

2.4. Analytical methods

The pH and dissolved oxygen (DO) were measured with a pH meter (pHS-3C, China) and a DO meter (JPB-607, China), respectively. The COD, BOD, volatile fatty acids (VFAs), sulfate, MLSS, and MLVSS were determined according to the standard methods [16].

The organic constituents were analyzed with Agilent 6890N/5973N GC-MS. The capillary column model number was DB-5 ms (30 m × 0.25 mm × 0.25 μm). The GC was equipped with an injector at 280℃. The temperature control program registered an initial 45℃, which was retained for 3 min, increased to 200℃ in increments of 10℃/min and then increased to 310℃ in increments of 25℃/min. The final temperature was retained for 10 min. The helium carrier had a flow rate of 1 mL/min. The organic compounds were identified by searching the NIST05a library.

3. Results and discussion

3.1. HABR performance

In the first 47 days, the HABR influent COD concentration fluctuated substantially from 676 to 1,166 mg/L, with corresponding COD loading rates of 0.36–1.17 kgCOD/(m^3·d) (Fig. 2(b)). In line with this variation in the influent strength, the HABR effluent COD varied from 514 to 980 mg/L. The daily effluent samples at this stage always mixed a high concentration sludge, which resulted in an uneven COD removal effect by the HABR. This implied that although the HABR startup had a low OLR, the seeding sludge still required an adjustment period to adapt to the influent [17,18].

In the later stage of the experiment (days 49–150), the COD removal rate varied between 1.63 and 36.95%, whereas the HABR influent COD concentration fluctuated from 744 to 1,566 mg/L. The OLR in this stage was in the range of 0.93 to 2.66 kg COD/(m^3·d), with an average value of 1.70 kg COD/(m^3·d). The average COD removal rate was approximately 15% (Fig. 2(b)) with an average OLR of 1.89 kg COD/(m^3·d) from day 151. Fig. 2(c) indicates that the average BOD removal rate in the first stage (day 1–48) was 14.90%, and this increased to 23.64% after day 49. The average BOD removal rate decreased to 9.16% after day 150, indicating that increasing the OLR significantly affected the ability of the HABR to remove easily degradable substances. The HABR was running during the hydrolytic/acidification stage, when part of the BOD was metabolized by the micro-organisms. However, some BOD was also produced by the degradation of the recalcitrant organic compounds.

As can be seen in Fig. 2(d), the BOD/COD ratio of the influent ranged from 0.21 to 0.76, with an average value of 0.51 over the entire experimental period. The BOD/COD ratios fluctuated irregularly during the startup stage and then increased by 0.03 between day 49 and 150 and by another 0.07 after day 151.

Fig. 3(a) shows the variation in VFA in the HABR influent and effluent. Most of the influent organic matter was degraded into VFAs and other minor fermentation products. Regardless of the presence of other minor fermentation products, VFAs can be used to assess the biodegradability of an effluent. The effluent VFA concentration was calculated using the following simplified equation:

$$\begin{aligned}\text{Effluent VFA} = {} & \text{Influent VFA} \\ & + \text{Generating VFA} \\ & - \text{Consuming VFA}\end{aligned} \tag{1}$$

The influent VFA concentration fluctuated between 95 and 255 mg/L, as shown in Fig. 3(a). Most of the effluent concentrations were higher than the influent concentrations in the initial 30 days, indicating that the amount of VFA generated by the HABR was greater than the amount consumed. This suggests that acidification bacteria were distributed throughout the

