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Analysis of iron ore sinter particles (IOSP) on the treatment of wastewater by a biological aerated filter

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ABSTRACT

Iron ore sinter particles (IOSP) and clay ceramic particles (CCP) were applied in two lab-scale up-flow biological aerated filters (BAF) for wastewater treatment to investigate the availability of IOSP used as biofilm medium compared with CCP. It took about 16 days for IOSP reactor to reach the stable effluent and 4 days late for CCP reactor, according to the removal efficiencies of chemical oxygen demand (COD_{Cr}) and ammonia nitrogen (NH_4^+-N). Compared with CCP reactor, higher efficiencies of COD_{Cr} and NH_4^+-N were found in IOSP reactor in the whole experiment. And there was a great difference on the final biomass densities (39.12 mg/g for IOSP and 25.95 mg/g for CCP). Better efficiency on removing total phosphorus (TP) under aeration was found in IOSP reactor (about 70%), while less than 40% in CCP reactor. Moreover, better capabilities responding to low pH in IOSP reactor were found compared with CCP reactor. For dye wastewater with C.I. Reactive Blue 19, it was shown that IOSP reactor had greater removal efficiency than CCP reactor.

Keywords: Iron ore sinter particles; Biological aerated filter; Total phosphorus; Low pH

1. Introduction

Biological aerated filters (BAF), which embrace numerous advantages citing an apparent example of filtration capacity [1], have been applied widely for wastewater treatment [2,3]. In BAF system, the selection of filter medium plays a significant role in maintaining the amount of active biomass and species of active microbe population. Many kinds of mineral support medium have been studied frequently, involved in clay, zeolite and plastic based ones, such as polyethylene (PE) and polyesterene (PS) [4–6]. Research also reveals that several waste materials such as grain-slag, waste ceramics, polyethylene plastic and blast furnace dust have been successfully utilized as filter medium for the biological aerobic filter (BAF) [2,3,7–9]. However, how to increase the biomass densities and enhance the activity of microorganism on the surface of filters is still a focal point in the field at present [10], and phosphate can be removed only if the BAF must be operated under alternating anaerobic and aerobic conditions [11], thus it is hard for microorganism to complete removing phosphate under purely aerobic conditions.

In this research, iron ore sinter was employed as the main material for fabrication of a novel kind of filter particles — iron ore sinter particles (IOSP). The characteristics of IOSP used as filter media were studied compared with clay ceramic particles (CCP) in two BAFs by treating synthetic wastewater. COD_{Cr} and NH_4^+-N removals were traced to provide a detailed statement about the growth of biofilms. Then the total phosphorus removals were

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studied under the condition of aeration. Moreover, the capability responding to the low pH of IOSP reactor and the relation between iron ions and microorganism were discussed in comparison with that of the CCP reactor. Finally, the results of removing C.I. Reactive Blue 19 were observed.

2. Experiment

2.1. Materials and experimental set-up

The filter media (IOSP) was made from Iron ore sinter (IOS), carbon (C) and calcium oxide (CaO). A precursor of IOSP was mixed with IOS, C and CaO at a ratio of 1:1:0.1 (g IOS/g C). The steps to incorporate the precursor as homogeneously as possible were as follows: (1) Transfer 50 g of the precursor into a granulator. (2) Adopt about 30 ml distilled water as cementing agent to mix with the above compound. (3) Operate the granulator for pelletizing. (4) Dry the solids at room temperature (about 20°C) for 12 h. (5) Sinter them at 1000°C for 60 min in a tube furnace. Cooled under ambient temperature, samples with diameter in the range from 3.0 to 6.0 mm were selected to perform the experiment.

CCP were supplied by Lixing Ceramic Particle Plant in the city of Jinan. The characteristics of IOSP and CCP are specified in Table 1 and Table 2.

