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SIMULTANEOUS TREATMENT OF SULFATE WASTEWATER AND DOMESTIC SEWAGE WITH MICRO-AERATION

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Abstract

High sulfide inhibited the activities of sulfate-reducing bacteria (SRB) and methane-producing bacteria (MPB). In addition, a large number of domestic sewages should be treated in China. Thus, it is the first time that domestic sewage was collected to dilute high-sulfate wastewater in this study to achieve simultaneous treatment of sulfate wastewater and domestic sewage in the anaerobic baffled reactor (ABR). The results showed that there were obvious separation of sulfate reduction phase and methane production phase in the ABR, and large sulfate were reduced in the front compartments. Micro-aeration (oxygen demand of 0.4-0.6 mg/L) could significantly decrease sulfide concentration from 45 mg/L to 5 mg/L. Furthermore, micro-aeration between sulfate-reducing and methane-producing phases could significantly promote the synergistic removal of organics by SRB and MPB, while COD removal efficiency increased from 80% to 95%, and sulfate removal efficiency increased from 80% to 90%. And the highest elemental sulfur recovery rate was 0.27 kg/(m^3·d). Finally, the ABR that operated with C/S of 2, HRT of 12 h and COD load of 4 kg/ (m^3·d), micro-aeration (DO of 0.4-0.6 mg/L) at the end of sulfate reduction phase (the compartment 5) has proved to be efficient in simultaneous sulfate wastewater and domestic sewage treatment and is suitable for elemental sulfur recovery.

Key words: anaerobic baffled reactor, dissolved oxygen, sulfate-reducing bacteria, methane-producing bacteria, synergy

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1. Introduction

Paper, food processing, leather, pharmaceutical and other industries discharge a large amount of organic wastewater containing sulfate (Lens and Pol, 2000). If directly discharged, the wastewater will cause a wide range of environmental pollution. To date, quite a lot of efforts have been made to treat the sulfate-rich wastewater. Biological method is widely used for sulfate-rich wastewater treatment due to the relatively low cost and energy consumption (Liang et al., 2013). The formation of high concentration of hydrogen sulfide and other sulfide compounds hinder the wastewater treatment and the production of methane gas (Zub et al., 2008). It is necessary to investigate the removal performance of biological process in low-sulfate wastewater treatment. Moreover, many issues such as processes often encountered in wastewater treatment of low pH of sulfate wastewater, substrate competition between sulfate-reducing bacteria (SRB) and methane producing bacteria (MPB), the inhibition of sulfides

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on the activities of SRB and MPB. Although the activities of SRB were inhibited at high oxygen demand (DO) concentration, while limited aeration had no serious inhibition (Xu et al., 2013). However, the hydrogen sulfide was blow off and the sulfide was oxidized when limited aeration was used (Zhou et al., 2007). Thus, the weakened inhibition of sulfide on MPB improved the chemical oxygen demand (COD) removal. Tana et al. (2018) have reported research results for wastewater biological treatment promoted by SRB. Sequencing batch biofilm reactor shows clear technical and biological advantages over others configurations (Saba et al., 2017; Sarti et al., 2010). However, the advantage of anaerobic baffled reactor (ABR) as a third-generation anaerobic reactor satisfies the design concept of staged mufti-phase anaerobic reactor (SMPA) (Barber and Stuckey, 1999; da Silva et al., 2018), could effectively weaken the competition for substrates between SRB and MPB through creating suitable microbial growth conditions in different compartments for different populations. It is beneficial to sulfate reduction. And the generated toxic sulfide was used for the production of power and value-added elemental sulfur (Chen et al., 2018).

In addition, the proportions of domestic sewage treated are as low as 18.1% for county towns and 4.9% for rural villages in China by 2009 (Gong et al., 2012). The untreated wastewater discharged contains excess nutrient compounds, leading to the eutrophication of receiving waters and potentially threatening the safety of drinking water resources. In order to achieve the simultaneous treatment of sulfate wastewater and domestic sewage, the domestic sewage was collected to dilute high-sulfate wastewater to decrease the formation of high concentrations of sulfur compounds to hinder wastewater treatment and the production of methane gas in this study (Sarti et al., 2009). The performance of the ABR treating sulfate wastewater under middle temperature (33±0.5 ℃) with different positions of aeration was investigated to optimize the sulfate wastewater treatment and reduce the cost in practical application. Effects of various loads of sulfate and COD, hydraulic retention time (HRT) on conversion of SO_4^{2-}, COD, S^{2-}, and pH in each compartment were evaluated during start-up of eight months. In addition, the impact and effectiveness of micro-aeration on synergistic effects of organic matter removal by SRB and MPB were analyzed.