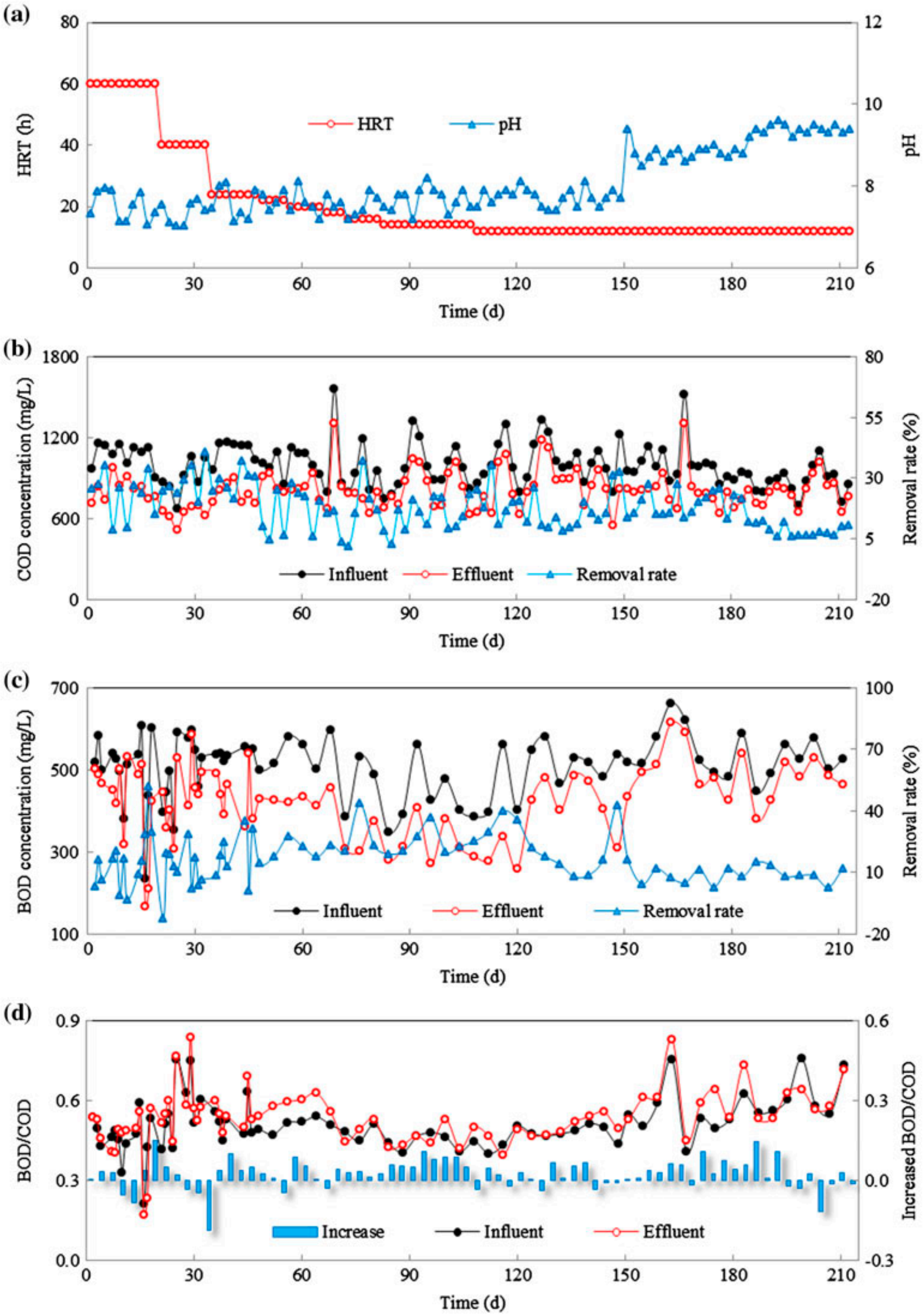

Fig. 2. Variation in the operational parameters and organic pollutants of the HABR (a) HRT and influent pH; (b) Influent and effluent COD; (c) Influent and effluent BOD; (d) Influent and effluent BOD/COD.

reactor and that their activities improved continuously during that period. However, a decrease in the effluent VFA concentration was found with a substantial increase in the OLR of the HABR effluent after day 35; the concentration ranged from 77 to 230 mg/L with an average of 140 mg/L. The HABR reached a steady state in this stage because although the acidification process occurred in the front compartments, the generated VFA was consumed in the later compartments by other micro-organisms, such as sulfate reduction bacteria (SRB) and methane-producing bacteria (MPB). This finding is consistent with the study of Lei et al. [19], in which the VFA concentration decreased as the OLR increased in a hydrolysis/acidification process. Bayrakdar et al. [20] also reported that the VFA concentration decreased as wastewater moved through the reactor.