Table 1
The comparison of characteristics between IOSP and CCP

Filter material	IOSP	CCP
Total porosity, %	1.81	24.7
Pore size distribution, μm	0.02–1.0	0.5–1.0
Particle diameter, mm	3–6	3–5
Bulk density, g cm^{-3}	1.24	1.89
Total surface area, m^2 g^{-1}	20.5	3.05

Table 2
The composition of raw materials (%)

	IOSP	CCP
SiO_2	3.19	71.3
Al_2O_3	1.225	14.6
MgO	1.605	2.89
CaO	5.515	1.99
Fe_2O_3, FeO	4.53	0.51
K_2O, Na_2O	0.069	0.46
C	40.6	0.002
Fe	27.12	0

The two lab-scale BAF reactors were set up as shown in Fig. 1. The reactors made from polymethyl methacrylat were designed similarly, each of which had a diameter of 100 mm and an effective volume of 1.41 L with a height of 900 mm. 0.4 L pebbles were placed at the bottom of the two columns respectively. 1.1 L filter media (IOSP or CCP) was filled in each column. The effluent port was located at 10 cm to the top.

The synthetic wastewater stored in the only feed water tank was injected to the bottom of each column, and two feed pumps were controlled at the same rate. The method synthetic wastewater flowed in both two columns was

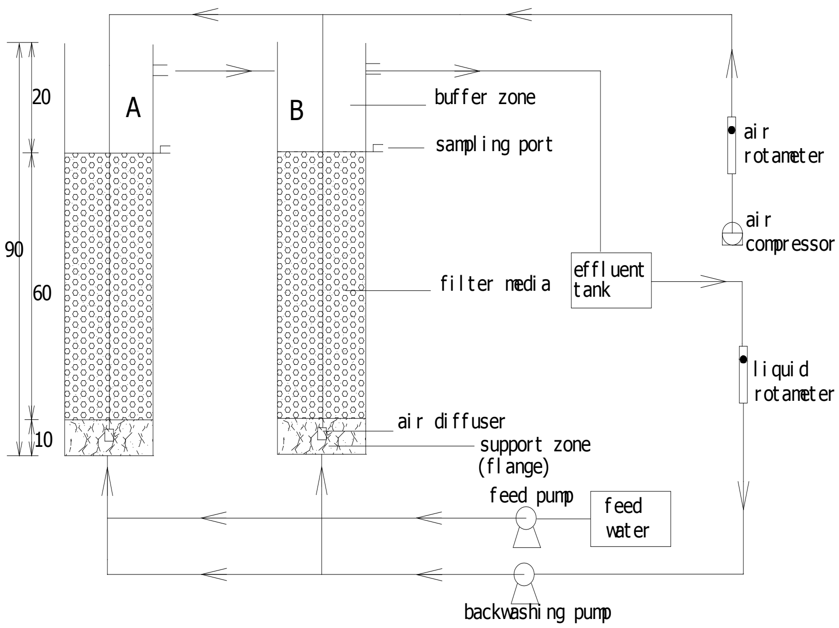

Fig. 1. Schematic diagram of experimental system (dimensioning unit: cm). A: column full of CCP; B: column full of IOSP.

upward and the effluent was collected in another tank for the purpose of backwashing. Because of the growth of biofilm, the two reactors were carried out to be backwashed every 48 h so as to prevent the columns being blocked. Three phases in proper order were included in the process: air scour, air–water scour and water scour. They were carried out as follows: firstly, air scour was started after turning off the feed pump for 10 min at the bottom of the column and the air rotameter was adjusted to a velocity of 3 L/min; then the valve linked to the backwashing pump was started and the liquid rotameter was adjusted to a velocity of 0.4 L/min; finally, air scour diffuser was stopped and recycled water was pumped to substitute for backwashing water of the second phase for another 10 min.

2.2. Wastewater characteristics

Through all the experimental period, the two reactors were fed with synthetic wastewater with glucose ($C_6H_{12}O_6$ — 187.5 mg/L), sodium acetate trihydrate ($CH_3COONa.3H_2O$ — 106.5 mg/L), ammonium chloride (NH_4Cl — 95.5 mg/L), potassium dihydrogen phosphate (KH_2PO_4 — 35 mg/L) and rare element solution to substitute the characteristics of raw domestic wastewater in Jinan, Shandong Province of China. They were diluted with tap water to the desired concentration before being injected to the columns.