2. Material and methods

2.1. Wastewater

In order to simultaneous treat sulfate wastewater and domestic sewage, domestic sewage was collected to dilute high-sulfate wastewater in this study. The simulated influent was a mix of raw and synthetic wastewater that had an average COD concentration of approximately 2000 mg/L and C: S: N: P of 200:100:5:1, respectively. The selected synthetic wastewater contained glucose, ammonium chloride, dipotassium hydrogen phosphate and potassium dihydrogen phosphate, which were used as the primary organic, nitrogenous and phosphorous components, respectively. A solution of the trace elements (Zhang et al., 2011) was added to sustain the microbial growth. The trace elements composed of: 5.0 g/L EDTA, 5.0 g/L $FeSO_4 \cdot 7H_2O$, 0.99 g/L $MnCl_2 \cdot 4H_2O$, 0.19 g/L $NiCl_2 \cdot 6H_2O$, 0.011 g/L H_3BO_4, 0.2 g/L $ZnCl_2$, 0.12 g/L $Na_2MoO_4 \cdot 2H_2O$, 0.24 g/L $CoCl_2 \cdot 6H_2O$. Raw wastewater from a campus housing facility's sewer line was pumped into a storage tank for sedimentation, and then mixed into the synthetic wastewater.

2.2. The ABR setup

The ABR used in these experiments is shown in Fig. 1. The reactor, consisting of 11 compartments, was made of perspex with a total effective volume of 12 L. The ABR was inoculated with anaerobic sludge collected from the Municipal Wastewater Treatment Plant (Suzhou, China). The temperature of the process was maintained around 33 ± 0.5 °C. In addition, the membrane cloth (PVDF) was immersed in the top liquor of each compartment to collect sulfur every week.

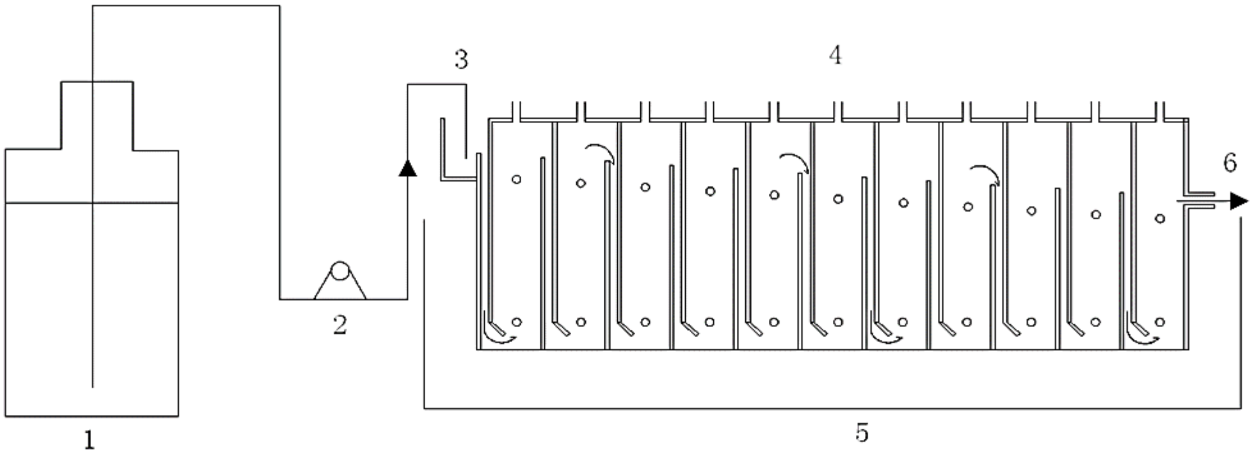


Fig. 1. Schematic diagram of the experimental ABR: (1) Feed tank; (2) pump; (3) influent; (4) ABR; (5) constant temperature water bath; (6) effluent

2.3. Experimental procedure

In order to simultaneous treat sulfate wastewater and domestic sewage effectively, and enrich SRB and MPB, the ABR should be started up. The start-up of the ABR with C/S of 2:1 consisted of three stages. For the first stage (days 1-50), the influent COD load was 2 kg/(m^3·d) with HRT of 24 h. For the second stage (days 51-80), the influent COD load was 2 kg/(m^3·d) with HRT of 12 h. For the third stage (days 81-110), the influent COD load was increased from 2 kg/(m^3·d)to 4 kg/(m^3·d) with HRT of 12 h.

