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ORIGINAL PAPER

Biofiltration of paint solvent mixtures in two reactor types: overloading by hydrophobic components

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Abstract Steady-state performance characteristics of a trickle bed reactor (TBR) and a biofilter (BF) in loading experiments with increasing toluene/xylenes inlet concentrations while maintaining a constant loading rate of hydrophilic components (methyl ethyl and methyl isobutyl ketones, acetone, and *n*-butyl acetate) of 4 g m^{-3} h^{-1} were evaluated and compared, along with the systems' dynamic responses. At the same combined substrate loading of 55 g m^{-3} h^{-1} for both reactors, the TBR achieved more than 1.5 times higher overall removal efficiency (RE_W) than the BF. Increasing the loading rate of aromatics resulted in a gradual decrease of their REs. The degradation rates of acetone and *n*-butyl acetate were also inhibited at higher loads of aromatics, thus revealing a competition in cell catabolism. A step-drop in loading of aromatics resulted in an immediate increase of RE_W with variations in the TBR, while the new steady-state value in the BF took 6–7 h to achieve. The TBR consistently showed a greater performance than BF in removing toluene and xylenes. Increasing the loading rate of aromatics resulted in a gradual decrease of their REs. The degradation rates of acetone and *n*-butyl acetate were also lower at higher OL_{AROM}, revealing a competition in the cell catabolism. The results obtained are consistent with the proposed hypothesis of greater toxic effects under low water content, i.e., in the biofilter, caused by aromatic hydrocarbons in the presence of polar ketones and esters, which may improve the hydrocarbon partitioning into the aqueous phase.

Keywords Bacteria · Organic air pollutants · Biofilm · Trickle bed bioreactor · Biofilter

Introduction

Paint solvents are mixtures of VOCs containing both hydrophobic and polar/hydrophilic components. Their removal from waste air, e.g., from paint booths, has been attempted using both biotrickling reactors [1–6] and biofilters [7–11]. The greatest challenge of paint solvent biofiltration involves the removal of complex mixtures of pollutants, i.e., a multi-parameter system with inherently complex interactions. This challenge is enhanced by variation of the paint solvent vapor composition, e.g., due to changes in ambient temperature, resulting in frequent system overloads.

The rate-limiting step for biofiltration, for most of the typical, i.e., readily biodegradable, substrates is mass transfer (through the aqueous layer surrounding the biofilm) rather than biological processes [12–14]. This feature of biofiltration offers an advantage such that biofilters may

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be the reactors of choice, with their inherently low water content. However, the reactor selection is also based on other considerations involving the reactor configuration; even when the rate-limiting step does not involve biochemical reactions, accumulation of toxic chemicals in the aqueous medium often necessitates the use of trickling reactors [15]. Biotrickling reactors have a substantial advantage over biofilters in controlling the medium pH [16, 17]. This feature is particularly important for the removal of chemicals producing acidic metabolites, e.g., esters. However, pH control is critical for the removal of any substrate when its mass loading is high [18]. Another advantage of biotrickling reactors over biofilters is biomass control by a periodical removal of excess biomass from the liquid medium [15–18].

The objectives of this study were to evaluate the simultaneous degradation of the individual hydrophobic and polar/hydrophilic components in a paint solvent mixture and to test the effect of reactor overloading by aromatic substrates, with a constant loading rate of polar components, under both steady-state and dynamic conditions. When overloaded with hydrophobic substrates, higher aromatics' removal rates in a biotrickling reactor were observed compared to a biofilter, which was run under similar operational conditions. This novel and unexpected effect was explained by higher toxicity of aromatic hydrocarbons in the presence of polar substrates, which could increase the solubility of hydrophobic chemicals in water. Such an enhancement of toxicity due to improved partitioning into the aqueous medium surrounding the biofilm would be more pronounced in biofilters.

Materials and methods

Reactors

A schematic diagram of the bench-scale biotrickling filter is shown in Fig. 1. The height of the reactor was 1.70 m and the internal diameter was 0.15 m. The sump was separated from the column by a perforated plate. The packing material consisted of Pall rings made of hydrophilized polypropylene. The parameters of Pall rings were as follows: 15 × 15 × 1 mm, void volume of 0.862, specific surface area of 350 m^2 m^{-3} and a bulk density of 120 kg m^{-3}. The packed bed height was 1.0 m.

A schematic diagram of the biofilter system is shown in Fig. 2. The biofilter was made of glass with an internal diameter of 100 mm and a bed height of 1.0 m. The packing material was Poraver (a crushed recycled glass product) with a grain size of 8–16 mm and porosity of 0.44.