2.3. Analytical methods

Samples from influent and effluent were taken regularly and the concentrations of COD_{Cr}, NH_4^+-N, nitrate nitrogen (NO_3^--N), nitrite nitrogen (NO_2^--N) and TP were measured according to standard methods [12]. UV-Vis Spectrophotometer (SP-752/752PC, shanghai) was used to measure the concentration of C.I. Reactive Blue 19, whose wavelength was 593 nm.

The characteristics of CCP and IOSP were evaluated by the instrument, Poremaster 60 (Quantachrome, USA). Scanning electron microscopy (SEM) (Hitachi S-520) was used to observe and analyze the surface of IOSP and CCP, and the microbial characteristics in the BAF reactors. Iron element in samples from IOSP reactor was conducted by inductively coupled plasma (ICP) (IRIS Intrepid II XSP equipment).

2.4. Inoculation and start-up

Seed sludge was inoculated with parameters as follows: SV = 31%, MLSS = 3.84 g/L, SVI = 76 mL/g. The two BAF reactors were operated under the same conditions. Throughout the period of start-up, basic operation parameters were chosen as follows: Hydraulic Retention Time (HRT) of 8.0 h, rate of aeration of 300 mL/min, temperature of 28–31°C. The columns was backwashed every other day after the 8th day from the beginning of inoculation, because in the first 8 days the biofilm was fixed not enough firmly. Samples should be taken before backwashing, because the backwashing would dilute the concentrations of pollutants in the effluent.

3. Results and discussion

3.1. Surface morphology of filter materials

According to Table 1, it can be seen clearly that IOSP were much better in many aspects, including larger total surface area and lower bulk density compared to CCP. These characteristics were necessary for filter materials applicable to use as filter media, in the light of the investigation of standard [13]. Fig. 2 shows the surface morphology of IOSP and CCP with SEM at the visual pore diameter of 100 μm. It can be seen that rougher surface of IOSP distributed with large pores was represented clearly compared with those of CCP. The surface mor-

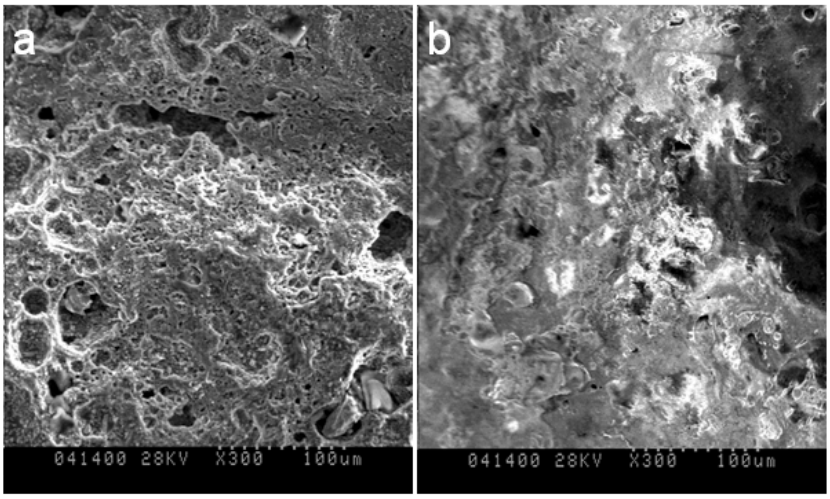

Fig. 2. SEM photographs on the IOSP (a) and CCP (b) surface.

phology well agreed with the results about total surface area in Table 1. It could be inferred that IOSP were more beneficial to the immobilization of microorganisms if they were utilized as filter media.