The sulfate reduction phase and methane production phase were effectively separated after start-up. Sulfate reduction mainly occurred in compartments 1-4. And there was no obvious sulfate reduction after the compartment 5, thus the initially micro-aeration (0.4-0.6 mg/L of DO) was controlled in the compartment 5. In addition, the position of micro-aeration was timely adjusted according to the changes of sulfide in each compartment. Then, effects of micro-aeration on sulfate reduction and methane production were investigated during days 111-230.

In this study, a steady-state condition was considered to be reached when the variation of the measurements was less than 10%. The average values of the data obtained under the steady-state condition were used for further calculations.

2.4. Chemical analysis

The concentrations of sulfate and sulfide were determined by the ICS-3000 ion chromatograph (Dionex Co., USA). Samples were passed through a syringe filter (0.45μm) before analysis. COD were measured by 5B-1 Fast COD Detection Instrument (Lian-hua Tech. Co., Ltd., China). The COD removal by SRB and MPB were calculated by assuming that the electron donor was completely oxidized by SRB (Eqs. (1-3)). The fraction of organic matter removed by MPB was calculated as the difference between total COD removed and COD – S^{2-}, corresponding to the quantity of COD removed by SRB.

$$\begin{aligned} &Total\ COD\ removal\ (COD_{Total})(\%) = \\ &= \left(\frac{(COD_{Influent} - COD_{Effluent})}{COD_{Influent}}\right) \times 100 \end{aligned} \quad (1)$$

$$\begin{aligned} &COD\ removal\ by\ SRB\ (COD - S^{2-})(\%) = \\ &= \left(\frac{0.67(Sulfate\ Influent - Sulfate\ Effluent)}{COD_{Influent} - COD_{Effluent}}\right) \times 100 \end{aligned} \quad (2)$$

$$\begin{aligned} &COD\ removal\ by\ MPB\ (COD - CH_4)(\%) = \\ &= [COD_{Total} - (COD - S^{2-})](\%) \end{aligned} \quad (3)$$

DO and ORP were continuously monitored by WTW, pH/oxi340i meter with DO and ORP probes (WTW Company, Germany), respectively. The pH and temperature were measured on-line by using WTW level 2 pH meters (WTW Company, Germany).

The thick layer of white sticky solid that was observed in each compartment by the S-4800 Field Emission Scanning Electron Microscopy (SEM) (Hitachi Ltd., Japan) equipped with Energy Dispersive X-ray Analysis (EDAX) (Genesis XM2) for the detection of elements. All of the tests were performed in triplicate.

2.5. Statistical analysis

T test was performed using Microsoft Excel 2007, and significance level (*p*) was determined.

3. Results and discussion

3.1. Start-up of the ABR

3.1.1. Changes in biomass and volatile fatty acids

The main changes in microbial is represented by a sludge change. The sludge in the first two compartments of the ABR increased rapidly during the start-up of five months. The sludge in the first compartment discharged two times, and once in the second compartment, while no discharge was determined in other compartments. The removal of COD mainly occurred in the first compartment when organic load was low. The level of organic removal in the back compartments could be improved through increasing the organic load. Granular sludge was formatted in the ABR after one month operation, and the maximum diameter was about 3 mm after four months operation. In addition, the activity of sludge in the front compartments was much higher than that of the back compartments.

Volatile fatty acids (VFA) mainly formed from hydrolytic degradation by organics acidification bacteria. Low concentration of VFA might not provide sufficient carbon for SRB and MPB, while high concentration of VFA easily results in acid accumulation. Thus, it is conducive to the normal operation of the ABR while maintaining a reasonable concentration range of VFA. The results showed that acidification has been basically completed in the first two compartments. It was related to the influent COD mainly constituted by small molecules organic. The concentration of VFA was highest in the first compartment under low organic load and gradually decreased in the later compartments. VFA increased in the first two compartments with the increase of the load, and also decreased in the later compartments. The ABR was normal during the start-up and the concentrations of VFA were between 4 and 16 mmol/L with no rancidity phenomenon, indicating that C/S of 2 and COD of 1000 or 2000 mg/L were conductive to the stable operation of the ABR.

