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Simultaneous removal of ethyl acetate and toluene in air streams using compost-based biofilters

Yonghui Liu [a], Xie Quan [a,*], Yumei Sun [a,b], Jingwen Chen [a], Daming Xue [a], Jong Shik Chung [c]

[a] *School of Environmental Science and Technology, Dalian University of Technology, Zhongshan Road 158-129, Dalian 116012, PR China*

[b] *Department of Bioengineering and Food Science, Dalian Institute of Light Industry, No. 1 QinggongYuan Road, Ganjingzi District, Dalian 116034, PR China*

[c] *School of Environmental Engineering, Pohang University of Science and Technolgy, San 31, Hyoja-Dong, Pohang 790-784, South Korea*

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Abstract

Biofitration was successfully applied to treat air streams containing a mixture of ethyl acetate and toluene. The experiment was performed by two identical bench-scale biofilters, which were acclimated by ethyl acetate and toluene, respectively. During a 3 month steady-state performance, the two biofilters showed equivalent elimination capacity (EC) for toluene (50 g/m^3 bed/h of pure toluene). However, the biofilter acclimated with ethyl acetate showed a much higher EC for ethyl acetate (400 g/m^3 bed/h of pure ethyl acetate) than that acclimated with toluene (250 g/m^3 bed/h). The concurrent biofiltration of toluene was inhibited by the presence of ethyl acetate. The results also showed that more nitrogen and phosphorus were consumed in the process of the biofiltration of toluene compared with the treatment of ethyl acetate. After the 3 month experiment, the pH of the media treating ethyl acetate dropped from 6.71 to 5.50, whereas the pH of the media treating toluene increased from 6.71 to 7.08.

Keywords: Biofitration; Ethyl acetate; Toluene; VOC; Media

1. Introduction

Air streams discharged from a variety of industries contain volatile organic compounds (VOCs), which are controlled by increasingly stringent environmental regulations. In the

* Corresponding author. Tel.: +86-411-3685113; fax: +86-411-3685113.
E-mail address: xiequan@mail.dlptt.ln.cn (X. Quan).

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color printing works, ethyl acetate and toluene are among the key pollutants included in exhaust air. Ethyl acetate is a kind of irritative and explosive compound with fragrant odor, which is harmful to respiratory systems of mankind. Toluene is a toxic and hydrophobic compound listed in Title III of the 1990 Clean Air Act Amendment proposed by US-EPA. Ethyl acetate, toluene, and some other VOCs in the emissions of color printing works and other industries, have been the subject of recent environmental regulations in China (GB16297-1996, China). The relevant enterprises are thus required to adopt appropriate technologies to reduce those VOCs in the emissions. The current control technologies for these VOCs, such as thermal incineration and wet scrubbing, are usually costly, especially in cases when the concentrations of these pollutants are not high.

In the field of air pollution control, it is a relatively new technology to treat emissions containing VOCs by biofitration. It has raised increasing interests in recent years because it is less expensive and more effective than those traditional methods [1–3].

The biofiltration of ethyl acetate [1,4] and toluene [5–9] has been investigated by some previous studies. Deshusses and Johnson [10] studied the biofiltration of ethyl acetate and toluene as a mixture. In their experiment, a temperature increase and drying-out test were conducted and a byproduct of ethyl acetate was observed. However, few studies, if any, have revealed the change of media content and pH during the biofiltration of the mixtures. Furthermore, no research work has been found concerning the acclimation approach and explaining the interaction between theses two compounds, although the coexistence of ethyl acetate and toluene has been found in a large number of emissions, and interactive inhibition has been observed in the biofilters treating a mixture of different VOCs.

It is the purpose of this study to investigate the biofiltration of gas stream containing a mixture of ethyl acetate and toluene. The biofilters to be studied were acclimated with ethyl acetate and toluene, respectively. The maximum removal rates for biofilters and the interactions between ethyl acetate and toluene were reported. In addition, the variations of the total organic matter content (TOM), total nitrogen (TN), total phosphorus (TP) and ammonia nitrogen (NH_3–N) content were determined.

2. Materials and methods

2.1. Biofilter system

As shown in Fig. 1, two parallel bench-scale biofilters A and B, were used in this experiment. Lava (3–5 mm), compost and soil were used as packing media. The volume proportion of the mixture is 3:5:2. The initial moisture content of the media was about 50%. The biofilters, made of polymethyl methacrylate, consists of three segments (top, mid and bottom segments) connected in series. Each segment has an internal diameter of 7.5 cm and a height of 30 cm. There was an outlet at the top of each segment for sampling the air stream along the column and providing an access to the media. Water was sprayed every day from a nozzle at the top of the biofilters to maintain proper moisture content in the packing media.