3.2. Reactors performance during start-up

As can be seen in Fig. 3, the removal efficiencies of COD_{Cr} and NH_4^+-N changed irregularly. It took about 20 days until the removal efficiencies of COD_{Cr} (a) and NH_4^+-N (b) reached over 95% and 83% respectively. From the 5th day, it can be shown that IOSP column had greater removal efficiencies than CCP column, which demonstrated that the microorganisms adapted to the environment more quickly and stable in the IOSP than in the CCP. Because IOSP had lager total surface area than CCP, the population of microorganisms adhering on the surface of IOSP could be a larger number, thus lager total surface area of IOSP could be responsible for the increased removal efficiency of the system. There was another important reason for the phenomenon. Due to the presence of iron (Fe) and carbon (C), primary cell was formed in the IOSP, resulting in the production of iron (Fe^{2+} or Fe^{3+}) which has the function of keeping nitrobacteria active or even enhancing the activity [14].

The removal efficiency dropped quickly in the first 4 days as shown in Fig. 3a, though it was relatively high in the first day, which was even apparent for that of NH_4^+-N shown in Fig. 3b, because the microorganisms from seed sludge had relatively high removal efficiencies for the wastewater at the beginning. But due to the weak stability of the biofilm in the phase, some of the microorganisms on the surface of particles could be washed away. On the 4th day, the initial biomass densities were 3.93 mg/g and 3.89 mg/g for IOSP and CCP respectively. It was concluded that there was little difference on the biomass densities in the initial phase. Since the 5th day COD_{Cr} and NH_4^+-N removal increased because of the growing of the heterotrophic bacteria, while NH_4^+-N removal efficiency was still low because the nitrifying bacteria were autotrophic and have a long generation cycle [15]. Since the 8th day, backwashing was carried out, and NH_4^+-N and COD_{Cr} removal efficiencies fluctuated up and down for the first 3 days of backwashing period, which may be caused by the growing nitrifying bacteria and other biomass that attached on the particles infirmly. From the 11th day to the 15th day, NH_4^+-N removal efficiency in the CCP column fluctuated up and down, while increasing smoothly in the IOSP column, which showed that the microorganisms adapted to the environment in IOSP faster than that in CCP. On the 16th day, the CODCr removal kept steady while NH_4^+-N removal rose, and this illustrated that nitrifying bacteria took precedence.

In the last 5 days, both NH_4^+-N removal and COD_{Cr} removal in the IOSP column worked out smooth, which reached conclusion that a balance of competition was achieved between heterotrophic bacteria and nitrifying bacteria. Start-up period was completed for removal efficiencies reached steady state [16], while it was about 4 days late for the balance between heterotrophic bacteria and nitrifying bacteria in the CCP column. At this time, there was a great difference on the final biomass densities (39.12 mg/g for IOSP and 25.95 mg/g for CCP). The reasons for the difference could be explained by larger surface area and the effect of iron ions for the microorganism as recounted before.

In order to study the NH_4^+-N removal further, NO_3^--N and NO_2^--N were traced as shown in Fig. 4. There was little production of NO_2^--N in both reactors, even if the content of NO_2^--N in CCP was higher than that in IOSP. It might be implied that the denitrification existed with little level. However, there was a conspicuous difference — the production of NO_3^--N from effluent. Reactor of IOSP performed better in the production of NO_3^--N all through the experiment, compared with reactor of CCP, which indicated that the rate of nitrification in reactor of IOSP was higher than that in reactor of CCP. Therefore, this section illustrated the difference of NH_4^+-N removal between two reactors from the point of trace of nitrogen.

Fig. 5 shows the SEM photographs on the IOSP (a) and CCP (b) surface after start-up. It can be seen that the biofilms formed on the surface of the two materials

