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Biological aeration filter post-treating effluent from Fenton oxidation process of wastewater containing rhodamine B

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ABSTRACT

In order to investigate the treatment of refractory organic pollutants in textile wastewater, Rhodamine B (RhB) was selected as a model pollutant to prepare the simulated wastewater. Aim to treat the effluent from Fenton oxidation process for wastewater containing RhB, biological aerated filter (BAF) was used as post-treatment process. The start-up process of the BAF reactor and the optimum operation conditions for the wastewater treatment process were investigated in this study. It was demonstrated by a series of bench-scale tests that the reactor could be successfully started up by exogenously inoculated activated sludge and gradual acclimation method. Operation condition experiments indicated that the BAF could achieve relatively high performance when gas–water ratio was 5–6, hydraulic retention time was controlled at 6–8 h and influent organic loading was within the range from 0.77 to 2.04 kgCOD/(m^3 d).

Keywords: Rhodamine B; Biological aerated filter; Textile wastewater; Start-up

1. Introduction

Textile wastewater includes many kinds of chemicals such as enzymes, dyes, auxiliaries, acids, and sodium salts [1]. Anaerobic, aerobic, and their combination biological treatment processes are the most widely used in conventional textile wastewater treatment plants (WWTPs) [2]. However, with the increasing usage of new synthetic dyes and auxiliaries, many wastewater discharged from the textile industry cannot meet the discharging standard regulated by the pollution control boards in China. This is mainly due to the low BOD to COD ratio, which is identified to be caused by toxic/non-biodegradable residual dyes and auxiliaries in the textile wastewater [3]. At this circumstance, conventional biological treatment processes are not efficient enough to remove these recalcitrant organics [4]. Thus, how to remove these recalcitrant organics in textile wastewater has drawn considerable attention from researchers [5].

Rhodamine B (RhB), is one of the most important red synthetic dyes of xanthene class, it is widely used as a colorant in textiles, food stuffs, and water tracer fluorescents [6]. It is highly water soluble, carcinogenicity, and neurotoxicity to human and animals [7]. Besides, it has been proved to be recalcitrant in

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conventional biological treatment processes, even high resistance to photo and oxidation degradation [8]. It has been detected to be remained in effluent from many WWTPs for textile industry. To satisfy the safety requirements of aquatic environment, the residual RhB in the effluent from the conventional textile WWTPs should be removed. Therefore, RhB has been used as common objective organic pollutant to explore the efficient removal methods in many studies [9,10]. Also, RhB is selected as a representative model pollutant in this work.

Advanced oxidation processes have been considered as the most efficient method for partial or complete removal of recalcitrant organic pollutants in wastewater or their transformation into less toxic and more biodegradable products. Fenton oxidation, as a representational advanced oxidation technology, was studied extensively by many researchers for the removal of recalcitrant/non-biodegradable organics from wastewater [11]. The reaction mechanisms of Fenton oxidation [1] for organics in wastewater are illustratively depicted by Eqs. (1)–(3).

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + {}^{\cdot}OH \quad (1)$$

$${}^{\cdot}OH + RH \rightarrow R^{\cdot} + H_2O \quad (2)$$

$$R^{\cdot} + Fe^{3+} \rightarrow R^+ + Fe^{2+} \quad (3)$$

By Fenton reaction, recalcitrant/non-biodegradable organics can be partially or completely removed or transformed into less toxic and more biodegradable products. These intermediates produced from the partial decomposition of non-biodegradable organics still need to be removed by other treatment technologies. Therefore, the Fenton oxidation is usually used as a pre-treatment process to improve the biodegradable ability of organics, and subsequent biological treatment process is essential to remove the intermediates in the effluent from the Fenton oxidation process.

Biological aerated filter (BAF) is one of the rapidly developed technologies in the field of wastewater treatment in recent years [12]. It is a comprehensive process of biological oxidation, adsorption, and filtration. BAF has a lot of advantages, such as little footprint, high concentration of active biomass, high organic loading, no need for separated secondary clarification, and no sludge expansion problem [13]. Besides, it can run intermittently and recover its performance in a short time even after interruption for a long time.