The sulfate concentration was overly high for the influent COD concentration. Sulfates stem from three major processes: (a) they are generated by various businesses and thus exist in raw sewage; (b) from the $FeSO_4$ used in the pretreatment before the biological process; and (c) from the H_2SO_4 added to the wastewater to adjust the pH. SRB can remove sulfates through anaerobic processes and suppress MPB through competition for substrates such as hydrogen and acetate and consequently result in a primary inhibition to MPB [21,22].

The sulfate concentration fluctuated in the range of 821–1,230 mg/L, as shown in Fig. 3(b), which corresponded to a COD/SO_4^{2-} ratio (C/S) of around 0.7 to 1.5 with an average of 1.0. Parallel to this variation in

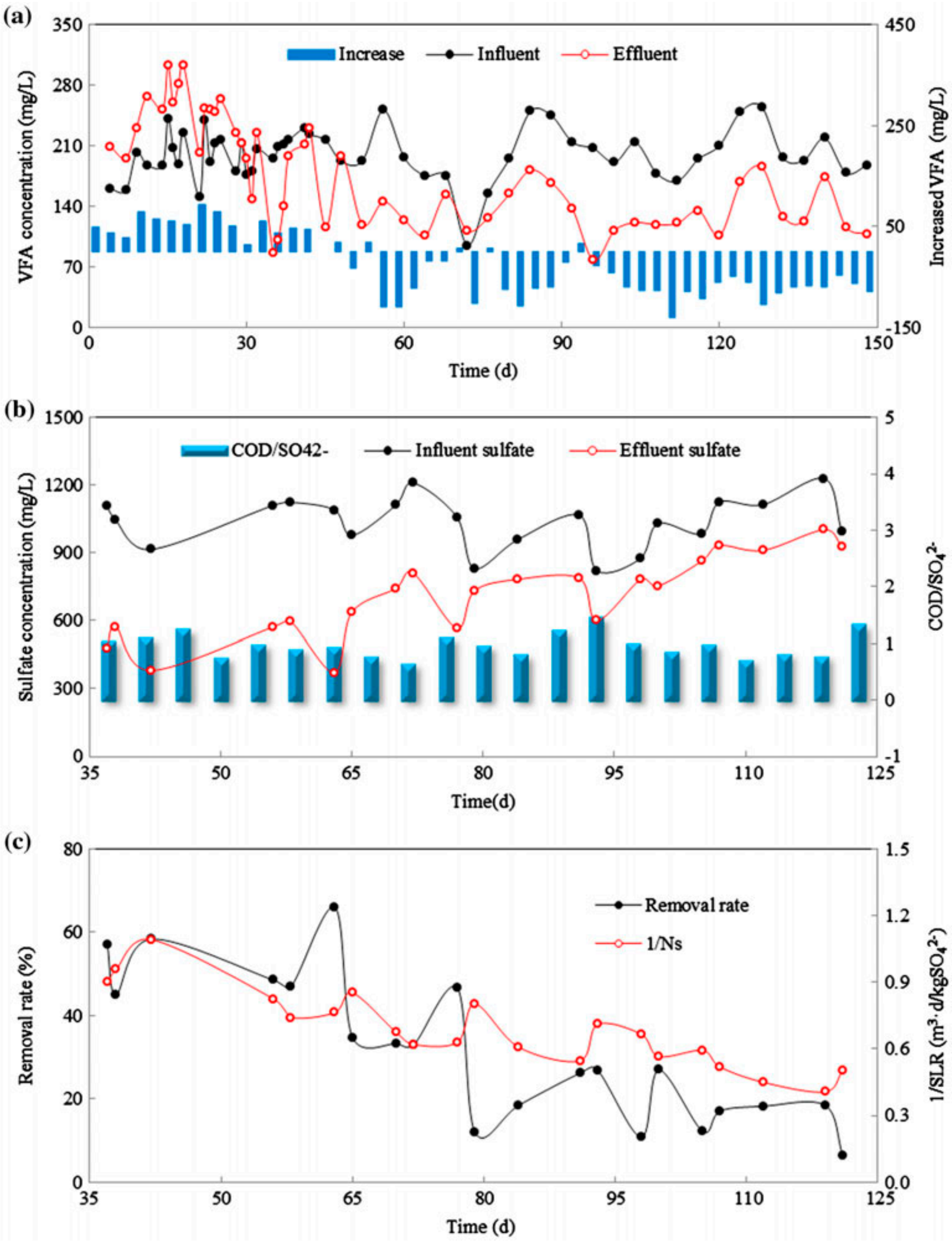

Fig. 3. Variation in VFAs and sulfates in the HABR (a) Influent and effluent VFA; (b) Influent and effluent sulfate and influent COD/SO_4^{2-}; and (c) Variation tendency of the sulfate removal rate and 1/Ns.

the influent strength, the sulfate-loading rate (Ns) varied from 0.92 to 2.46 kg $SO_4^{2-}/(m^3{\cdot}d)$. Many previous studies have explored the influence of the COD/SO_4^{2-} ratio and Ns on sulfate removal. Wang et al. [14] explored the influence of COD/SO_4^{2-} on sulfate reduction in a continuous flow in an acidogenic reactor using molasses wastewater as the carbon source and found that sulfate was nearly completely removed when the COD/SO_4^{2-} ratio was above 2.7. Mockaitis et al. [23] achieved sulfate reduction rates of 58% (fill time of 3 h) and 55% (fill time of 6 h) in an ASBR with a COD/SO_4^{2-} ratio of 0.67. The COD/SO_4^{2-} ratio needed to be above 0.67 to completely reduce the sulfate [24]. Zhao et al. [25] demonstrated that when the COD becomes a limiting restricted factor, the sulfate reduction efficiency drops sharply: the sulfate reduc-