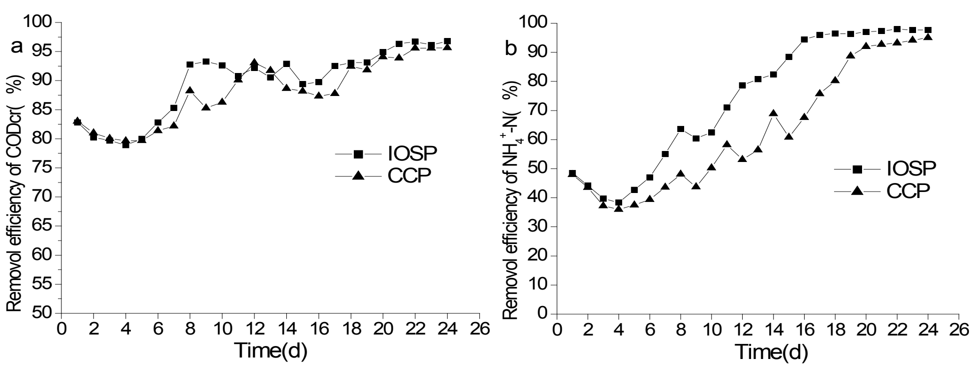

Fig. 3. Removal efficiencies of COD_{Cr} (a) and NH_4^+-N (b) during start-up.

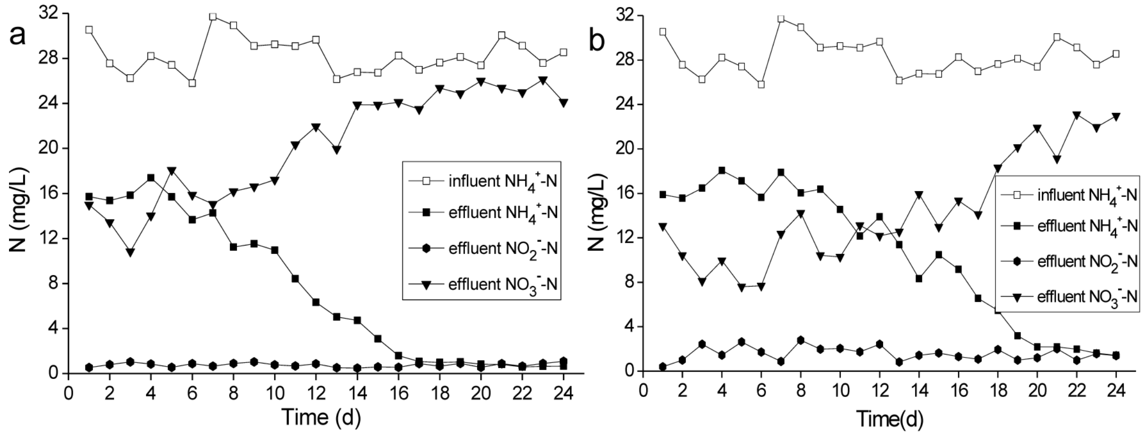

Fig. 4. Time courses of various nitrogen in BAFs (a) IOSP; (b) CCP.

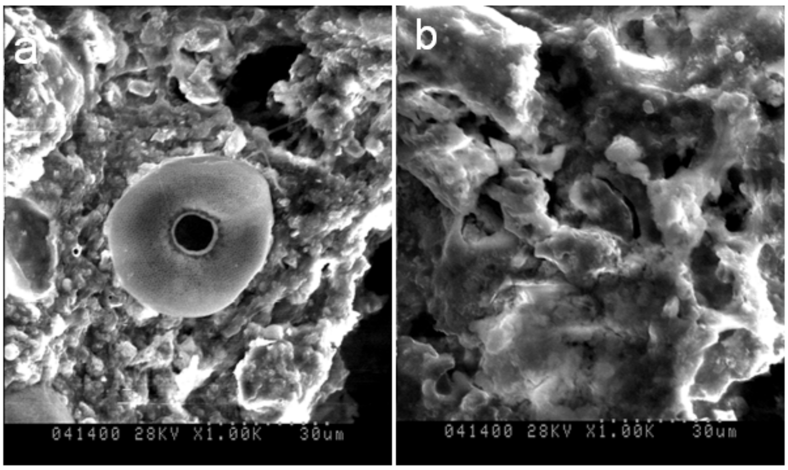

Fig. 5. SEM photographs on the IOSP (a) and CCP (b) surface after start-up.

grew well. However, some large-physique microorganisms survived on the surface of IOSP, which might be explained that the rougher surface and lager total surface area of IOSP could provide more opportunities to enhance microbial diversity.