The two VOCs were produced by passing air through a water-bathed vessel containing liquid ethyl acetate and toluene. The concentrations were controlled by adjusting the flux of air stream and the temperature of the water bath. Air was first passed through a humidifying

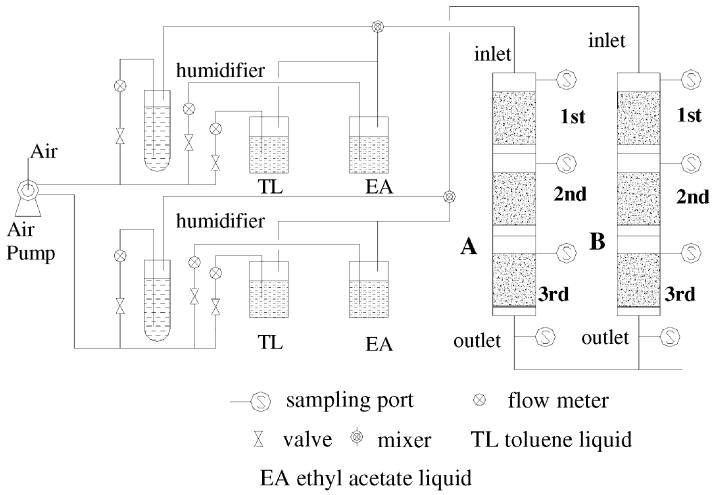

Fig. 1. Schematic diagram of experimental system.

equipment and then mixed with the air streams carrying VOCs. The contaminated air stream was fed to the biofilters from the top.

2.2. Operation of the biofilters

During a 20 day acclimation period, the two biofilters were exposed to various concentrations of ethyl acetate and toluene. The number of the microorganisms in the media was counted after the acclimation. The biofilter acclimated with ethyl acetate is referred to as biofilter A, and the other one, which was acclimated with toluene, is referred to as biofilter B. The biofiltration of ethyl acetate and toluene, separately or jointly, was investigated for three months at various loading rates (grams of ethyl acetate and toluene per cubic meter of medium per h). Table 1 lists the VOC concentrations in the air emissions of one particular color printing works. In the current experiment, the average influent concentration of ethyl acetate and toluene was twice as that listed in Table 1. In view of the fact that toluene is much more difficult to eliminate through biofiltration than ethyl acetate, toluene and ethyl acetate were introduced herein at a high ratio than those reported in Table 1.

The biofiltration experiment was performed at normal room temperature (20–25 °C). Water, approximately 3–5 ml, was sprayed after the sampling each day. The contaminated

Table 1
VOC concentrations in the emission from a particular working site

VOC	Highest concentration (mg/m^3)	Lowest concentration (mg/m^3)	Average concentration (mg/m^3)
Ethyl acetate	3500	261	1000
Toluene	323	21	100

air stream to the biofilter was then stopped for about 20 min. The gravimetric moisture content in the media was measured once a week, to be about 50%. The contents of TOM, TN, TP, NH_3–N and pH of the media were also monitored periodically.

2.3. Analytical methods

The gas-phase concentrations of ethyl acetate and toluene were determined using a gas chromatography (Chromatography 1102, Shanghai, China) equipped with a flame ionization detector (FID). The inlet and outlet streams, as well as those between the biofilter segments, were sampled using a 100 ml injector. A 1 ml air stream sample was taken from the injector and injected into the gas chromatograph using a gas-tight syringe.

The gravimetric moisture content of the media was measured by weight loss after the medium sample was dried at 120 °C to a constant weight. For determining TOM, TN, TP and NH_3–N, 5 g solid samples were taken from the sampling ports of the biofilters, impregnated into distilled water and separated from the water phase. The concentrations of the TOM, TN, TP and NH_3–N in the effluent water were determined according to China Standard Analytical Methods [11–13]. To determine the pH value of the media, samples were taken using the method introduced by Liang et al. [14], and pH of the media was measured by a digital pH meter (PHS-3B, Hongzhou, China).

For microbial counting, 2 g of media was taken from the entrance of the biofilter, 100 ml sterilized water was added to each media sample, and then mixed well. The mixed solution were inoculated into agar medium and allowed to grow for 2–3 days in the incubator at 30 °C before counting the colonies. Bacteria were cultured in beef extract and peptone agar medium. Fungi were cultured in potato extract and glucose agar medium. The counting experiments were performed in triplicate.

3. Results and discussion

Bulk VOC elimination capacities of biofilter A and biofilter B were measured at different periods of the experiment as a function of VOCs influent concentrations and loads. Bulk load (L), elimination capacities (EC), and removal efficiencies (RE) were calculated as follows:

$$L = \frac{3.6 C_{\text{in}}}{\text{EBRT}} \tag{1}$$

$$\text{EC} = \frac{3.6(C_{\text{in}} - C_{\text{out}})}{\text{EBRT}} \tag{2}$$

$$\text{RE} = \frac{C_{\text{in}} - C_{\text{out}}}{C_{\text{in}}} \times 100\% \tag{3}$$

where 3.6 is a conversion factor that converts mg to g and s to h. Therefore, EC and L are in unit of g of the pollutant per m^3 of medium and per h ($g\,m^{-3}\,h^{-1}$). The inlet and outlet concentrations, C_{in} and C_{out}, were measured in $mg\,m^{-3}$. The empty bed retention time (EBRT) is 30 s for one segment and 90 s for the entire column. Thus, three EC values

can be obtained for a given inlet concentration of ethyl acetate or toluene, corresponding to the first, second, and third segment of the biofilters, respectively. In this experiment, ethyl acetate and toluene were generally eliminated in the first two segments. The third segment played a minor role in the removal of VOCs, thus, the third segment will not be discussed in detail.