3.1.2. Conversion of sulfate in each compartment

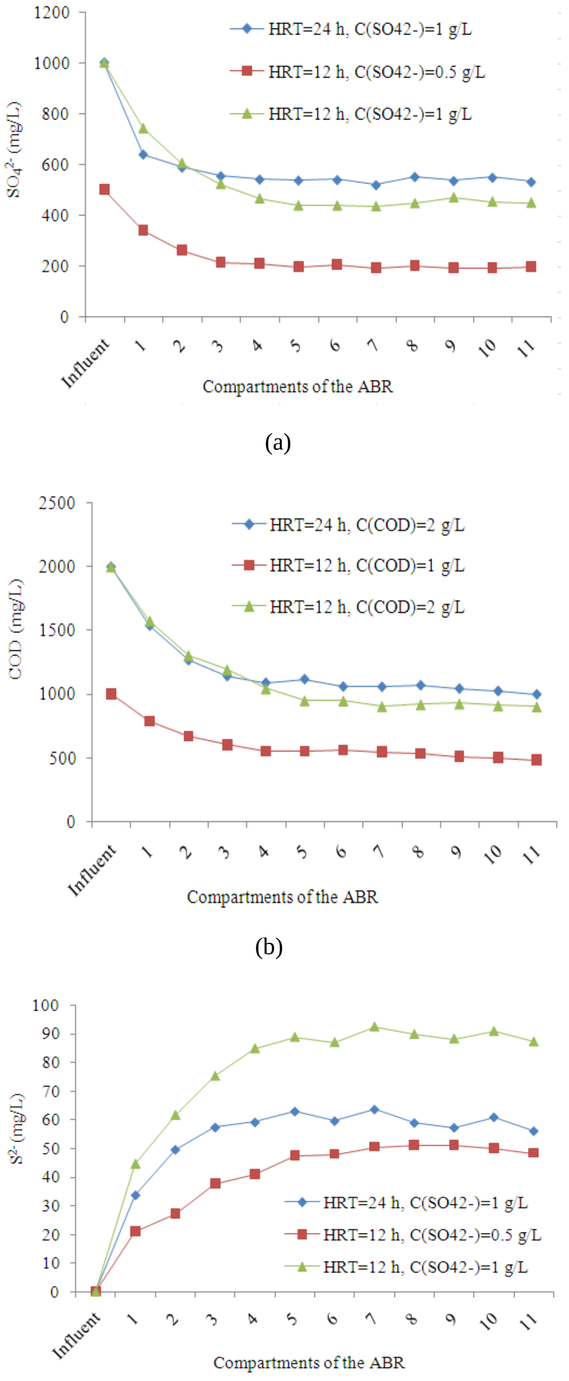
Fig. 2(a) shows the conversion of SO_4^{2-} at different HRT and influent concentration in each compartment of the ABR. The sulfate reduction (35%) mainly occurred in the first compartment with the HRT of 24 h, and significantly ($p<0.05$) decreased in the second (6%) and third (3%) compartment, finally with total removal of 48%. When the HRT reduced to 12 h, the total removal efficiency of sulfate was significantly ($p<0.05$) increased to 60% due to sufficient carbon source in the front compartments. In addition, the sulfur not only can be oxidized by the sulfur-oxidizing bacteria (SOB), but also can be reduced by the SRB. When carbon source was sufficient in the front compartments, it is beneficial to the growth of SRB. In addition, the concentration of sulfur and sulfide was very low, thus the activity of SOB was inhibited, mainly occurred the reduction of sulfate in the front compartments. However, the VFA gradually decreased in the front compartments, while the concentrations of sulfide increased. It is beneficial to the growth of SOB. Finally, the equilibrium between sulfide oxidation by SOB and sulfate reduction by SRB was achieved in the fifth compartment. It was also the reason why there was very low conversion of sulfate in the back compartments.