3.4. *TP removals in two columns during start-up*

Due to the presence of iron (Fe) and carbon (C), primary cell was formed in the IOSP, resulting in the production of iron (Fe^{2+} or Fe^{3+}) which was combined with phosphate ion (PO_4^{3-}) to constitute precipitate of ferrous phosphate ($Fe_3(PO_4)_2$) or ferric phosphate ($FePO_4$) [17]. Thus TP removal could be achieved promptly even if in the condition of aeration.

As can be shown in Fig. 6, it took about 13 days until TP removal efficiency of two reactors kept steady. On the first day, as the wastewater contacted with IOSP absolutely, high removal efficiency (about 64%) was reached from Fig. 6a. However, as the accumulation of ferrous phosphate on the surface of IOSP, the TP value of effluent rose gradually in the following days. Moreover, there was another reason that sludge and other litter excreted from bacteria was considered to be another obstacle between IOSP and wastewater, which could be certificated by the fact that the first backwashing was carried out on the 8th day and the removal efficiency raised a little. But the effluent TP began to be steady from the 9th day, in spite of another time of backwashing, revealing that the reaction to ferrous phosphate between Fe^{2+} and PO_4^{3-} reached their balance.

In addition, due to the characteristics of filter media, TP removal efficiencies of CCP reactor remained below 16% from Fig. 6b during the period, whose effect was far inferior to that of IOSP. As is well known, it is under the alternative aerated and anaerobic condition that phosphate can be utilized and removed by poly-P bacteria [18]. The experiment was carried out in the condition of aeration,

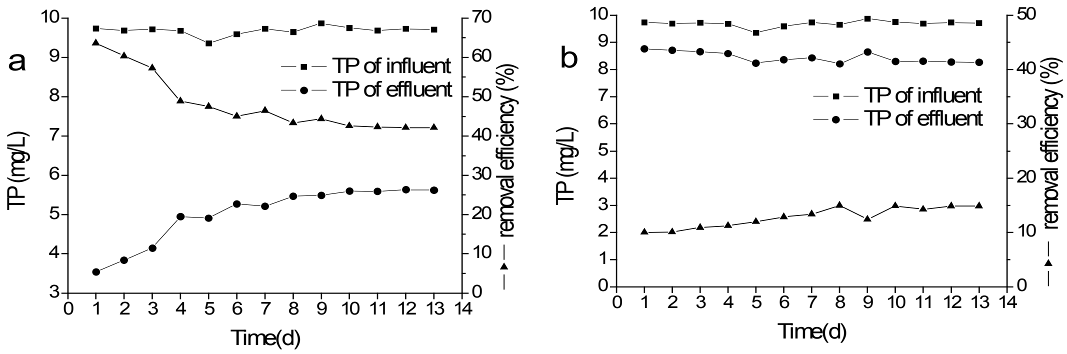

Fig. 6. TP removal in IOSP column (a) and CCP column (b).

so TP removal in the CCP column was considered as the fact that phosphate was utilized by microbe as nutrition. It was hard to survive in the surrounding of aeration all the time for poly-P bacteria.

3.5. *Effect of iron in low pH*

In order to investigate the relationship between low pH and the effect of treatment, 15 days were taken to complete the experiment. From 8th day, the condition of low pH was adopted for two reactors. Throughout the experiment, influent pH value was controlled ranging from 5.2 to 5.8, by adding diluted hydrochloric acid (HCl).

An investigation on the relationship between effluent pH value and influent pH value for two reactors was shown in Fig. 7a. Due to the difference of composition (Table 2) between IOSP and CCP, two materials showed different capabilities responding to low pH. For the first two days, it can be seen that effluent pH value of IOSP was higher than that of CCP, it was because that oxides on the surface of IOSP such as ferric oxide (Fe_2O_3) and ferrous oxide (FeO) were reacted with hydrogen ion (H^+) firstly, and Fe^{3+} or Fe^{2+} was produced (Fig. 7b), while most percentage of oxide on the surface of CCP was silicon dioxide (SiO_2), leading to the difficulty in receiving H^+. However, the capabilities responding to low pH of two materials were inverted absolutely from the 10th day. Because the reaction between H^+ and other oxides (CaO, MgO etc.) took precedence, and it was known from above that the percentage of the oxides (CaO, MgO etc.) of the CCP was higher than that of IOSP. But both pH values of effluent were below 6.4 through the 8 days.