Owing to above advantages, BAF is selected as a post-treatment process for the simulated wastewater from traditional textile WWTP in this study. RhB contained wastewater was used as source wastewater to simulate the effluents containing biological refractory pollutants. Taking into consideration that RhB is difficult to be directly degraded by micro-organisms, Fenton oxidation pretreatment process is carried out for the source wastewater. The BOD_5/COD value can reach about 0.75 after the Fenton oxidation process, indicating that RhB can be oxidized into small molecules, which are easier for biodegradation. This issue will be discussed elsewhere.

The current work will focus on the effectiveness of a lab-scale BAF for treating the effluent from Fenton oxidation process of RhB. Various operation conditions including gas–water ratio, hydraulic retention time (HRT), and influent organic loading were investigated and the optimized operation conditions were determined. This study may be helpful for scaling up and application of the BAF in the textile wastewater treatment.

2. Materials and method

2.1. Wastewater

Glucose nutrient solution was used in the stage of biofilm cultivation, which includes glucose 500 mg/L, NH_4HCO_3 125 mg/L, KH_2PO_4 25 mg/L, and micronutrient solution 2 mL/L. The pH value of the solution is adjusted from 6.5 to 7.5 by H_2SO_4 or NaOH.

The micronutrient solution includes EDTA 50.0 mg/L, $ZnSO_4$ 2.2 mg/L, $CaCl_2$ 5.5 mg/L, $MnCl_2 \cdot 4H_2O$ 5.06 mg/L, $FeSO_4 \cdot 7H_2O$ 5.0 mg/L, $(NH_4)_6Mo_7O_2 \cdot 4H_2O$ 1.1 mg/L, $CuSO_4 \cdot 5H_2O$ 1.57 mg/L, and $CoCl_2 \cdot 6H_2O$ 1.61 mg/L. All these chemicals are AR grade and used without further purification.

2.2. Pre-oxidation process

RhB solutions with certain concentrations were pre-oxidized by heterogeneous Fenton reaction. The oxidation reactor was a typical continuous stirred tank reactor with effective volume 1.2 L. Influent wastewater was fed to give a reaction time of 30 min for the oxidation process. The pH value of the wastewater was adjusted to 5.0. The oxidation reagent was peroxide and its dosage is 50 mmol/L. Fe/Fe_3O_4/carbon granules were experimentally made and used as heterogeneous Fenton catalyst. Typical COD removal efficiency for Fenton oxidation process was about 65%. NH_4HCO_3 and K_2HPO_4 were added into the wastewater after the pre-oxidation to serve as the N and P nutrients for micro-organisms and the ratio was controlled at COD:N:P = 00:5:1. Finally, the pH value of the wastewater was adjusted to neutral and the wastewater was used as influent wastewater for the BAF in the experiment process.

2.3. Experimental reactor

The BAF reactor used in this study was consisted of gas distribution system, water distribution system, packing materials layer, backwash system, and temperature control system. The schematic configuration of the whole system is shown in Fig. 1.

The main column of the BAF reactor was made of transparent Perspex. The inner diameter of the main reactor was 150 mm. The height of water and gas distribution zone was 200 mm. The total height of the packing materials zone was 2,000 mm, in which the gravel supporting layer at the bottom is 200 mm and the upper ceramic packing layer is 1,800 mm. The characteristics of the ceramic packing materials are shown in Table 1.

At the top of the packing layer, there is a buffered water layer with 300 mm in height. The highest part of the whole reactor is an enlarged degassing and outflow zone with 350 mm in height. The empty bed volume of packing materials zone is 35.3 L, volume of water and gas distribution zone is 3.5 L, and the total volume of the water layer at the top of the packing materials is 43.5 L. The orifice diameter of gas distribution pipe mounted at the bottom of the reactor and below the supporting layer is 1 mm. There are six sampling holes at the side wall of the packing layer. The location heights of these sampling holes when calculated from the bottom of the reactor are 0.4, 0.7, 1.0, 1.4, 1.7, and 2.0 m, respectively.