3.1.3. Conversion of COD in each compartment

The conversion of COD at different HRT and influent concentration in each compartment are shown in Fig. 2(b). There were mainly two ways that COD was consumed in the system. (1) The high molecules organic was degraded to VFA, and then were converted to methane by MPB. (2) VFA was consumed by SRB when sulfate was reduced to sulfide or sulfur. The COD was mainly removed in the first compartment of the ABR due to the influent COD mainly constituted of small molecules organics. In addition, the concentration of small molecules organic gradually decreased in the back compartments, and the inhibition of sulfur, a sulfur hydride and hydrogen sulfide on bacteria was increased, resulting in the lower removal efficiency of COD in the back compartments. The removal efficiency of COD in the front compartment significantly decreased with an increasing in sulfate-reducing, indicating that the COD were consumed mainly used as a carbon source for sulfate reduction in the front compartments and for methane production in the back compartments.

3.1.4. Conversion of sulfur ions in each compartment

There were no sulfur ions in the influent, thus the sulfide ions in the ABR were all produced from sulfate reduction by SRB. As shown in Fig. 2 (c), the sulfide significantly increased in the front compartments, and there were no obvious changes in the back compartments. In addition, a thick layer of white material that was observed in each compartment was elemental sulfur. It was mainly produced from the reduction of sulfate by SRB. Otherwise, the layer of sulfur was formed quickly and need to be collected once a week. And the elemental sulfur recovery rate was 0.27 kg/(m^3·d) when the influent SO_4^{2-} load was 1 kg/(m^3·d) with micro-aeration in the compartment 5, and 0.21 kg/(m^3·d) with micro-aeration in the compartment 4, as well as 0.12 kg/(m^3·d) with micro-aeration in the compartment 3.



(a)

(b)

(c)

Fig. 2. SO_4^{2-} (a), COD (b) and S^{2-} (c) conversion at different HRT and influent concentration in each compartment

Furthermore, a high concentration of sulfide has strong toxic effects on the bacterial communities. Sulfide concentration also increased with the sulfate increasing in influent. Previous research indicated that the inhibition on microbial activity was significantly increased with an increasing of sulfide, hydrogen sulfide and sulfur in the back compartments (Chen et al., 2013). It was the underline reason why sulfate and COD in the back compartments were difficult to removal. Thus, most of the influent sulfate ended up as dissolved sulfides and the accumulation of elemental sulfur, and the amount of hydrogen sulfide gas were trivial.

3.2. Effects of micro aeration on removal efficiencies of ABR

3.2.1. Effects of micro aeration on sulfide removal

Sulfide was a product of sulfate reduction by SRB in the ABR, and had a positive correlation to sulfate load of influent, while had negative correlation to C/S. Sulfide that mainly included H_2S, $S^{2-}$and HS^- in the system inhibited the activities of SRB (Zhou and Xing, 2015), otherwise H_2S play a major inhibition (Xu et al., 2011). Variations of S^{2-} without aeration or aeration in compartments 5, 4 or 3, respectively in steady state, were shown in Fig. 3(a).

When there was no aeration in the ABR, the concentration of sulfide significantly increased from the compartment 1 to 4, but the rate of the increase gradually decreased. In addition, there was no obvious increase after the compartment 5 with the final sulfide of 45 mg/L. The reason why sulfide significantly increased from the compartment 1 to 4 may be that the high concentration of sulfate in the compartment 1-4 provided sufficient substrate for SRB, and there were no obvious inhibitions on the activities of SRB due to low concentration of sulfide in the compartment 1-4. With the decrease of sulfate and increase of sulfide, the activities of SRB after the compartment 5 were both inhibited by the substrate and products, thus sulfide increased very slowly. It was necessary to notice that the sulfide concentrations in the aerated compartment were significantly ($p<0.05$) reduced to 5 mg/L, and then increased after the aerated compartment. However, the final sulfide (30-40 mg/L) was lower than that of no aeration. The reason may be that micro-aeration in the ABR resulted in (1) SOB transform S^{2-} to elemental sulfur (Chen et al., 2014), (2) parts of the sulfide were oxidized to thiosulfate or sulfate, as well as (3) hydrogen sulfide were directly stripping out of the system. However, the SRB in methane production phase still had the ability to reduce sulfate under the low sulfide condition. It indicated that micro-aeration in the ABR could effectively reduce the concentration of sulfide, especially in the compartment 5 with the lowest sulfide (30 mg/L). It may be relevant to that the low sulfate in the compartment 5 resulted in small amount of sulfate reduction, thus it was most beneficial to the removal of sulfide in the ABR while aerated in the compartment 5. It was also revealed that the sulfide of 30 mg/L has significant inhibition on the activities of the SRB in the system.