As is revealed in Fig. 8, the removal efficiencies of COD_{Cr} (a) and NH_4^+-N (b) in two reactors showed different tendencies under the pH ranging from 5.2 to 5.8 during the later 8 days. In the first 7 days, both of the removal efficiencies of COD_{Cr} and NH_4^+-N in two reactors kept steady and remained 94% and 93%, respectively. However, from the 8th day, the difference of two reactors on the removal efficiencies began to appear. The removal efficiencies of COD_{Cr} and NH_4^+-N in CCP decreased sharply, while the effect on the removing COD_{Cr} and NH_4^+-N became better than that before in IOSP column. In general, lower pH value suppresses nitrification but not in the biofilter with IOSP, which could be explained by the effect of Fe^{3+} or Fe^{2+} that is capable of keeping nitrobacteria active or even

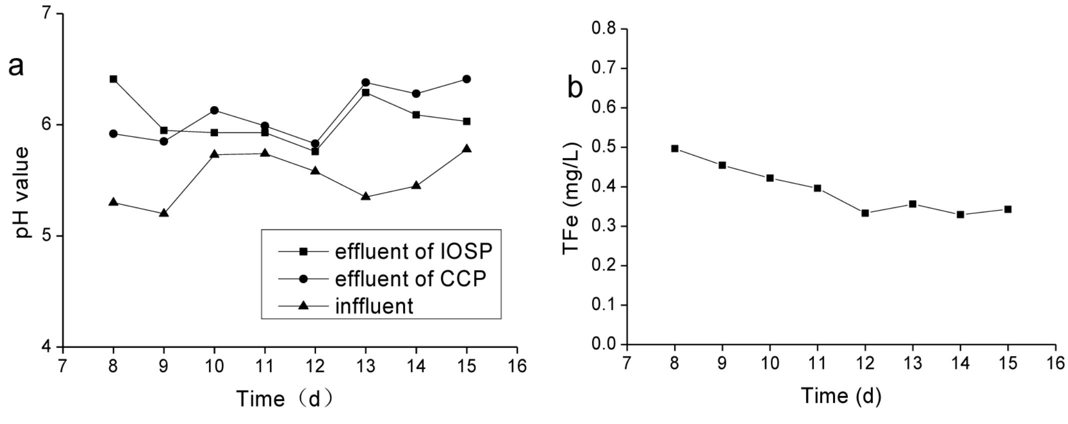

Fig. 7. Relation between effluent pH value and influent pH value (a) and the content of total iron in the IOSP reactor (b) from 8th day.

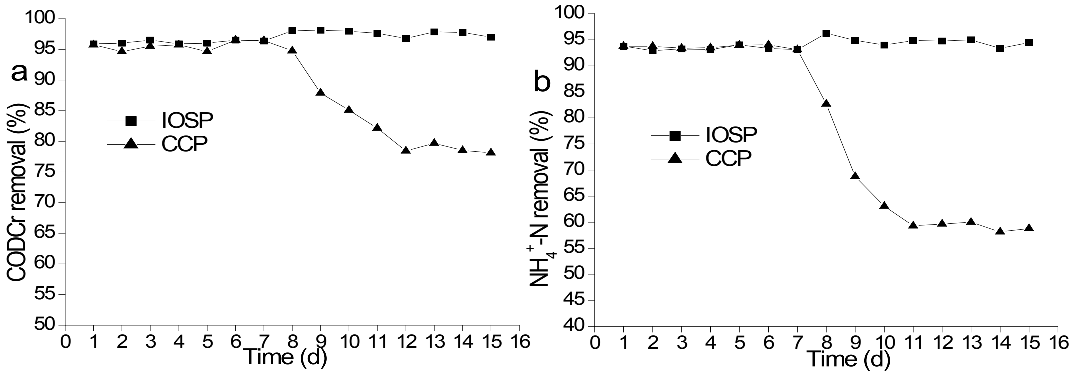

Fig. 8. Removal efficiencies of COD_{Cr} (a) and NH_4^+-N (b) in low pH.