3.1. Acclimation of the media

During the 20 day acclimation period, the two biofilters were operated with EBRT of 90 s. In the first 2 weeks, the loads of ethyl acetate and toluene were about 400 and 40 g m^{-3} h^{-1}, respectively. After acclimation of 11 days, the removal efficiencies of the two biofilters for ethyl acetate/toluene reached 99%, which is in contrast to the initial removal efficiencies, below 5%. In the subsequent 2 days, no obvious decrease in removal efficiency was observed. Then, the inlet concentrations of ethyl acetate and toluene were gradually increased. With nearly constant inlet VOC concentrations, the removal efficiency of the biofilters was quite stable for 2 days, indicating that the biofilters had reached a microbial steady state. Microbial counting was then performed, for which the results are shown in Table 2. As can be found from Table 2, after the 20 day acclimation, the fungi number of biofilter A acclimated with ethyl acetate was higher than that of biofilter B acclimated with toluene.

3.2. Performance of biofilter A

The performance of biofilter A was divided into three operating stages (Fig. 2a and b). In the first stage (for which the total influent gas volumes varied from 0 to 19 m^3), the biofilter was run with ethyl acetate only. Toluene was introduced in the second operating stage, for which the total influent gas volumes varied from 19 to 50 m^3. In order to study the biofilter's response to the variation of VOC concentrations, inlet concentrations of ethyl acetate were increased from 3500 to 4500 mg m^3 and then dropped to 2500 mg m^3. The concentrations of both toluene and ethyl acetate were gradually increased in the third operating stage (gas volumes: >50 m^3), thus the maximum elimination capacities of the biofilter could be evaluated.

During the 3 month operation, the third segment played a minor role in removing VOCs, because the VOC concentrations at the end of the second segment (EBRT = 60 s) were too low to be detected by GC. Therefore, the data for the third segment are not presented here. Fig. 2a and b show that most ethyl acetate was removed at the end of the second segment (EBRT = 60 s). In addition, 100% removal of toluene was achieved in the second operating

Table 2
The number of microorganisms in the media before and after acclimation

Compost	Bacteria (CFU)	Fungi (CFU)
Before	10^7	10^5
After 20 days acclimation (biofilter A)	10^8	10^7
After 20 days acclimation (biofilter B)	10^8	10^5

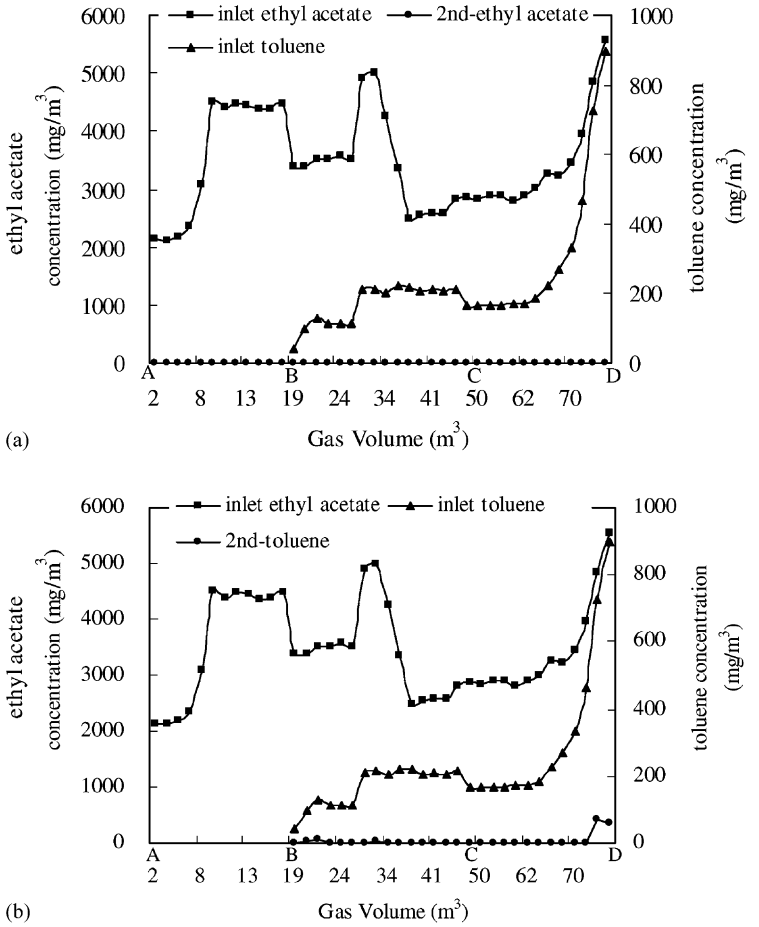

Fig. 2. The performance of biofilter A after the acclimation period. Notes: stage 1, A–B; stage 2: B–C; stage 3: C–D; (a) inlet VOC concentrations and the concentration of ethyl acetate at the end of second segment (EBRT = 60 s); (b) inlet VOC concentrations and the concentration of toluene at the end of second segment (EBRT = 60 s).

stage with EBRT of 60 s. Only when ethyl acetate and toluene concentrations exceeded 4500 and 600 mg m^{-3}, respectively, can toluene be detected by GC at EBRT of 60 s.

