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Treatment of tannery wastewater using a combined UASB (2 stage)-ozonation-BAF system

Fu Chen[a,b,*], Xiaoxiao Li[b], Zhanbin Luo[b], Jing Ma[a], Qianlin Zhu[a], Shaoliang Zhang[b]

[a]*Key Laboratory of Coal-based CO_2 Capture and Geological Storage, China University of Mining and Technology, Xuzhou, Jiangsu 221008, China, email: chenfu@cumt.edu.cn (F. Chen), jingma2013@cumt.edu.cn (J. Ma), zhuql@cumt.edu.cn (Q. Zhu)*
[b]*School of Environment Science and Spatial Informatics, China University of Mining and Technology, Xuzhou, Jiangsu 221008, China, email: lixiaoxiao@cumt.edu.cn (X. Li), lzbin1991@cumt.edu.cn (Z. Luo), slzhang@cumt.edu.cn (S. Zhang)*

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ABSTRACT

A four-stage lab-scale system was employed to treat tannery wastewater. This system was mainly comprised of a two-stage upflow anaerobic sludge blanket (UASB) reactor, an ozonation reactor, and a biological aerated filter (BAF) in series. The optimum operating conditions were found to be 42 h hydraulic retention time (HRT) for two-stage UASB, $[O_3]/[COD_0]$ of 0.2 for 50 min, and 20 h HRT for BAF. Under these conditions, the removal ratios of chemical oxygen demand, total suspended solids, oil-grease, total nitrogen, ammonia, and chromium were all higher than 90%. The water quality of final effluent could satisfy the national discharge standard of China set for the leather tanning industry. The sulfidogenesis and methanogenesis were separated in two sequential UASB reactors. The first UASB process removed a considerable proportion of sulfate, which alleviated the possible toxicity of sulfide to methane-producing bacteria in the second UASB. The ozonation process enhanced the biodegradability of UASB effluent, and finally, post-polish treatment was completed in the BAF. The combined process demonstrated a promising potential for treatment of high-strength tannery wastewater.

Keywords: Tannery wastewater; Sulfidogenesis; Methanogenesis; Ozonation; Microbial activity

1. Introduction

The leather tanning industry is one of the most significant pollution sources in terms of both conventional (such as color, smell, turbidity, pollutant levels) and toxic parameters [1]. China has the largest scale of leather manufacturing industry in the world with annual discharge of 200 million tones of wastewater from thousands of leather factories. Tannery wastewater has very complex compositions, which typically consists of high concentrations of salts, organic compounds, suspended solids, ammonia, sulfides, and toxic metals [2].

Coagulation–flocculation has been used as a pretreatment process prior to aerobic biological processes for tannery wastewater treatment [3]. Nevertheless, this process could produce large quantity of sludge, which requires further treatment. For the reason of reducing surplus sludge, anaerobic treatment was selected to transform the bulk of the organic load into biogas during the pretreatment of tannery wastewater. However, high contents of sulfate in the wastewater could be reduced to sulfide by sulfate-reducing bacteria (SRB) under anaerobic conditions, causing a poisonous effect on anaerobic microbes especially methane-producing bacteria (MPB) [4]. In sulfate-rich wastewater digestion, it is impossible to avoid desulfurization reactions, SRB often outcompete MPB, and SRB has much higher tolerance to sulfide compared to MPB [4]. To solve this problem, two-phase anaerobic digestion process was developed for separating SRB and MPB in different reactors, which could restrain the suppression of methanogenesis caused by sulfide toxicity [5]. The first phase is for sulfate reduction and the second for methanogenesis. The

*Corresponding author.

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hydrogen sulfide produced can be used in the process of hair removal after recovery, which not only eliminates air pollution, but also saves the production cost.

Ozone is a strong oxidant which reacts rapidly in water without the production of any solid residue. Therefore, ozonation is increasingly applied as a pre-treatment or a final polishing step for advanced water treatment [6,7].

As an alternative to the traditional activated sludge processes, biological aerated filter (BAF) possesses some advantages such as the reduced footprint, low hydraulic retention time, and high removal of organic substances [7]. Thus, BAF has been successfully combined with ozonation process for industrial wastewater biodegradation [7,8].

In terms of leather tanning wastewater, due to the high organic load, biotoxicity and the presence of sulfate and ammonium, a single unit is not efficient for treating the wastewater to meet the discharge standards currently enforced in China. Therefore, a combination of two-stage upflow anaerobic sludge blanket (UASB) reactor, ozonation and BAF process was investigated for its performance on advanced treatment of tanning wastewater. The performance, characteristics, and microbial activity changes of the system were investigated.