enhancing the ability [14]. Theoretically, nitrification is pH-sensitive and rates decrease significantly at pH values below 6.8. Optimal nitrification rates take place at pH values in the range of 7.5–8.0 [19,20].

Both IOSP and CCP can adjust low pH in some degree, but both of them could hardly reach the acceptable pH to the nitrobacteria. However, due to its high percentage of iron in the composition of IOSP, Fe^{3+} and Fe^{2+} could be produced from IOSP in low pH (Fig. 8 b) to keep nitrobacteria active or even enhance the ability [14]. In the CCP reactor, rare iron was found, and it was hard to keep nitrobacteria active, even if CCP could reach higher pH than IOSP in low pH.

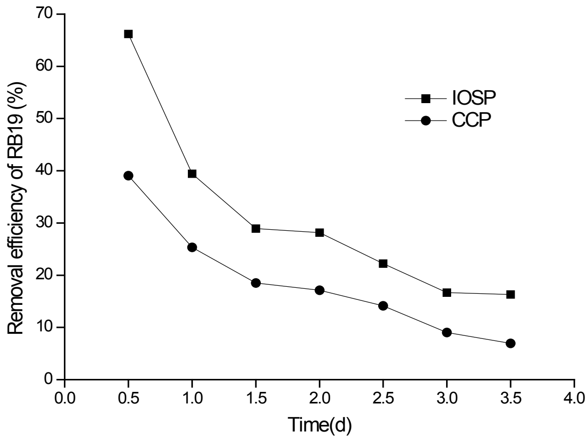

Fig. 9. The performance of two reactors on the removal of RB19.

3.6. *Efficiencies for C.I. Reactive Blue 19*

For the purpose of investigating the effect of two reactors on removing some kind of dye, C.I. Reactive Blue 19 (RB19) was chosen for the experiment. The concentration of RB19 in the influent was kept in the range of 21–27 mg/L. It is well known that RB19 is adsorbed poorly by biomass, and do not degrade under the condition of typical aeration found in conventional biological treatment plants [21,22]. Fig. 9 reveals the curves of two reactors on the removal of RB19. IOSP showed much higher removal efficiency than CCP (IOSP of 69% compared with CCP of 39%) in the beginning at the very start, and the efficiency of IOSP was higher all through the experiment than that of CCP, which could be explained by the fact that the iron oxides possess adsorption capability due to the intervention of hydroxyl groups during dissociative chemisorption of the adsorbate [23]. The removal efficiencies of two reactors decreased gradually with time. There were about three reasons for the phenomenon. First of all, the content of iron oxides in reactor of IOSP was limited, so the adsorption capability could be decreased gradually. In addition, as the tendency to the saturation of adsorption for RB19 on the surface of two kinds of media, the removal efficiencies decreased. Moreover, the biofilm and sludge including some litter excreted from bacteria had ability of absorbing in some degree [24]. However, it might be implied that reactor of IOSP could have relatively higher efficiency on the removal of RB19. In other words, IOSP can be utilized as pretreatment for the wastewater with RB19.

4. Conclusions

The microorganisms adapted to the environment in IOSP faster than that in CCP as IOSP had larger total surface area, and performed better in IOSP reactor than in CCP reactor for efficiencies of COD_{Cr}, NH_4^+-N removals and C.I. Reactive Blue 19 removals. Greater ability of removing total phosphorus under the condition of aeration was found in IOSP reactor, compared with CCP reactor. Moreover, IOSP reactor had significantly better capability responding to low pH than CCP reactor. Based on the above results, it was demonstrated that IOSP could be applied in the wastewater treatment as a novel filter medium.

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