Fig. 3a and b show the ethyl acetate and toluene removal efficiencies after the first segment of biofilter A (i.e. EBRT = 30 s). The average ethyl acetate removal efficiency was about 85% with ethyl acetate inlet concentrations between 2000 and 4500 mg m^{-3} when toluene was not present in the influent air stream. When toluene was introduced and its influent concentration reached about 130 mg m^{-3}, a decrease in the ethyl acetate removal efficiency was observed, which may be due to the transient state of the biofilter at the beginning of toluene introduction. When toluene concentrations were increased from 130 to 400 mg m^{-3}, ethyl acetate removal efficiencies were not affected obviously. The results imply that the presence of toluene at a level of less than 800 mg m^{-3} does not affect the removal of

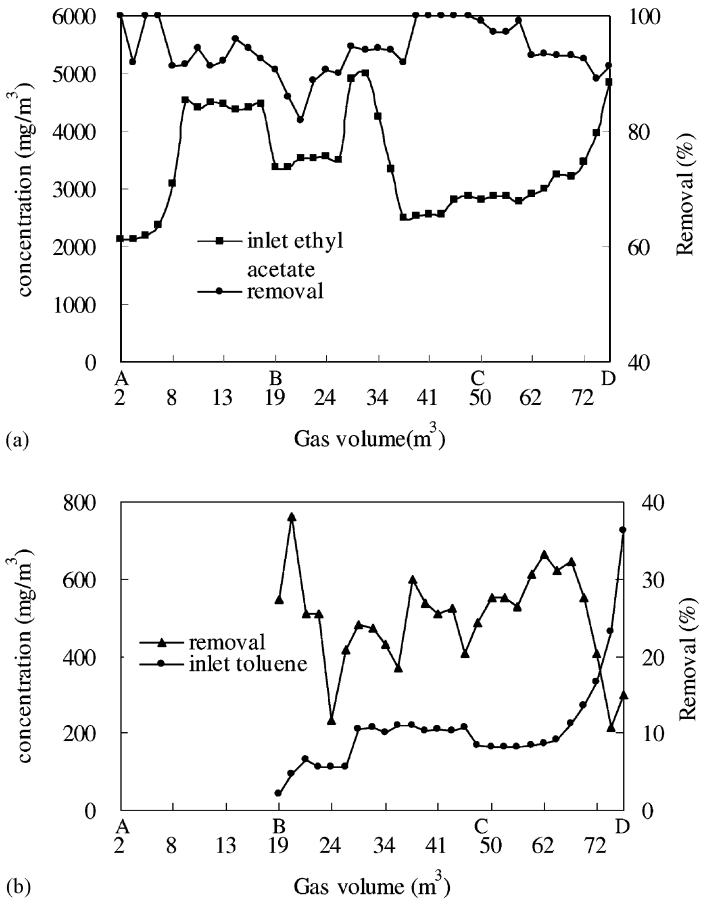

Fig. 3. Ethyl acetate and toluene removal efficiencies after the first segment of biofilter A (EBRT = 30 s). Notes: stage 1, A–B; stage 2: B–C; stage 3: C–D; (a) ethyl acetate; (b) toluene.

ethyl acetate. During the entire experiment, toluene removal efficiencies were very low (20–40%), suggesting that the removal of toluene was effected at the presence of ethyl acetate.

Fig. 4a and b show the elimination capacities for ethyl acetate after the first segment and for toluene after the second segment. Fig. 4a shows that whether or not toluene was present in the inlet air stream, the elimination capacities for ethyl acetate after the first segment were greater than 400 g m^{-3} h^{-1}. Due to the suppression of ethyl acetate, toluene was mainly degraded in the second segment. The maximum EC for toluene was about 50 g m^{-3} h^{-1} after the end of the second segment. When toluene load was beyond 60 g m^{-3} h^{-1}, the elimination capacities for toluene did not increase obviously with the increase of the load, indicating that the biofiltration followed zero-order kinetics in this concentration range, in agreement with “the reaction limited” scenerio [15].