3.2.2. Effects of micro-aeration on the sulfate removal

When the sulfate load of influent was 2 kg/(m^3·d), variations of SO_4^{2-} without aeration or aeration in compartments 5, 4 or 3, respectively in steady state, were shown in Fig. 3(b). When there was no aeration in the ABR, the concentration of sulfate significantly decreased in from the compartment 1 to 4. However, there was no obvious increase in the removal efficiency of sulfate after the compartment 5 with the final removal efficiency of 80%. Otherwise, the final removal efficiency of sulfate significantly ($p<0.05$) increased to 90% with aeration in the compartments 5 or 4. However, the final removal efficiency of sulfate was 6% lower than that of no aeration.

The removal of sulfide mainly relies on the role of stripping in the micro- aeration (DO of 0.4-0.6 mg/L) conditions, and very low amounts of sulfur or sulfate were oxidized from sulfide. Since the micro-aeration significantly decreased the concentration of sulfide in the back compartments, and the inhibition of sulfide on the activities of SRB also decreased, thus the final removal efficiency of sulfate significantly increased while the ABR aerated in the compartment 4 or 5. However, the final removal efficiency of sulfate with aeration in the compartment 3 was lower than that of no aeration due to the sulfate in the first two compartments were not sufficiently converted to the sulfide, and the sulfate in the compartment 2 was still up to 400 mg/L.

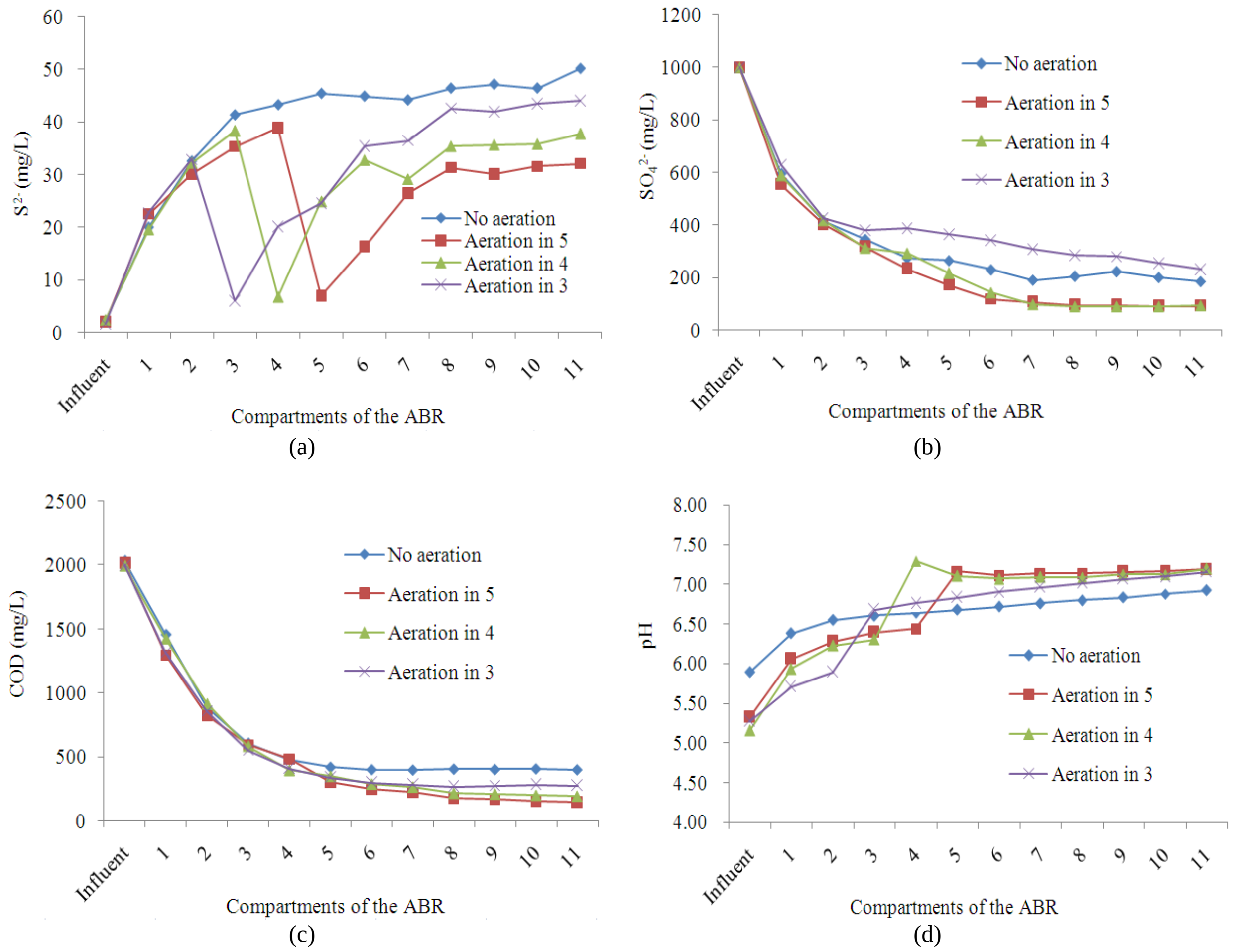
3.2.3. Effects of micro-aeration on COD removal