2.4. Operation methods of the reactor

The BAF operating mode is upward flowing in the same direction for gas and water phases. The influent wastewater stored in the high wastewater tank flows into the bottom of the reactor. Compressed air is also released into the bottom space of the reactor from air distribution pipe and mixes uniformly with the influent wastewater. The mixture of gas and wastewater go upward through the packing materials layer, the buffer water layer, and the degassing zone successively. Finally, the effluent wastewater overflows through the outflow weir and collects in effluent tank. After running for a certain period of time, the filter resistance increase obviously and the packing materials should be backwashed to recover its filter ability. The backwash process includes both large flow rate air backwash and high pressure tap water backwash.

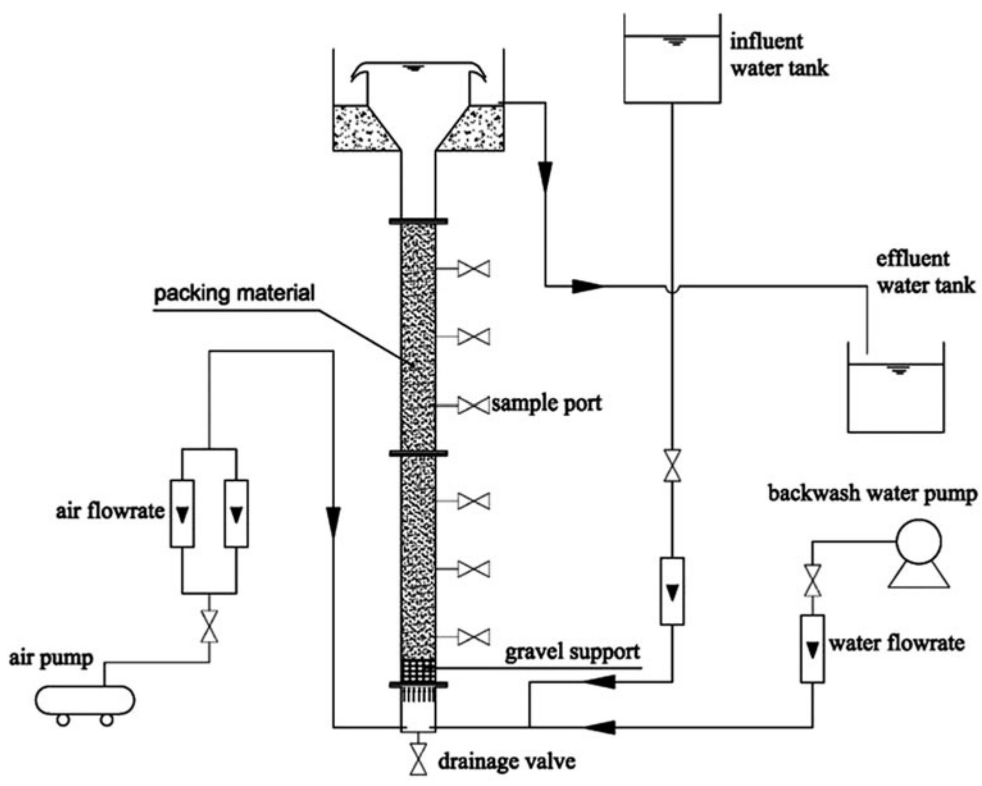

Fig. 1. Schematic configuration for the biological aerated filter reactor.

Table 1
Characteristics for homogeneous ceramic packing materials

Physical characteristics	Values
Appearance	Approximate sphere, red brown, porous
Size	4–6 mm
Bulk density	0.89 g/cm
Specific surface area	3.85 cm^2/g
Porosity	55–60%
Compressive strength	>5.0 MPa
Actual density	1.56 g/cm

The packing materials are controlled to be slightly expanded in the backwash process. The backwashed water is discharged separately.

During all the experiment processes, the gas and water flows are measured by the pre-calibrated gas and water flow meters. Air was supplied by an air pump (Haili, ACO-003D, China). The air flow rate is determined as the demand of air/water ratio and controlled by gas valve. Temperature in the reactor is controlled within 28 ± 1˚C by an electrical heater, which is wrapped outside the reactor and controlled by a thermal sensor imbedded into the packing materials layer. In order to ensure stable temperature, influent wastewater is also preheated by water bath.