2. Materials and methods

2.1. Wastewater properties

Tannery wastewater used in this study was collected from a tannery factory located in Jiangsu, China. The wastewater was filtered beforehand through a 200-mesh filter to remove larger particles, leather shavings, animal hairs, etc. The filtered wastewater was stored at 4°C until use. Unless specified otherwise, tannery wastewater means the filtered wastewater in this study. The typical characteristics of wastewater are listed in Table 1.

Table 1
Characteristics of tannery wastewater after filtering

Parameter	Range
pH	6.5–6.7
COD_{cr} (mg/L)	3050–3320
BOD_5 (mg/L)	660–730
Total suspended solid (mg/L)	434–520
Total dissolved solid (mg/L)	3340–4250
Turbidity (NTU)	243–340
Color (CU)	470–520
Total phosphor (mg/L)	5.3–6.4
Total nitrogen (mg/L)	320–362
Ammonia nitrogen (mg/L)	185–240
Oil-grease (mg/L)	210–245
Sulfide (mg/L)	42–65
Sulfate (mg/L)	334–428
Total chromium (mg/L)	16.5–19.3
Cr^{6+} (mg/L)	2.1–2.4
Cl^- (mg/L)	2120–2630

2.2. Integrated system

2.2.1. Two-stage UASB

The system consists of PVC-made tanks carried on steel elements to ensure good fixation of various units. The tanks include storage tank, UASB1 (serving as sulfate-reducing phase), UASB2 (serving as methane-producing phase), and sedimentation tank (all are connected in series) (Fig. 1). The two reactors have the same working volume (15 L). A gas–liquid–solid separator device was installed at the top of reactor to retain granular sludge. A gas collecting system was connected to the reactor top through a pipe for biogas collection. A water trap was used as hydrogen sulfide (H_2S) scrubber.

The UASB reactors were seeded with sludge obtained from an anaerobic pond at the tannery factory. The excess sludge was regularly extracted from the UASB reactors to maintain the mixed liquor suspended solids (MLSS) concentration at 8000 mg/L. The UASB temperature was maintained at mesophilic condition (36±2°C) with a heating sleeve. The two-stage UASB was continuously operated for 36 weeks. Five operational hydraulic retention times (HRT) of 24, 30, 36, 42 and 48 h were applied. The process started up with HRT of 24 h for an operational period of six weeks.

2.2.2. Ozonation process

Ozonation treatment of UASB effluent was conducted in a plexiglass-made reactor (3.5 L working volume) at room temperature and atmospheric pressure. O_3 was produced from pure oxygen with an O_3 generator. O_3 was continuously bubbled into the wastewater through a microporous diffuser fixed at the reactor bottom. The wastewater was introduced by a peristaltic pump. The undecomposed O_3 emitted from the reactor was trapped by 10% KI solution in two serial wash bottles. The inlet concentration of O_3 was determined before each test. The outlet concentration was measured continuously during the experiments. The effluent was collected in an adjustment tank for further treatment.

2.2.3. BAF process

Following the above ozonation treatment, further biological treatment was performed in a BAF column with an inner diameter of 8 cm and a height of 100 cm. A microporous air diffuser was fixed on the bottom of the BAF column. Pebbles with diameter of 1–3 mm were packed in the BAF bottom to improve the distribution of air bubbles. The BAF was filled with particle ceramsites (diameter 5–6 mm, density 2.2×10^3 kg/m^3) to reach a filling height of 80 cm.

The BAF was inoculated with aerobic activated sludge from the tannery factory. In the start-up stage, the feed steam was the ozonation effluent supplemented with glucose (2 g/L). In the first five days, the sludge was not drained from the reactor in order that microbes could attach strongly to the ceramsites. After the fifth day, the ozonation effluent was introduced continuously into the BAF using a peristaltic pump. The HRT was adjusted in the range 24–12 h by adjusting the flow rate. Dissolved oxygen con-