Variations of COD without aeration or aeration in compartments 5, 4 or 3, respectively in steady state, were shown in Fig. 3(c). The results showed that the final COD removal efficiency with no aeration was 80%, and the COD removal efficiency significantly ($p<0.05$) increased (85%-95%) with aeration in the compartment 5, 4 or 3.

Although the activities of SRB were inhibited by micro-aeration, COD can be consumed by aerobic bacteria due to the presence of DO. In addition, micro-aeration decreased sulfide concentration, and the inhibition of sulfide on MPB and SRB were also weakened, thus the COD were significantly consumed by MPB and SRB. Furthermore, aeration also promoted the degradation of the poorly biodegradable organics, and the produced VFA were easily utilized by SRB and MPB. The final COD removal efficiencies of micro-aeration in the compartment 5, 4 or 3 were 95%, 91% and 82%, respectively. The reason why the final COD removal efficiency of micro-aeration in the compartment 3 was the lowest may be that the lower amounts of sulfate reduction the smaller COD consumption by SBR.

3.2.4. Changes of pH in the aeration ABR

Although SRB can survive at low pH condition, while the influent pH of Sulfate wastewater was too low, it will inhibit the activities of SRB and MPB, leading to a decrease in removal efficiencies. Furthermore, under acidic conditions, the sulfide mainly composed of H_2S which has a serious inhibition on MPB and SRB.



(a) (b) (c) (d)

Fig. 3. Variations of S^{2-} (a), SO_4^{2-} (b), COD (c) and pH (d) concentration at different aeration positions in each compartment

Thus, the low pH is also an important factor that limited the use of biological technology to treat sulfate wastewater because the optimum pH for SRB removing sulfate was neutral (Wang et al., 2013).