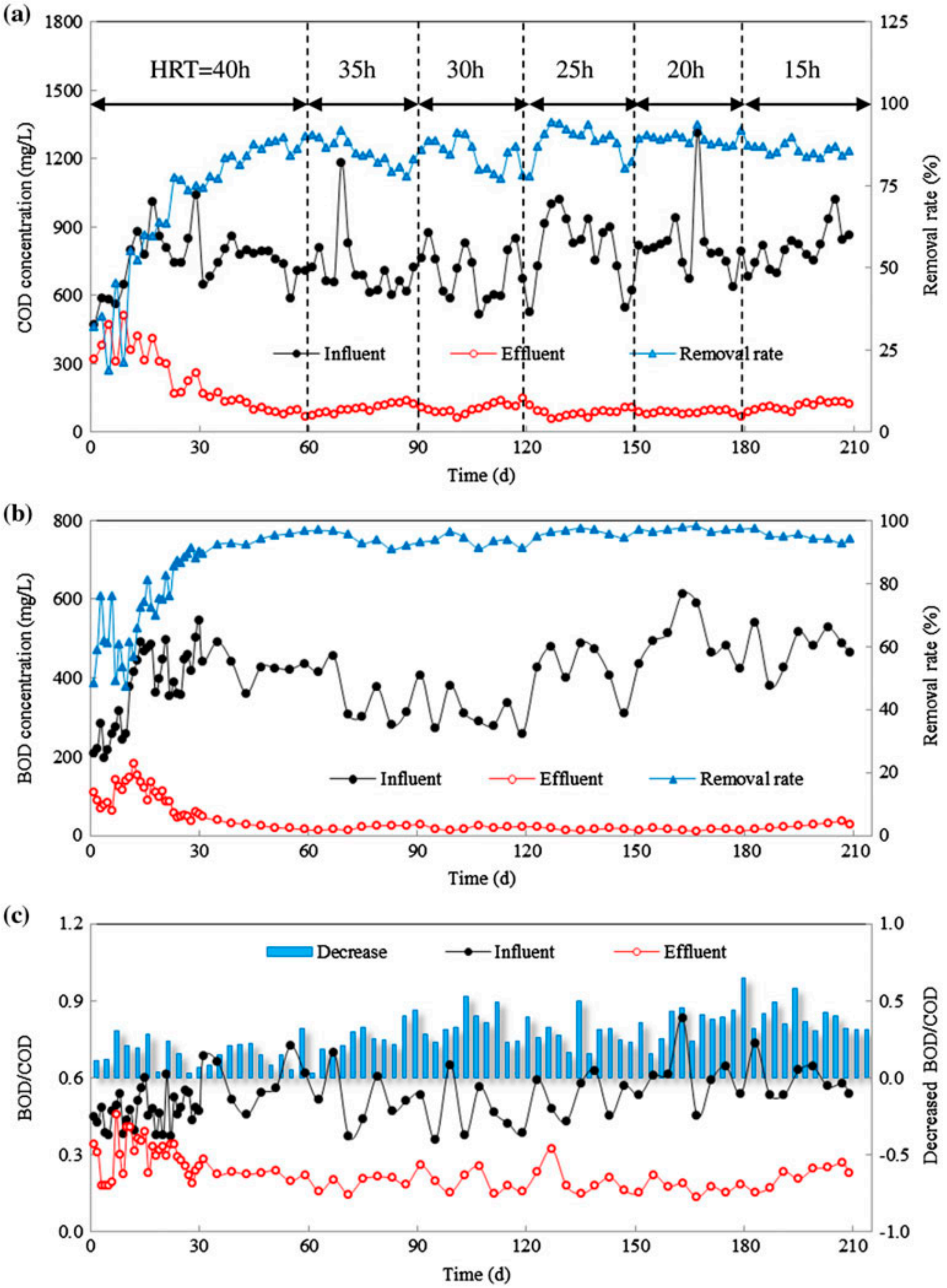

Fig. 4. Performance of the CFASR (a) HRT and influent and effluent COD; (b) Influent and effluent BOD; and (c) Influent and effluent BOD/COD.

tion efficiency decreased to about 20% when the COD/SO_4^{2-} ratio changed to 0.5.

In this study, the sulfate removal rate varied between 32.98 and 66.04% in the first 80 days with an average value of 46.95%, and then stayed at a relative steady level. After day 81, the removal rate was in the range of 6.44 to 26.96% with an average of 17.59%. Fig. 3(c) clearly shows that the sulfate removal rate continuously declined as the Ns increased (due to the reduction in HRT) during the experimental period, and that the sulfate removal rate was very similar to the trend in the 1/Ns. Most of the Ns exceeded 1.5 kg SO_4^{2-}/(m^3·d) after day 81. This suggests that the SRB acclimated to the new circumstance better than the MPB in the initial stage, when approximately half of the sulfate was removed from the influent. Once the HABR reached the steady stage, the SRB gradually lost its competitive edge and the MPB became dominant. As a consequence, the sulfate removal rate in this stage varied within a relatively low yet steady range, and the VFA concentration in the HABR effluent was lower than the influent.

3.2. CFASR performance

In the start-up stage (first 10 days), the CFASR was fed with the diluted wastewater. (The influent used in the start-up stage of the CFASR was the HABR effluent diluted with tap water.) During the first 10 days, the COD concentration in the influent fluctuated between 470 and 646 mg/L (Fig. 4(a)), corresponding to COD loading rates of 0.28–0.39 kg COD/(m^3·d). This gradually increased to an average of 1.0 kg COD/(m^3·d), when the HRT was decreased from 40 to 20 h between days 31 and 180. During this period, the influent fluctuated between 514 mg-COD/L and 1,310 mg-COD/L. Most of the COD removal rates of the CFASR were above 80% from day 45 onwards, with an average value of 86.79%. With the improvement in the HABR effluent, the CFASR effluent COD concentration also mostly improved. The COD concentration was less than 100 mg/L when the HRT was kept around 20 h, and the effluent concentration decreased to around 130 mg COD/L when the HRT was further reduced to 15 h. The effluent quality in this study met the discharge standard when the HRT