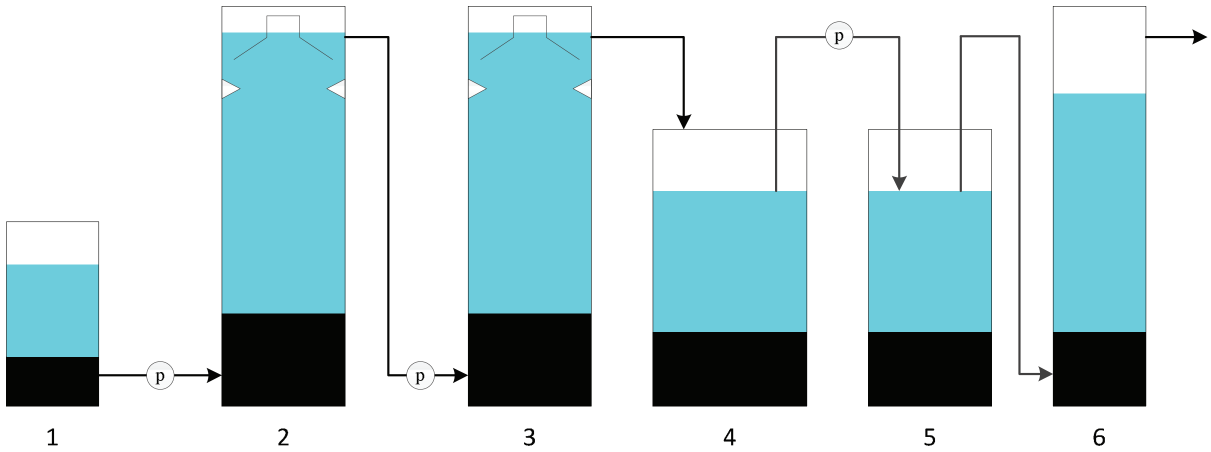

Fig. 1. Schematic diagram of the combined treatment system. (1) storage tank, (2) UASB1, (3) UASB2, (4) sedimentation tank, (5) ozonation tank, (6) BAF column.

centration was controlled to be 2–4 mg/L by adjusting the gas flow. The sludge retention time was maintained at six days by sludge withdrawal from the reactor. In this study, the overall operation temperature was kept at 23–28°C by a room air conditioner.

2.3. Analytical methods

Chemical oxygen demand (COD), ammonia nitrogen (NH_4^+-N), five-day biochemical oxygen demand (BOD_5), MLSS, color, total dissolved solids (TDS) and total suspended solids (TSS) were analyzed according to protocols set out in standard methods [9]. Turbidity was determined using a Hach 2100P portable turbidity meter (Hach, CO, USA). Sulfide was determined using a 721 UV–visible spectrophotometer (Xinmao Instruments Co., Ltd., Shanghai, China) following the method of Ruwisch [10]. The anions were determined using an ICS-2000 ion chromatography system (Dionex Corp., Sunnyvale, CA, USA). Total chromium, Cr^{6+} and Cr^{3+} were determined by flame atomic absorption spectrometry (F-AAS, Shimadzu AA-7000, Kyoto, Japan). Volatile fatty acids (VFAs) were determined by titration method [11]. Biogas composition was determined using a Agilent 7890 gas chromatograph (Agilent Technologies, Wilmington, DE, USA). In this study, all measurements were performed in triplicates and arithmetical averages of the results were taken.

2.4. Biochemical analysis

The specific methane-producing activity (MPA) and sulfate-reducing activity (SRA) of the retained sludge in the UASB were determined by a serum vial test [12]. The sludge was disintegrated under anaerobic conditions and was used for the activity tests. Sodium acetate solution (2000 mg COD/L) and H_2/CO_2 (80:20, v/v, 1.4 atm) were used as MPA and SRA test substrates, respectively. In the SRA test, sodium sulfate solution (200 mg-S/L in the vial) and chloroform solution (5 mg/L in the vial) were added to the test vials to quench methane production. All vials were incubated at 120 rpm and 35°C.

3. Results and discussion

3.1. Start-up of the UASB

During UASB treatment, a good balance needs to be established among various microbial species. Thus, the start-up of UASB is the most sensitive and challenging stage in the process [13]. In general, anaerobic bioreactors should be started by acclimatizing the seeding biomass with readily biodegradable substrates, and subsequently, the substrate would be replaced with the original wastewater in a stepwise manner. In this work, considering that the inoculated sludge was collected from a tannery wastewater treatment plant, the biomass should have been adapted to wastewater. Thus, the UASB was directly fed with the tannery wastewater at a HRT of 24 h and an organic loading rate (OLR) of 3.10±0.15 g COD/(L·d) during the start-up period.