2.5. Analysis methods

The analysis methods of wastewater, including COD concentration based on potassium dichromate and biofilm concentration, were based on the standard methods [14]. The morphology of biofilm was observed by the scanning electron microscope (Hitachi, S-3000 N). The thickness of the biofilm was measured by microscope equipped with micro scale ruler in eyepiece [15]. Fifty packing particles with biofilm were randomly selected from six sampling holes and their diameters were measured as d_i. The average diameter of the particles with biofilm (d_p) was calculated by Eq. (4). By the same method, average diameter of clean particles was measured and calculated as d_m. The average thickness of biofilm (δ_w) was acquired by Eq. (5). The biomass of biofilm was determined by the weighting method [16].

$$d_p = \frac{\sum d_i^3}{\sum d_i^2} \tag{4}$$

$$\delta_w = \frac{d_p - d_m}{2} \tag{5}$$

3. Results and discussion

3.1. Start-up of BAF, acclimation, and stabilization

3.1.1. BAF start-up process

The exogenous activated sludge inoculation was introduced to start up the BAF reactor. Inoculated sludge was acquired from oxidation ditch of local wastewater treatment plant (Ganzhou, Jiangxi Province, China) and MLSS concentration of inoculated sludge was 4,250 mg/L. The volume of added sludge for inoculating was just able to cover the upper surface of packing materials layer. Then glucose nutrient solution was added in the reactor until the fluid level was the same as the outflow weir. The aeration devices were started and kept running for continuous 24 h without influent wastewater. After that, the mixture liquid was settled for 1 h and the supernatant above the packing materials layer was removed. The glucose nutrient solution was refilled into the reactor and aeration was restarted. These procedures were repeated for 4 d and biofilms could be obviously observed on the surface of the packing materials. At last, the nutrient solution was all drained out.

The following are the continuous water feeding stages. In this stage, little influent flow was fed to the reactor and HRT was controlled at about 24 h. The glucose nutrient solution was still used at this stage. The detailed operation conditions used in this continuous water feeding stage are summarized in Table 2. The reactor ran for another 7 d and relatively uniform biofilm could be observed on the surface of the packing materials. At this stage, the effluent COD maintained at 75–95 mg/L and the corresponding COD removal ratio was more than 80%, which indicated that BAF reactor had been successfully started up.

3.1.2. Acclimation and stabilization

In order to let the BAF acclimate the RhB wastewater and accumulate stable biofilm, the following processes were carried out on the BAF reactor. Firstly, the

Table 2
Operation conditions in the continuous water feeding stage

Item	Unit	Value
Inflow rate	L/d	20.2
COD of influent	mg/L	534
Aeration rate	L/h	16
HRT	h	24
Organic loading	$kgCOD/m^3 d$	0.31

influent was switched to mixture wastewater of glucose nutrient solution and RhB wastewater with the ratio of 1:1 (*V*/*V*). HRT was still controlled at 24 h and the reactor was kept running for 7 d. Secondly, the influent was switched to 100% RhB wastewater and the HRT was set as 16 h. At this operation condition, the reactor ran 7 d as one cycle. Finally, the HRT was shortened to 12, 8, and 6 h, respectively. At each HRT, the operation cycle of the reactor was 5 d. The total acclimation and stabilization process was ended and it lasted for 29 d. Influent and effluent COD concentration in the whole process are showed in Fig. 2.