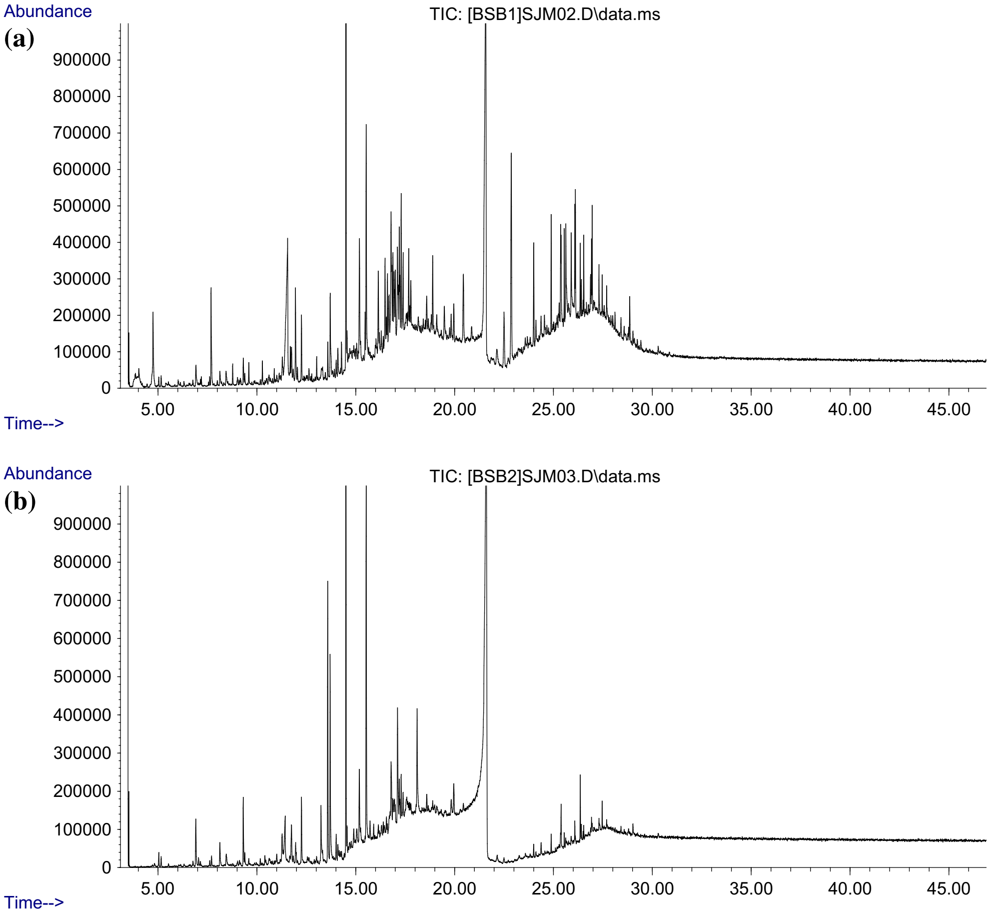

Fig. 5. GC/MS chromatograms of organic compounds in the influent (a) and effluent (b) of the HABR.

was fixed around 20 h. In that sample, the corresponding OLR was approximately 1.0 kg COD/(m^3·d).

The influent concentration of the CFASR varied from 198 mg to 316 mg BOD/L in the first 10 days, as shown in Fig. 4(b). In line with the changes in the influent BOD, the CFASR effluent fluctuated substantially between 62 and 141 mg BOD/L, corresponding to a BOD removal rate of 48.33–76.06%. The unsteady BOD removal was probably due to incomplete microbial acclimation. Most of the effluent BOD was less than 25 mg/L from day 47, with an average removal rate of 95.16%. The effluent BOD clearly increased from day 191 as the HRT declined.