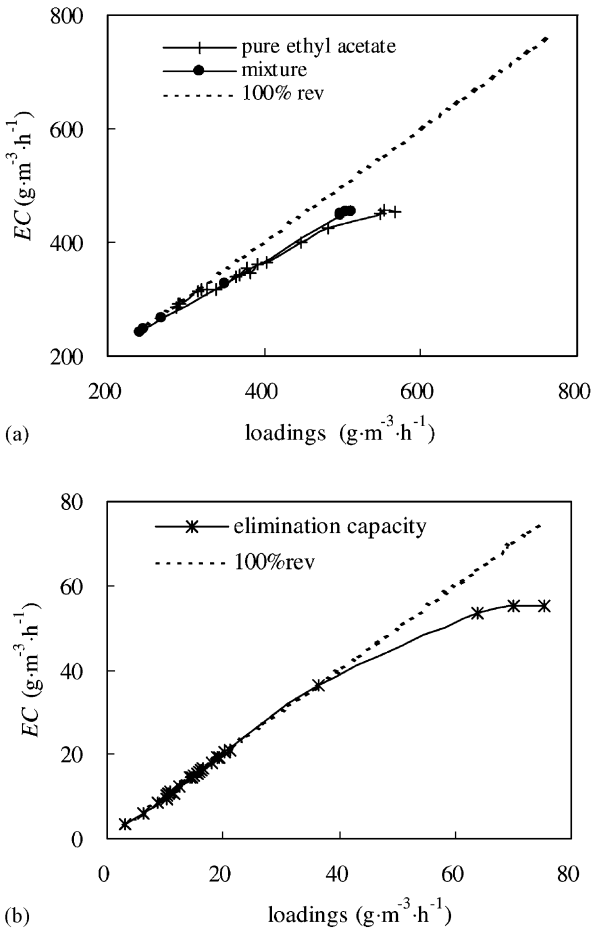

Fig. 4. The EC for ethyl acetate after the first segment and the EC for toluene after the second segment of biofilter A (a) ethyl acetate; (b) toluene.

3.3. *Performance of biofilter B*

The performance of biofiler B (Fig. 5a and b) was also divided into three operating stages. The removal of pure toluene was studied in the first stage (for which the total influent gas volumes varied from 2 to 21 m^3). In the second stage (for which the total influent gas volumes varied from 21 to 54 m^3), with toluene concentrations ranging from 200 to 400 mg m^{-3}, ethyl acetate was introduced with concentrations ranged from 400 to about 1500 mg m^{-3}. In the third stage, the level of ethyl acetate was increased to 2300 mg m^{-3} and the level of toluene was kept around 350 mg m^{-3}. For the three operating stages, both ethyl acetate and toluene could not be detected at the end of the third segment, than could ethyl acetate at the end of the second segment. The highest effluent concentration for toluene was below 10 mg m^{-3} at the end of the second stage (EBRT = 60 s).

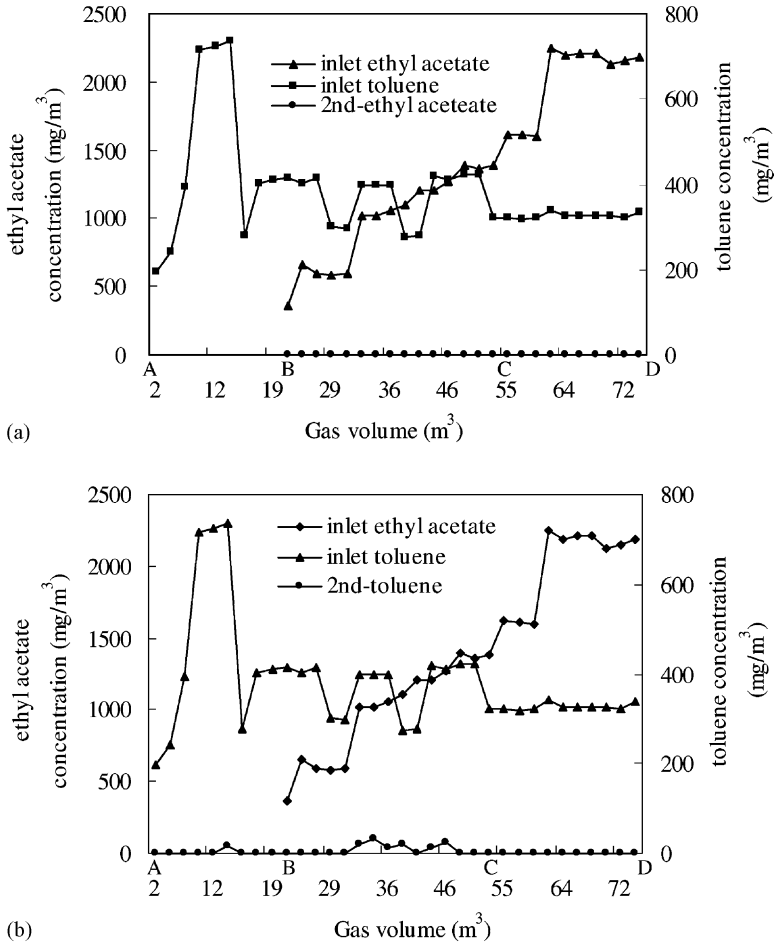

Fig. 5. The performance of biofilter B after the acclimation period. Notes: stage1, A–B; stage 2, B–C; stage 3, C–D; (a) inlet VOC concentrations and the concentrations of ethyl acetate at the end of second segment (EBRT = 60 s); (b) inlet VOC concentrations and the concentration of toluene at the end of second segment (EBRT = 60 s).