Fig. 2 shows the UASB performance during the six weeks of start-up stage. As can be seen, a lag phase of about one week was observed with characteristics of very low removal of COD and SO_4^{2-} and low CH_4 yield. This phenomenon indicates that the sludge environment was not favorable for the growth of the seeding microbes. Nevertheless, significantly improved performance was obtained during the subsequent three weeks, indicating that the microbiota were gradually adapted to the wastewater environment. As a result of fast microbial growth and substrate utilization, stable UASB performance was achieved from week 5 to week 6. CH_4 yield is an indirect metabolic indicator for the evaluation of the start-up of anaerobic reactors [14]. Therefore, the UASB start-up was considered complete after week 6 since the CH_4 yield had been stabilized in the system.

3.2. Process performance of the two-stage UASB system

After the six weeks of start-up, the two-stage UASB system was continuously operated for another 30 weeks under various HRTs. Firstly, the HRT of the system was stabilized at 24 h for 6 weeks.

Fig. 3A shows the COD removal regularity with HRT in two-stage UASB reactor treating the tannery wastewater.

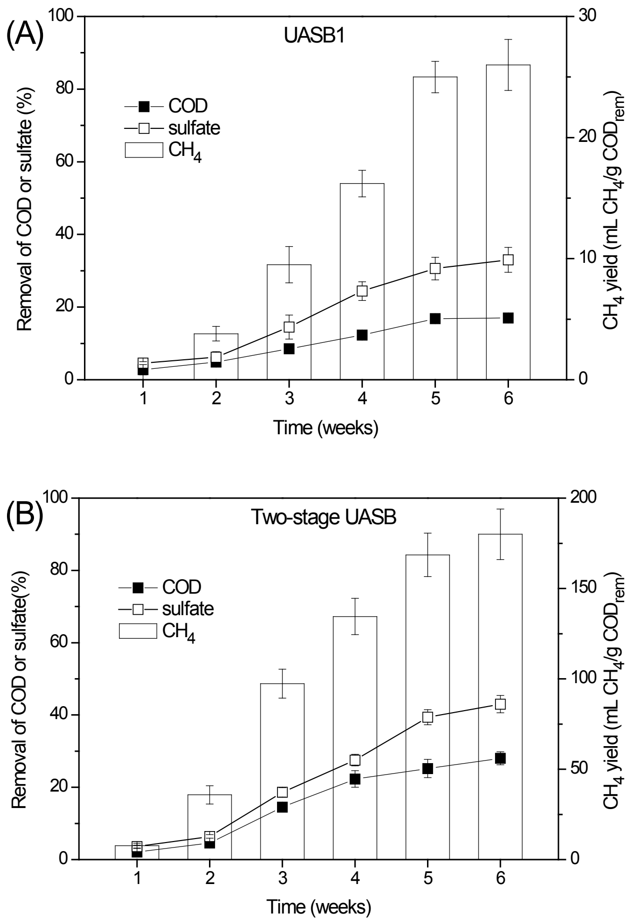

Fig. 2. Performance of (A) UASB1 and (B) two-stage UASB during the six weeks of the start-up period. Experimental conditions: HRT = 24 h.