It could be seen from Fig. 2 that during initial period which was designated as stage "a," biofilm acclimated well in the mixed influent partly containing the target wastewater, and no obvious biological inhibition effect could be detected. In this period, the COD removal ratios rapidly increased from 65.6 to 86.7%, which indicated that the micro-organisms had acclimated the mixed influent wastewater quickly. In the stage "b," after the influent was switched to the whole synthesized wastewater and the HRT was shortened to 16 h, the COD removal ratio declined to 69.8% quickly. However, the COD removal ratio recovered gradually to 79.8% after that switch. Similarly, in the stage "c" the HRT was changed to 12 h, the COD removal ratio rapidly decreased to 76.7%, and then gradually increased to 84.7%. In the stage "d," the HRT was reduced to 8 h, the COD removal rate decreased to 79.8% firstly, and then it gradually increased to 85.4%. In the stage "e," the HRT was reduced to 6 h, the initial COD removal rate decreased to 80.2% rapidly, and then gradually increased to 84.2%. From the COD removal efficiency analysis for these five stages, it could be concluded that after first two periods of "a" and "b," biofilm had fully acclimated to the effluent from Fenton oxidation process of Rhodamin B wastewater. The proposed reactor achieved relatively high COD removal ratio and the effluent COD was around 70 mg/L. During stages of "c," "d," and "e," despite the decreased retention time, the HRT affected little on COD removal ratio and it could recover to a relatively high level quickly. All these findings proved that the biofilm of BAF reactor had acclimated to the target wastewater and became mature and stable. The concentration of the mature biofilm was measured to be 16.32 g/L and the average thinness of biofilm was 93 μm. Including 10 d of the initial start-up time, the BAF reactor was successfully started up in 40 d and reached satisfactory and stable treatment efficiency.

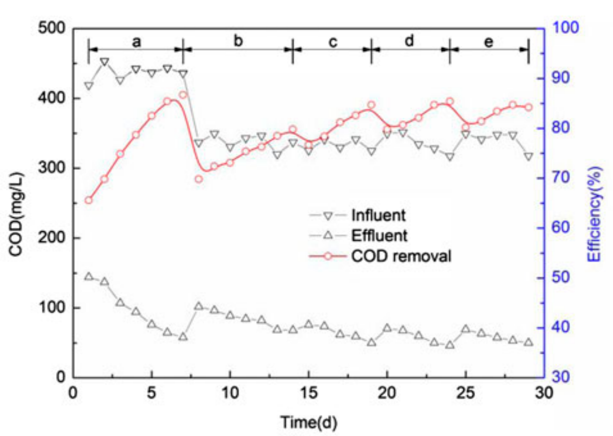

Fig. 2. Performance of BAF reactor at acclimation and stabilization stage. (a-glucose solution/wastewater = 1:1, HRT 24 h, 7 d; b-only wastewater, HRT 16 h, 7 d; c-only wastewater, HRT 12 h, 5 d; d-only wastewater, HRT 8 h, 5 d; e-only wastewater, HRT 6 h, 5 d).

3.1.3. Biofilm morphology observation by SEM

In order to investigate the biofilm growth in BAF, some packing materials attached with biofilm close to the sample ports were picked out randomly during biofilm formation, acclimation, and stabilization stages, respectively. The surface morphology of all these packing materials was observed by SEM and shown in Fig. 3.

Fig. 3(a) showed the surface morphology of clean packing materials. The surface was uneven with irregular holes and cracks. The thickness of biofilm in Fig. 3(b) was relatively thin and the coverage of biofilm was relatively sparse at that time. There was still some blank area on the surface and the thickness of biofilm was relatively uniform except the blank area. In Fig. 3(c), the biofilm was relatively thicker at this time. Biofilm was shown in an accumulational growth state and no blank area could be observed. Besides, the surface of biofilm was quite rough. These phenomena could be well explained by the plate theory established by predecessors [17].

3.2. Determination of best operating conditions for BAF system

3.2.1. Gas–water ratio

Gas–water ratio is the key parameter in BAF which directly determines BAF dissolved oxygen concentration. Gas directly provides oxygen for the micro-organism growth. Also, momentum of gas increases water turbulence, so as to enhance the mass transfer effect from gas to water phase.