The removal efficiencies for ethyl acetate and toluene after the first segment are shown in Fig. 6a and b. As shown in Fig. 6b, toluene removal efficiencies dropped from nearly 100 to about 40% with the inlet toluene concentration increased from 400 to 700 mg m^{-3}, and the removal efficiencies did not recover during the following 3 days. However, the removal efficiencies recovered rapidly as the inlet toluene concentrations declined from 700 to 300 mg m^{-3}. Toluene removal efficiencies decreased from nearly 100 to about 80% with the introduction of ethyl acetate at concentration of about 600 mg m^{-3}. With the increase of ethyl acetate concentrations in the inlet air stream, toluene removal efficiencies decreased gradually. No remarkable removal of toluene was observed throughout the first segment after the inlet concentration ratio of ethyl acetate to toluene was greater than 4:1, suggesting that the biodegradation of toluene was suppressed by the presence of ethyl acetate. In the

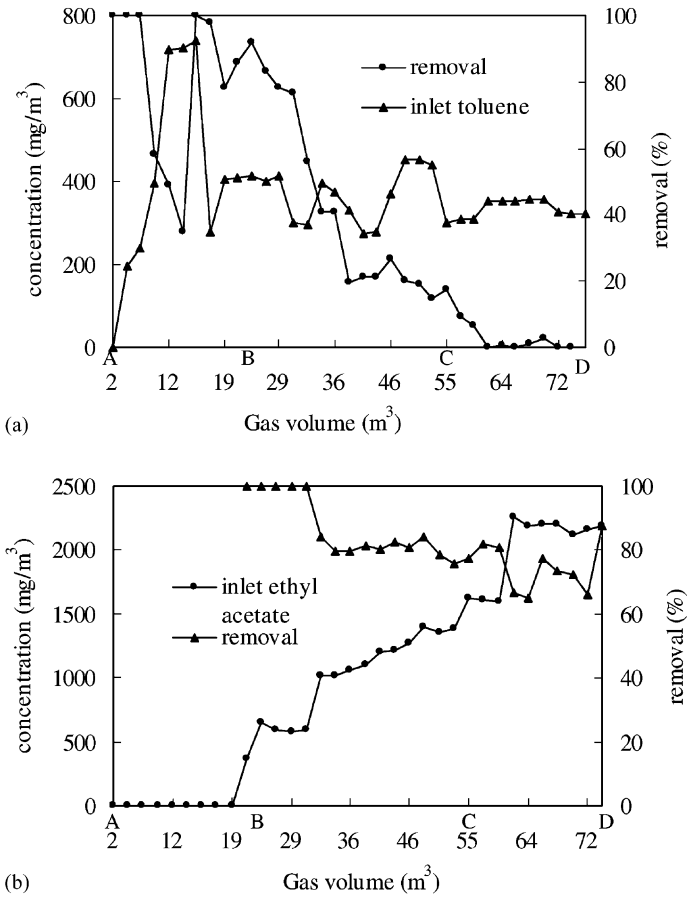

Fig. 6. Ethyl acetate and toluene removal efficiencies after the first segment of biofilter B (EBRT = 30 s). Notes: stage 1, A–B; stage 2: B–C; stage 3: C–D; (a) ethyl acetate; (b) toluene.

third stage, toluene removal efficiency dropped steadily to 0% when ethyl acetate inlet concentrations were around 2300 mg m^{-3} with corresponding average removal efficiency about 70%. This may due to the inhibition effects of ethyl acetate, which is in agreement with reports that ethyl acetate concentrations exceeding 500–2000 mg m^{-3} apparently inhibit the elimination of toluene [10,15]. The removal efficiency of ethyl acetate was below 80%, much lower than that of biofilter A, when its concentration was approximately 1500 mg m^{-3}.

The elimination capacities of ethyl acetate after the first segment of biofilter B are shown in Fig. 7a. Toluene elimination capacities after the first segment without the presence of ethyl acetate (pure toluene) and the elimination capacities after the second segment with ethyl acetate in the inlet air stream are shown in Fig. 7b. The maximum EC for ethyl acetate after the first segment was about 200 g m^{-3} h^{-1}, which is much lower than that of biofilter A. The elimination capacities for toluene after the first segment and after the second segment (shown in Fig. 6b) are quite similar. For the two segments, maximum elimination