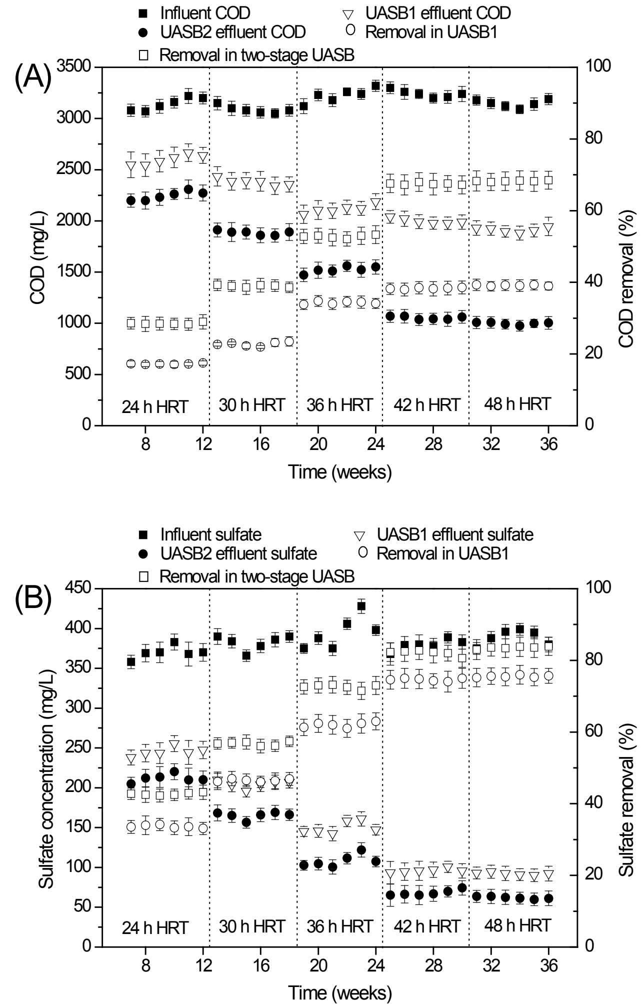

Fig. 3. Process performance of two-stage UASB system under different HRTs: (A) COD removal, (B) sulfate removal, (C) TSS removal, and (D) CH_4 yield.

During 7–12 weeks, the COD removal in sulfate-reducing stage (UASB1) and two-stage UASB was 17.3% and 28.6%, respectively, corresponding to the HRT of 24 h. With the HRT being raised, the performance of both was improved steadily and kept at about 38.2% and 67.4%, respectively, at 42-h HRT. When the HRT was further prolonged to 48 h at week 31–36, little increase of COD removal was achieved in both sulfate-reducing stage and two-stage reactor. Therefore, the optimum HRT operated in two-stage UASB was 42 h. This is in agreement with the results of other studies concerning tannery wastewater treatment using UASB reactors [2,15]. Residual metals and organics in tannery wastewater were toxic to microorganisms in the reactors. As a result, long HRT was required to offset decreasing metabolic rate resulted from wastewater toxicity.

The sulfate removal regularity with HRT was similar to COD removal presented above (Fig. 3B). When the HRT was kept at 24 h, sulfate removal efficiencies in UASB1 and two-stage UASB were 33.5% and 42.8%, respectively. Both of them increased with HRT and were maximum 74.6% and 82.3%, respectively, at 42-h HRT. During the whole 30-week operation process, the sulfate removal efficiencies in methane-producing stage (UASB2) were much lower than that in sulfate-reducing stage (UASB1) (Fig. 3B). The ratio of sulfate reduction in UASB2 and UASB1 was approximately 1/9. Sulfate removal in the UASB could be ascribed to the conversion of sulfate (SO_4^{2-}) or sulfite (SO_3^{2-}) to sulfide (S^{2-}) and the subsequent precipitation with metals into the anaerobic sludge as well as H_2S emission.

Fig. 3C shows that the suspended solid (TSS) removal efficiencies of 51.3% and 64.2% were observed in UASB1 and two-stage UASB, respectively, at 24-h HRT, which increased to 73.3% and 82.5% at 48-h HRT. This improved efficiency occurred due to decreasing sludge concentration in UASB reactors coupled with a decline in upflow velocity with the longer detention time. This caused a fall of the sludge blanket along the reactor height. Additionally, it was noted that the most of TSS removal occurred in UASB1 where the average values of TSS removal through all operational phases were 65.8% and 22.7% for UASB1 and UASB2, respectively.

Fig. 3D demonstrates the CH_4 yield regularity with HRT in the two-stage UASB system. As shown, CH_4 yield in UASB2 was much higher than that in UASB1, and both of them increased with decreasing the HRT (namely increased OLR) after week 12. The optimum for CH_4 production could be seen at 30 h retention time. However, this HRT (30 h) was not the optimum for other pollutant removal such as COD and sulfate as shown above. Therefore, the optimum operating HRT has been chosen to be 42 h because pollutant removal is more needed and more important than biogas production.

Based on the above-mentioned experimental results, the maximum acceptable OLR of two-stage UASB was approximately 1.8 g COD/(L·d) at 42-h HRT. In terms of the OLR and COD removal efficiency, the two-stage UASB system achieved similar or better performance than that of other UASB treatment system for treating tannery wastewater [2,15]. The MPA of granular sludge could be inhibited by a high concentration of H_2S. In UASB1, more than 70% of sulfate was consumed, and consequently the generation and inhibition of UASB1 in UASB2 was reduced. Therefore, it is suggested that good performance was achieved in the two-stage UASB system by separating the stages of organic removal by SRB and MPB.