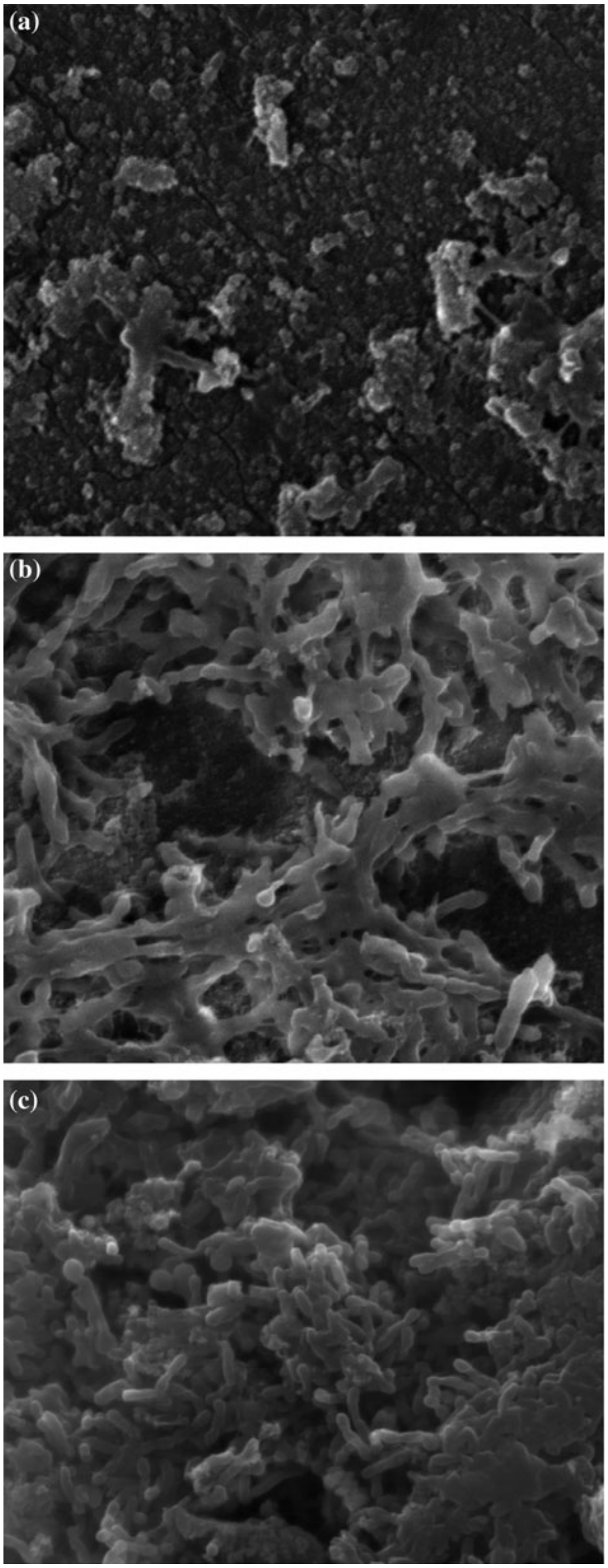

Fig. 3. Morphology of packing materials at various times. (a) Clean packing materials, (b) packing materials after running for 8 d, and (c) packing materials after running for 35 d.

The experiments of gas–water ratio determination were started from the 15th day after the BAF reactor being successfully started up and stable operation. The operating conditions of BAF reactor listed as follows: influent flow was 2.5 L/h and corresponding HRT was about 8 h, the reactor temperature was 28 ± 1℃, and initial biofilm mass concentration was 16.94 g/L. Air flow is changeable and the air–water ratio is controlled at 3, 4, 5, 6, 7, and 8, respectively. For each air–water ratio operation condition, the reactor ran for five continuous days as one cycle. Influent and effluent COD of wastewater were measured every day and the COD of latter three days in every 5 d cycle are shown in Fig. 4.

As it can be seen from Fig. 4, when the gas–water ratio increases from 3 to 6, COD removal ratio increased from 75.2 to 84.5%. However, when the gas–water ratio increased from 6 to 8, COD removal ratio declined slightly. When gas–water ratio was 7 and 8, the average COD removal rates are 84.2 and 83.3%, respectively. The average COD removal rate reached maximum when gas–water ratio was six. The influent COD fluctuates around 430 mg/L, the effluent COD was above 85 mg/L on average when the gas–water ratios were 3 and 4. When the gas–water ratios were 5, 6, 7, or 8, the effluent COD was all very close at about 68 mg/L.