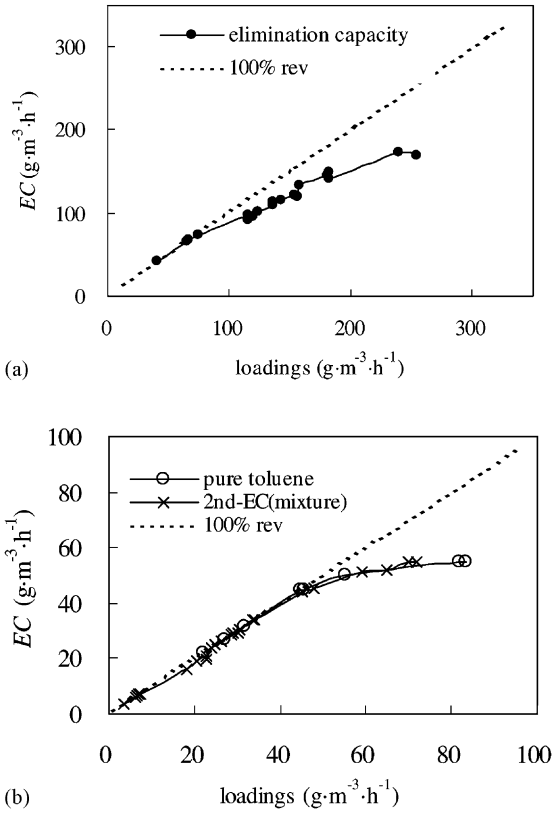

Fig. 7. The EC for ethyl acetate and toluene of biofilter B. Notes: (a) EC for ethyl acetate after the first segment; (b) EC for toluene after the first segment when treating pure toluene and the EC for toluene after the second segment when treating the mixtures.

capacities of toluene were about 50 g m^3 h^{-1}. When the corresponding load was greater than 60 g m^{-3} h^{-1}, the maximum EC for toluene did not increase obviously with the increase of toluene loads. The result is closely in agreement with that of biofilter A.

3.4. The comparison between biofilters A and B

During the 3 month experiment, the maximum EC for toluene by biofilter A is comparable with that of biofilter B. However, the elimination capacities of ethyl acetate by biofilter A are higher than that by biofilter B. These phenomena may be due to the following aspects. Firstly, there may be higher quantities and more kinds of microorganisms existing in biofilter A after the acclimation compared to that in biofilter B (Table 2). Secondly, nutrient in the media of biofilter B may not be sufficient. Many researchers have reported that nutrient addition, especially nitrogen, to VOCs degrading biofilters is necessary [16–18]. However, the effects of nutrient addition on biofilters vary with packing media and pollutants to be treated. It was found in our previous work that, under the same operating conditions, the mass of packing

Table 3
Some examples of the removal ethyl acetate efficiencies and toluene elimination capacities after the first segment of biofilter A at different inlet VOC concentrations

Inlet ethyl acetate (mg/m^3)	Removal ethyl acetate (%)	Inlet toluene (mg/m^3)	EC for toluene ($g\,m^{-3}\,h^{-1}$)
2500	92	218	8
2535	100	205	7
2575	100	210	6
2569	100	207	3
1019	100	396	27
1021	100	400	22
1061	100	399	24

media in the biofilter to treat toluene decreased 50% (dry mass) after a 5 month operation at the load of $45\,g\,m^{-3}\,h^{-1}$, while 26% in the biofilter to treat ethyl acetate at the load of $300\,g\,m^{-3}\,h^{-1}$. The consumption of the media during the acclimation and operation in biofilter B leads to the conclusion that the real VOC loading was higher than that by caculation.

When the biofilters were operated at EBRT of 60 s and at the highest loading (Table 1), the removal efficiencies for ethyl acetate and toluene were nearly 100%. At EBRT of 30 s, some examples of the steady state operation of biofilter A in removing ethyl acetate and toluene are listed in Table 3. From an operating perspective, if the biofilter operated at 30 s EBRT with the inlet ethyl acetate concentration of $2500\,mg/m^3$ (two times of the average concentration at working site) and toluene concentration at $100\,mg/m^3$ (the average concentration at working site), the removal efficiencies for ethyl acetate and toluene are in the ranges of 92–99% and 35–50%, respectively. When the biofilter was operated at an EBRT of 30 s and at mean inlet VOC concentrations (e.g. inlet ethyl acetate concentration $= 1000\,mg/m^3$, inlet toluene concentration $= 100\,mg/m^3$), the removal efficiencies for the two VOCs were around 99%.

3.5. *Variation of media content*

Biofilters have a shortcoming of drop in removal efficiency for a long run if no additional nutrient is provided. This drop is partly due to the depletion of nutrients with respect to microbial growth and metabolism. Nitrogen and phosphorus are essential nutrients for microbial metabolism and activity [16,19,20]. The nitrogen which microorganisms are able to utilize is present in inorganic forms (ammonia and nitrate). The concentration of ammonia was relatively uniform throughout the media of a biofilter, and the release of ammonia by the biomass in compost-based biofilters results in the recycle of nitrogen [16]. Ammonia was determined in order to observe the conversion and transition of inorganic nitrogen. Ammonia can exist in ether a neutral (NH_3) or a protonated (NH_4^+) form in the media, depending on the pH. At the pH associated with this experiment (5.50–6.71), most of the ammonia in the media exists as NH_4^+. At the same time, TOM, TP and TN were monitored to investigate the consumption of these substances during the experiment. The change in the media conditions of the third segment was not caused by the degradation of ethyl acetate or toluene, but mainly by the leakage from the above two segments and by the activity of the microorganism itself in the media. Therefore, changes of nutrients and pH in the third segments were not observed here.