3.3. Microbial activity of UASB-retained sludge

Fig. 4A presents the variation with time in the MPA of UASB-retained sludge. The acetate-fed methanogenic activity (MPA-Ac) and H_2/CO_2-fed methanogenic activity (MPA-H_2) of seed sludge was 0.24 and 0.05 g COD/(g VSS·d), respectively. In general, the sludge MPA of both UASB stages increased significantly after reactor start-up. In UASB1, MPA-Ac increased until week 12, and then decreased gradually (Fig. 4A). In UASB2, however, MPA-Ac increased up to week 18 and then remained stable with some fluctuations. After 12 weeks, MPA-Ac of UASB2 became greater than that of UASB1. For the MRA of UASB retained sludge, MPA-H_2 was much lower than MPA-Ac throughout the experimental period in the retained sludge of both UASB stages.

For the SRA of UASB-retained sludge, H_2/CO_2-fed SRA (SRA-H_2) was dominant for both reactors (Fig. 4B). Acetate-fed SRA (SRA-Ac) was not detected in seed sludge, and it was low in the sludge during the operation period. A slight decrease in SRA-Ac was observed in UASB1 after 18 weeks of operation. For an acetate substrate, MPA-Ac was higher than SRA-Ac for both UASB stages. Conversely, SRA-H_2 was higher than MPA-H_2 after 12 weeks. Overall, UASB1 had high sludge SRA but low sludge MPA compared to UASB2, which was consistent with the results of sulfate reduction and methane production as shown in Fig. 3.

SRB could suppress MA through competition for substrates such as hydrogen and organics. Moreover, sulfate could be transformed into sulfide by SRB, and sulfide could poison MPB and decrease methane production [16]. Therefore, in the sulfate-reducing stage (namely UASB1), SRB could outcompete MPA and predominate in both quantity and activity. The growth of SRB was dependent on carbon source and sulfate concentration, whereas the growth of MPB solely depended on carbon source concentration. In the methane-producing stage (namely UASB2), low sulfate concentration may limit SRB growth. This enabled MPB to outcompete SRB, and rendered MPA predominate in both quantity and activity. Accordingly, sulfidogenesis mainly occurred in UASB1, whereas methanogenesis mainly occurred in UASB2. These results indicate that the two-stage UASB system could separate sulfidogenesis and methanogenesis in two sequential reactors.

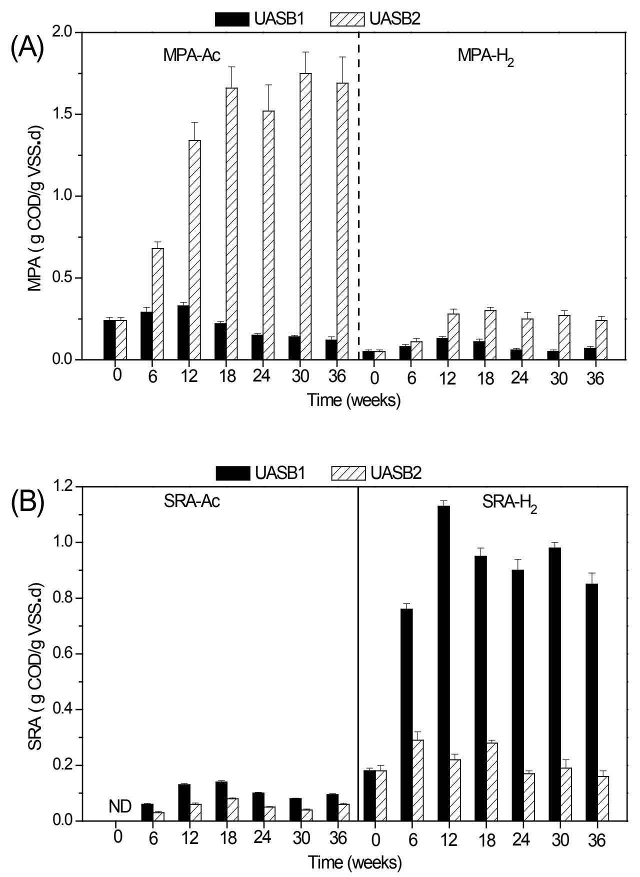

Fig. 4. (A) Methane-producing activity (MPA) and (B) sulfate-reducing activity (SRA) of UASB-retained sludge (ND: not detected).

3.4. Ozonation treatment

On the basis of the above-mentioned results, 42 h was chosen as the optimum HRT for the two-stage UASB system. Under this condition, the effluent COD (averagely 1050 mg/L) could not meet the Chinese discharge standard for leather tanning industry (GB30486-2013)) (COD ≤ 100 mg/L). Thus, UASB2 effluent at 42 h HRT was collected for ozonation treatment. Ozone dosage and reaction time are the two key parameters influencing ozonation performance [17]. Thus, batch experiments were conducted to optimize these two parameters. The feed flow rate of O_3 was regulated to adapt the duration of reaction time during each test with the optimum O_3 dosage.