The decrease in the BOD/COD ratio can be clearly seen in Fig. 4(c). It varied from 0.18 to 0.46 in the effluent during the initial 30 days. This relatively high value is an indicator that the CFASR was still unsteady. The average BOD/COD ratio then decreased to 0.20 in the later experimental period, demonstrating that most of the easily degradable contaminants were removed from the wastewater by the combined system.

3.3. Organic compound analysis of HABR by GC/MS

To further investigate the removal of different organic compounds and their biodegradability by the HABR system, a GC/MS analysis was performed. The chromatograms of the processed influent and effluent are shown in Fig. 5. Further details of the organic components are presented in Table 2. A comparison of the chromatograms of the HABR influent and effluent clearly shows that there are more peaks in Fig. 5 (a) than in Fig. 5(b) and that the response abundance presents a decreasing trend. Furthermore, the peaks related to retention times longer than 20 min are centralized in Fig. 5(a), showing a significant decline over the graph. This finding indicates that a large number of intricacy compounds were degraded or removed from the wastewater by the HABR. Table 2 shows that the influent of the HABR was complex, with 84 different types of compounds. The main constituents were alkanes (17 types), anilines (9 types), esters (8 types), organic acids (6 types), ketones (5 types), and phenols (5 types). Many of these organic compounds are complex and nonbiodegradable, giving the wastewater weak-biodegradability. If these contaminants remained in the MPDW effluent, then they might cause serious environmental problems. Unfortunately, many traditional processes do not remove these compounds. Table 2 shows that the number of categories of organic compounds was reduced to 44 in the HABR effluent. Furans, alcohols, pyridins, aldehydes, and oximes, which were present in the influent, were undetected in the HABR effluent. The number of types of alkenes increased significantly (from 2 to 7) during the HABR process, whereas there were fewer types (17 in the influent and 3 in the effluent), indicating that some recalcitrant compounds, such as straight-chain and branched-chain alkanes, were decomposed to simple and easily biodegraded compounds. Many studies have demonstrated that an abundance of alkanes degraders are involved in the microbial degradation of alkanes [26,27]. The GC/MS analysis results proved that in an advanced anaerobic process (e.g. a HABR), many refractory constituents are decomposed, which results in some new intermediates that are more readily degraded in the subsequent aerobic unit. These results explain why the biodegradability of the wastewater improved significantly as it passed through the HABR.

Table 2
GC/MS analysis of the influent and effluent of the HABR

Organic compounds	Categories (types)	
	Influent	Effluent
Alkanes	17	3
Cyclanes	2	1
Alkenes	2	7
Phenols	5	4
Anilines	9	8
Benzenes	3	1
Ketones	5	1
Furans	1	ND
Quinolines	3	3
Sulfurs	2	1
Organic acids	6	3
Alcohols	2	ND
Pyridins	1	ND
Amides	2	ND
Indoles	4	3
Esters	8	5
Aldehydes	1	ND
Benzothiazoles	1	1
Acridines	1	1
Oximes	1	ND
Others	8	2
Total	84	44

Note: ND means non-detectable.

4. Conclusion

The HABR–CFASR system had a successful start-up, with a low OLR of 0.66 and a 0.45 kg COD/(m^3·d) that reached 2.03 and 1.00 kg COD/(m^3·d), respectively, in the steady stage. The COD and BOD in the effluent were mostly below 100 and 20 mg/L after 12 h-HRT in the HABR and 20 h in the CFASR, respectively. The sulfate removal rate reached 17.59% and the COD/SO_4^{2-} ratio changed to around 1.0, regardless of the variation in Ns (between 1.5 and 2.5 kg $SO_4^{2-}/(m^3 \cdot d)$). The GC/MS analysis suggested that many recalcitrant organic compounds were removed or degraded by the HABR. These results demonstrate that a combined biological treatment system is a feasible and stable technique for upgrading and retrofitting CWWTPs.

Acknowledgments

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