The water–gas ratio denotes the amount of aeration per unit influent volume. High ratio will lead to high DO level in the influent, which is favorable for the aerobic micr-organisms and can increase metabolism activity. However, when gas–water ratio is beyond the optimum value, gas turbulence due to excessive aeration will be very significant. The biomass in the reactor will decrease because of the shear effect of gas. Actually, it can be observed obviously when the gas–water ratio is 7 and 8, the effluent contains shedding

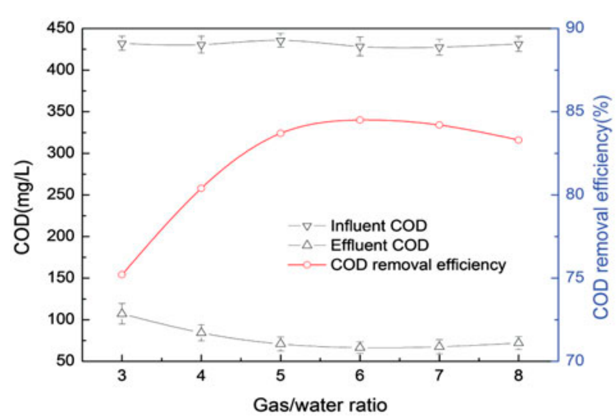

Fig. 4. Influence of gas–water ratio on performance of BAF.

biofilm fragments, which reduces the quality of the effluent. So it is more reasonable to control the air–water ratio between 5 and 6.

3.2.2. Influence of HRT on BAF performance

HRT represents the contact time of water and biofilm. It is a critical factor to determine the performance of the BAF reactor. Generally, the extension of HRT is preferable for increasing performance of the reactor. However, it is not economical to prolong the HRT from the viewpoint of acquiring the reasonable volume utilization efficiency. Therefore, operation condition experiments for HRT selection were carried out after the air–water ratio experiments were completed.

The controlled experiment conditions are listed as follows. Initial biofilm mass concentration is about 16.31 g/L, reactor temperature is 28 ± 1℃ and the gas–water ratio is five. Especially, the HRT is set at 12, 10, 8, 6, 4, and 2 h, respectively. The reactor continuous ran 5 d for each HRT. The influent and effluent COD of wastewater were tested every day and the experiment results are reported in Fig. 5.

As shown in Fig. 5, COD removal efficiency is gradually decreasing with the shortening of HRT. When HRT is 2 h, average COD removal is about 64.5% and the effluent COD is over 153 mg/L. Besides, each time after HRT was changed, COD removal ratio has a significant decline in the initial stage and recovered to a stable level quickly. This indicates that system has a strong ability on resisting the shock of loading. When HRT is 2 and 4 h, the COD removal ratio is relatively low. When HRT is more than 6 h, COD removal efficiency increases a little with the extension of HRT.

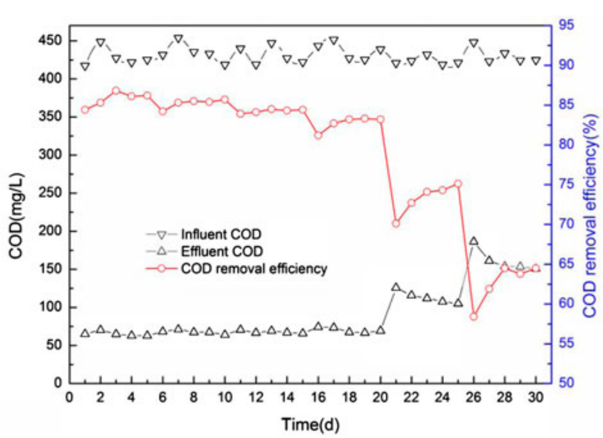

Fig. 5. Influence of HRT on performance of BAF.

In this study, the influent condition is relatively stable and the HRT was gradually shortened from 12 to 2 h, which is a gradual process and beneficial for the system stability. When HRT is more than 6 h, COD removal ratio can be over 83.2% on average. This indicates that majority of organics need more than 6 h to complete degradation process. When HRT is less than 6 h, the contact and reaction time is not enough. So part of organics are not fully degraded and flow out with the effluent, also short HRT means larger amount of influent wastewater and air, which can significantly intensify local turbulence. Biofilm may be washed off and this phenomenon was also observed in the experiment. Therefore, it is reasonable to control HRT between 6 and 8 for the proposed reactor.