Fig. 5A shows the results of ozonation experiments using $[O_3]/[COD_0]$ ranging from 0.05 to 0.40 under the conditions of 50 min and natural pH (6.5). As shown, COD removal increased with the increase of O_3 dosage up to 0.4. Moreover, the BOD_5/COD ratio was maximum at 0.2 O_3 dosage, and a further increase of O_3 dosage resulted in the continuous decrease of BOD_5/COD ratio. This is because the excess amount of O_3 could mineralize the biodegradable organics formed during ozonation. Therefore, this O_3

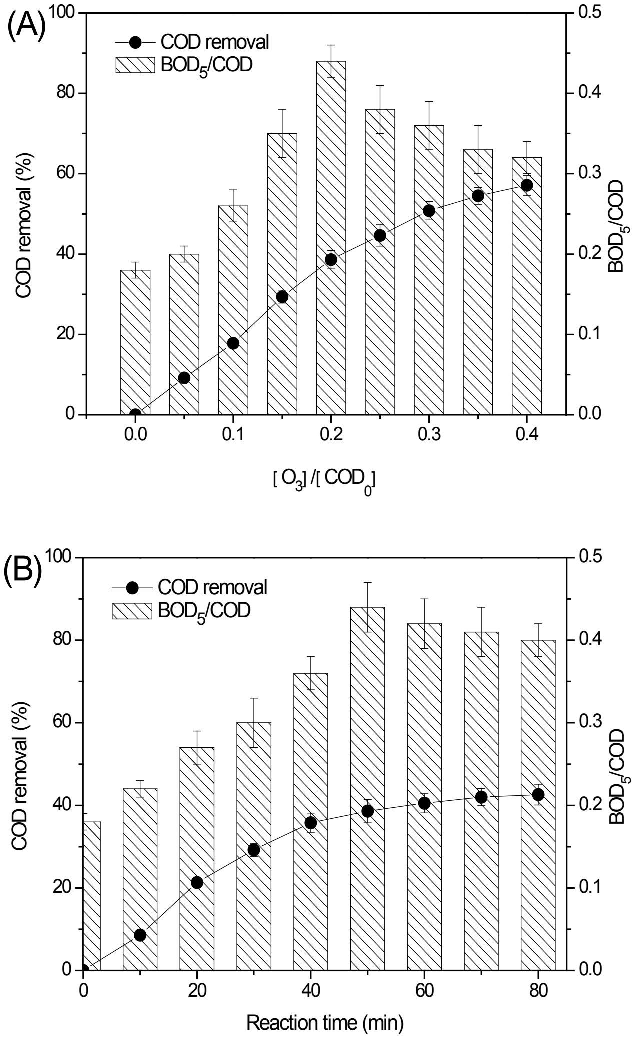

Fig. 5. (A) Changes in COD removal and BOD_5/COD ratio at 50 min of reaction time and different O_3 dosages. (B) Time course of COD removal and BOD_5/COD ratio at an O_3 dosage of 0.2.

dosage could be deemed optimum as it chemically produced good biodegradability enhancement while limiting COD mineralization.

Fig. 5B shows the results of ozonation experiments using $[O_3]/[COD_0]$ of 0.2 under the conditions of 0–80 min and natural pH (6.5). As shown, COD removal increased quickly within the initial 30 min. Thereafter, COD removal slowed down gradually with time. For instance, COD removal efficiency increased from 8.6% in 10 min to 29.2% in 30 min. Although COD removal efficiency reached 42.6% within 80 min, the option was not optimum and not economic for ozonation pretreatment, since a downstream biotreatment process was established for the final polishing. Moreover, when reaction time was increased from 30 to 50 min, BOD_5/COD ratio increased slowly, even presenting a slight decrease during 50–80 min (Fig. 5B). Thereupon, 50 min reaction time was reasonable in this work, since the primary aim of ozonation was to improve wastewater biodegradability rather than mineralization of organics.