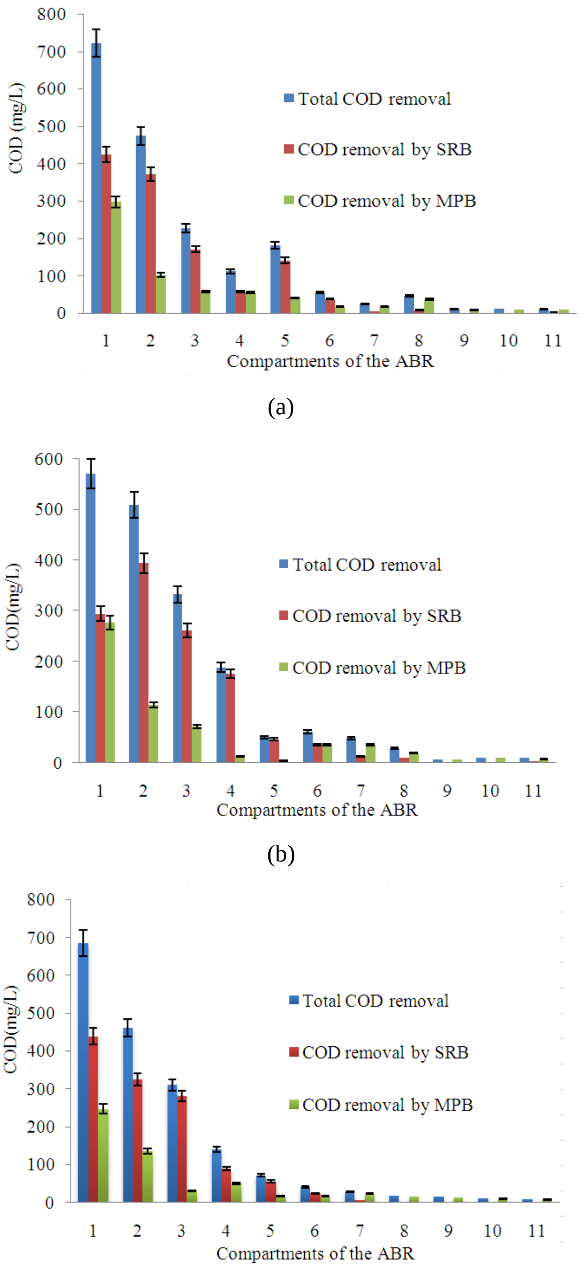
As shown in Fig. 3(d), the micro-aeration increased the pH of the back compartments. It may be that H_2S and VFA were striped out and oxidized by aeration. In addition, sulfide can be oxidized to sulfate by aeration, and the pH of the system will be reduced. However, while the compartment 3, 4 or 5 was aerated, the pH of the back compartments was higher than that of no aeration, indicating that the primary function of the micro-aeration was stripped out H_2S rather than the oxidized sulfide to sulfate. The results also showed that the ABR could effectively treat sulfate pollutants in a more wider range of pHs between 5 and 7 than that of previous study (Wang et al., 2013) that neutral condition was suitable for SRB to treat sulfate pollutants.

3.3. Synergistic COD removal by SRB and MPB

When the ABR aerated in the compartment 5, the removal efficiencies of COD by the SRB and the MPB in each compartment were shown in Fig. 4 (a). The results showed that the final COD removal was significantly increased in the compartment 5 and mainly relied on SRB, mainly due to the SRB can be survived in facultative anaerobic environment. However, the activities of MPB under micro-aeration condition (Do of 0.4-0.6 mg/L) were inhibited. It was the reason why the COD removal mainly relied on SRB in the compartment 5. In addition, the COD removal by SRB was higher than that by MPB mainly due to the rise of ORP in the compartment 6 when the ABR aerated in the compartment 5, and the MPB were also inhibited. Finally, the MPB in the back compartments with low sulfate gradually adapted to the high sulfide, and played a major role in COD removal.

When the ABR aerated in the compartment 4, the removal efficiencies of COD by the SRB and the MPB in each compartment were shown in Fig.4 (b). The results showed that the final COD removal was significantly increased in the compartment 4 and mainly relied on SRB. Furthermore, the COD removal by SRB was higher than that by MPB mainly due to the rise of ORP in the compartment 5. However, sulfates in the compartment 6-11 were low, and MPB played a major role in COD removal.

When the ABR aerated in the compartment 3, the removal efficiencies of COD by the SRB and the MPB in each compartment were shown in Fig. 4 (c). The results showed that the final COD removal was significantly increased in the compartment 3 and mainly relied on SRB. Furthermore, the COD removal in the compartment 4, 5 and 6 were also mainly relied on SRB. It may be that the COD and sulfate was still high, and the SRB acquired priority to utilize COD for the reduction of sulfate. Otherwise, sulfates in the compartment 7-11 were low, and sulfide which inhibited the activities of SRB was gradually increased (Sarti and Zaiat, 2011), thus MPB played a major role in COD removal.



(a)

(b)

(c)

Fig. 4. Removal of COD by aeration in compartment 5 (a), 4(b) or 3 (c)

4. Conclusions

The sulfate reduction phase and methane production phase could be effectively separated while the sulfate wastewater treated by the ABR with sulfate reduction in the front compartments and methane production in the back compartments.

Micro-aeration (DO of 0.4-0.6 mg/L) at the end of sulfate reduction phase could significantly decrease sulfide and effectively adjust the pH of the system, weakening the inhibition of sulfide on the SRB and MPB. Thus, the ABR has proved to be efficient in simultaneous sulfate wastewater and domestic sewage treatment and is suitable for elemental sulfur recovery.

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