3.2.3. Influence of organic loading on BAF performance

Generally speaking, organic loading of influent for biological wastewater treatment process should be controlled in a certain range. Too low and too high organic loadings both are not appropriate for acquiring satisfying performance. So it is essential to determine the appropriate organic loading level for the proposed reactor.

The organic loading verifying experiments operating conditions includes: influent flow 3.4 L/h, corresponding HRT about 6 h, the reactor temperature 28 ± 1℃, gas to water ratio five, and initial biofilm mass concentration about 15.75 g/L. Influent wastewater COD concentration is different to make the various organic loading conditions. Detailed influent COD concentrations and corresponding influent organic loadings are listed in Table 3. For each influent organic loading level, the reactor continuously ran for 6 d. Influent COD concentrations are given in the form of average COD concentration and its standard deviation in 6 d. Average organic loading is calculated by average COD concentration in each experimental stage and means the organic loading based on reactor volume. The influent and effluent COD of wastewater were tested every day and the results are shown in Fig. 6.

As it can be seen from Fig. 6, while influent organic loading increases, the average COD removal ratio increases first and then decreases. When organic loading is within the range of 0.3–0.77 kgCOD/m^{3}d, COD removal ratio increases rapidly from 65.7 to 83.1%, and effluent COD values are very close, which is around 56 mg/L. When organic loading is in the range of 0.77–2.04 kgCOD/m^{3}d, COD removal ratio increases from 86.3 to 83.1%, with a slower increasing rate. When the organic loading continues to increase

Table 3
Influent COD concentration and corresponding organic loading

No	1	2	3	4	5	6
Influent COD (mg/L)	160.3 ± 8.5	334.0 ± 7.8	522.7 ± 8.3	751.1 ± 9.5	882.7 ± 9.2	10,16.0 ± 10.4
Average organic loading (kgCOD/m^3 d)	0.37	0.77	1.21	1.74	2.04	2.35

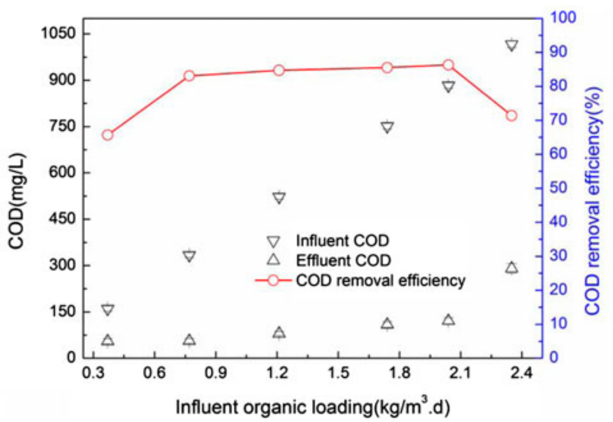

Fig. 6. Influence of different organic loadings on performance of BAF.

to 2.35 kgCOD/m^3 d, COD removal ratio decreases to 71.4% rapidly. It may be inferred that organic loading 2.35 kgCOD/m^3 d has exceeded the capacity of micro-organisms and metabolism activity of micro-organisms is restrained in some extent. Therefore, the BAF reactor maintains the influent organic loading within the range of 0.77–2.04 kgCOD/m^3 d is proper and it can achieve a relatively stable and effective performance.

4. Conclusions

RhB, a kind of common dye in textile industry, is hazardous and refractory to degradation in environment. It is used as representative organic pollutant to simulate textile wastewater in this study. A bench-scale BAF reactor was developed and employed for post-treatment of the simulated textile wastewater, which is effluent of RhB wastewater oxidized by Fenton reagent. The utilizability of the proposed BAF reactor for wastewater is investigated and the optimum operation conditions are acquired. By exogenous activated sludge inoculating and gradual acclimation method, the reactor can be started up successfully within 40 d. Operation condition experiments indicate that when gas–water ratio is 5–6, HRT is controlled at 6 ~ 8 h and influent organic loading is within the range of 0.77–2.04 kgCOD/m^3 d, the BAF can achieve relatively high performance.

Acknowledgments

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