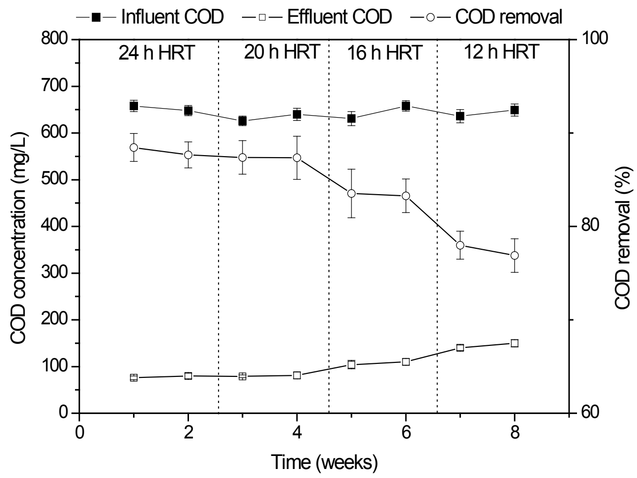

Fig. 6. Process performance of BAF system under different HRTs.

3.5. Biological treatment

After the start-up period, the BAF reactor was operated for eight weeks at HRT of 24, 20, 16 and 12 h, respectively. Each HRT lasted for two weeks. Fig. 6 illustrates the influent and effluent COD concentrations of BAF system and the corresponding removal percentages under various HRTs. As shown, the influent COD was relatively stable, whereas the effluent COD fluctuated with HRT. The influent COD concentration was in the range of 626–658 mg/L. When HRT was decreased from 24 to 20 h, the effluent COD changed little (Fig. 6). When HRT was further reduced to 16 h, the effluent COD only decreased slightly. This demonstrates that the BAF system had a high anti-shock ability because BAF has high biomass density [2,7,8]. Nevertheless, an apparent increase was observed for the effluent COD as HRT was transitioned from 16 to 12 h. At HRT of 24, 20, 16 and 12 h, the average effluent COD concentrations were 78, 80, 107 and 145 mg/L, respectively, corresponding to removal efficiency of 87.5%, 87.3%, 83.4% and 76.6%, respectively. At HRT of 24 and 20 h, the effluent COD could satisfy the national discharge standard of China set for the leather tanning industry (GB30486-2013).

3.6. Performance analysis of the combined treatment system

As mentioned above, the optimum treatment conditions for the combined system were: 42 h HRT for two-stage UASB, $[O_3]/[COD_0]$ of 0.2 for 50 min, and 20 h HRT for BAF. As listed in Table 2, for the combined system, the value of various water-quality parameters in the effluent could meet the Chinese wastewater discharge standard for the leather tanning industry (GB30486-2013). The removal of chromium occurred mainly through precipitating with sulfide. This demonstrates that the combined system is a feasible approach for treatment of heavily polluted tannery wastewater. As can be seen from Table 2, in the combined system, the two-stage UASB played an important role in COD and sulfate removal. The biodegradability of UASB effluent was significantly enhanced through ozonation,

Table 2
Average effluent quality, total removal of pollutants, and the contribution of each unit to total removal in the combined system

Parameter	BAF effluent	Total removal (%)	Contribution (%)			Discharge standard
			UASB	Ozonation	BAF	
pH	6.4	–	–	–	–	6–9
COD	80	97.5	68.2	11.7	20.1	≤100 mg/L
TSS	26	94.6	90.1	1.2	8.7	≤50 mg/L
Total nitrogen	22	93.5	36.2	0.3	63.5	≤30 mg/L
Ammonia nitrogen	8.6	95.9	6.6	0.6	92.8	≤15 mg/L
Oil-grease	5.3	97.6	75.2	8.5	16.3	≤10 mg/L
Color	12	97.4	42.3	26.8	30.9	≤50 CU
Total Cr	0.8	95.6	84.2	NAC	15.8	≤1.5 mg/L
Cr^{6+}	0.07	96.8	86.5	NAC	13.5	≤0.1 mg/L

NAC: no appreciable change.

which benefited the subsequent biological treatment. The BAF process shouldered a crucial role for polishing the final effluent to make the water quality finally satisfy the statutory discharge standard.

4. Conclusions

This work investigated the effectiveness of lab-scale two-stage UASB combined with ozonation and BAF processes in tannery wastewater treatment. The results show that the combined system could efficiently treat heavily polluted tannery wastewater. Under the optimum conditions, the effluent quality could satisfy the national discharge standard of leather tanning industry. Two-stage UASB shouldered a major role in pollutant removal; ozonation enhanced the biodegradability of UASB effluent, while the polishing of ozonation effluent was accomplished in the BAF process. Thus, biological–chemical treatment composed of UASB, ozonation and BAF is a viable technique for tannery wastewater treatment.

Acknowledgments

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