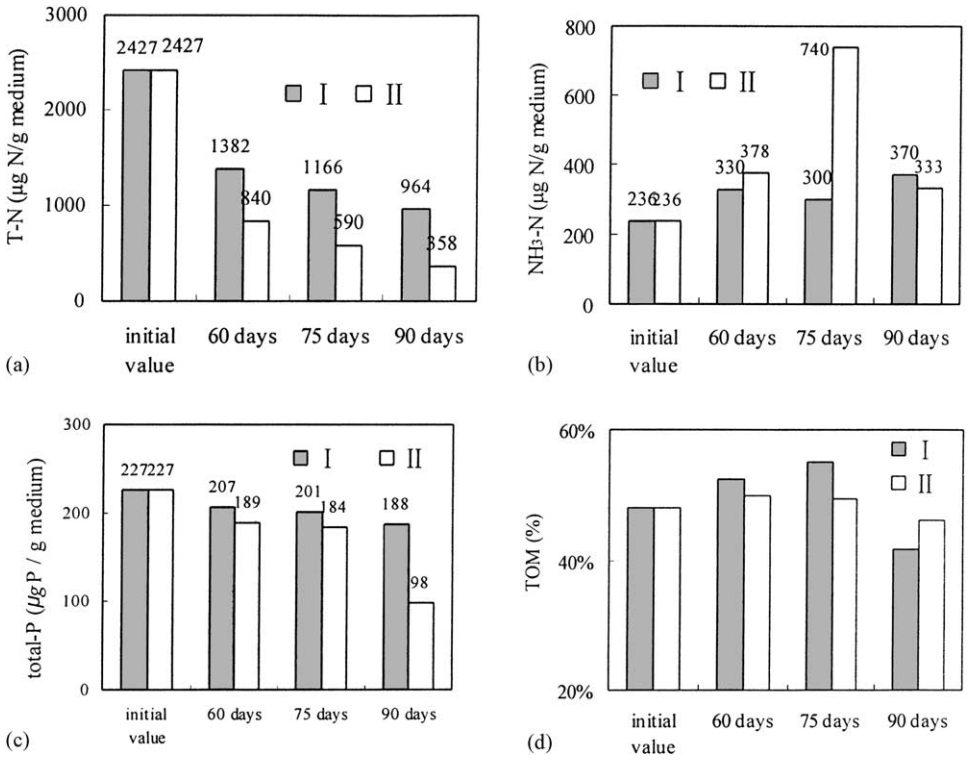

Fig. 8. Change of total nitrogen (a), ammonia nitrogen (NH_3–N) (b), total phosphorus (c) and total organic matter content (d) in media after operating time (I and II refer to the media at the top of the first and second segment, respectively).

As shown in Fig. 8, TN (Fig. 8a) in each segment decreased. Furthermore, more TN was consumed in the second segment, which mainly treated toluene, than in the first segment, which mainly treated ethyl acetate. Ammonia is mainly transferred from the organic nitrogen by the process of mineralization of cells in the media and ammonia may be converted to nitrite [16]. This transformation and conversion may contribute to the irregular distribution of NH_3–N content throughout the biofilters (shown in Fig. 8b). Similar to the case of TN, TP was also essential to the growth of microorganisms, but its decrease was not as obvious as TN (Fig. 8c). The content of TOM remained nearly constant in the experiment (Fig. 8d). This indicates that the content of TOM could be balanced via mass transfer among pollutants, biofilm and media.

3.6. Change of pH

The pH change is shown in Table 4. At the top of the first segment treating ethyl acetate mainly, pH decreased slightly. However, pH of the media at the top of the second segment increased gradually throughout the experiment despite the leakage from the first segment.

Table 4
The pH of the packing media at the top of the first and the second segment after different operation times

Time (days)	pH (the first segment)	pH (the second segment)
0	6.71	6.71
60	5.81	6.71
75	5.72	6.81
90	5.50	7.08

This may be attributed to the production of acidic byproduct (such as acetic acid) in the biofiltration of ethyl acetate and alkali byproduct in the biofiltration of toluene. The acidic liquid leaked from the first segment could not balance the alkali byproducts in the second stage.

4. Conclusions

Ethyl acetate and toluene were successfully treated in biofilters. The biofilter removed as high as 450 g ethyl acetate/m^3 bed medium/h with more than 90% removal efficiency at an EBRT of 30 s. For toluene, the biofilter achieved a maximum removal capacity of 50 g m^{-3} h^{-1} at 30 s EBRT. In the biofiltration of mixtures of ethyl acetate and toluene, the presence of ethyl acetate in the system significantly reduced its removal capacity of toluene. However, the removal efficiency of ethyl acetate was not affected by the presence of toluene in air streams.

The biofilter acclimated with ethyl acetate had a higher EC for ethyl acetate than the biofilter acclimated with toluene.

In the treatment of ethyl acetate and toluene, the content of TN and TP decreased while the TOM content remained nearly constant. Ammonia was transferred and converted throughout the biofiltration.

The pH of the media decreased slightly in the top of the first segment treating ethyl acetate mainly, but increased slightly in the second segment treating toluene mainly.

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