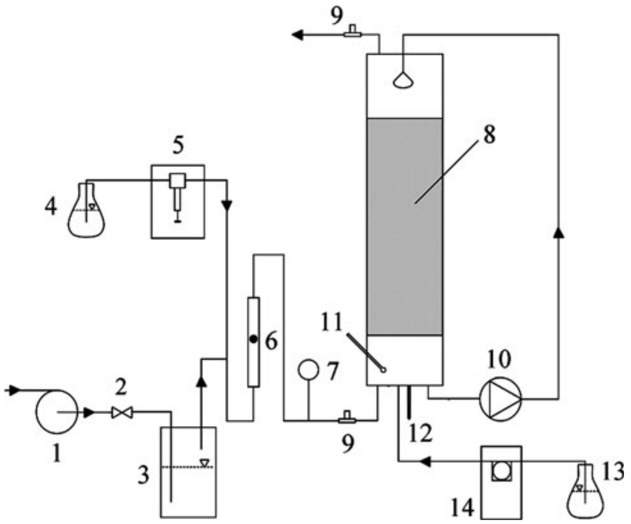

Fig. 1 Schematic of the trickle bed reactor setup: *1* blower, *2* needle valve for air flow rate control, *3* humidifier, *4* vessel with pollutant, *5* syringe pump, *6* flow meter, *7* manometer, *8* packing, *9* sampling ports, *10* membrane pump, *11* pH electrode, *12* thermometer, *13* NaOH solution, *14* peristaltic pump

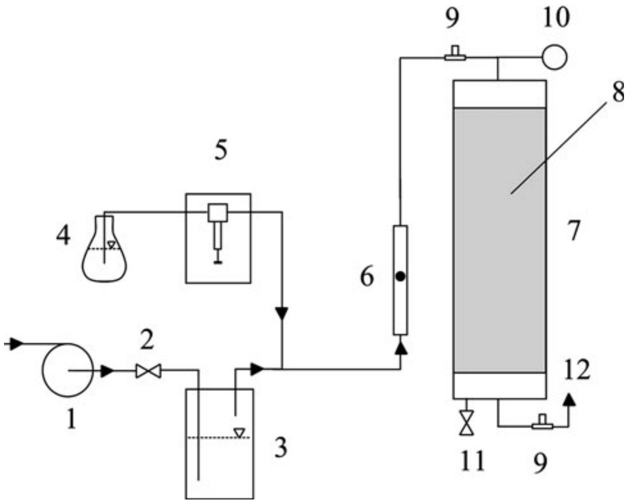

Fig. 2 Schematic of the biofilter set-up: *1* blower, *2* needle valve for flow rate control, *3* humidification vessel, *4* vessel with pollutant, *5* syringe pump, *6* flow meter, *7* biofilter, *8* packing, *9* sampling ports, *10* manometer, *11* valve for leachate, *12* gas outlet

Microorganism and inoculum preparation

The mixed microbial culture used to inoculate both the reactors contained the following bacterial strains: *Sphingobacterium multivorum* (G^- rods), *Comamonas testosteroni* (G^- rods), *Pseudomonas putida* (G^- rods) and *Bacillus cereus* (G^+ rods). All of these bacterial strains were primary toluene and xylenes degraders (i.e., each strain was able to use the individual pollutants as the sole carbon and energy sources for growth). The inoculation suspension was prepared by three-stage submerged cultivations with toluene vapor as the sole carbon and energy source. The medium composition for cultivation was (g l^{-1}): K_2HPO_4, 4.3; KH_2PO_4, 3.4; KNO_3, 2; $MgCl_2 \times 6$ H_2O, 0.34; $CaCl_2$, 1.3×10^{-3}; $FeSO_4$, 0.6×10^{-4}; Na_2MoO_4, 1.7×10^{-3}.

Biofiltration experiments were conducted using a mineral medium containing (g l^{-1}) $(NH_4)_2SO_4$, 0.4; KNO_3, 0.3; NaCl, 0.1; K_2HPO_4, 0.125; KH_2PO_4, 0.085; $MgCl_2 \times 6H_2O$, 0.34; $MnCl_2$, 7×10^{-4}; $CaCl_2$, 1.3×10^{-3}; $FeSO_4$, 6×10^{-5}; Na_2MoO_4, 1.7×10^{-3}. The content of phosphates and nitrate was lower than that for the inoculum preparation to suppress an excessive biomass growth. The pH of the circulating aqueous phase was maintained at 7.0 using a pH–stat; the hydraulic loading rate of circulation was 2.242 m h^{-1}.

Chemical properties of selected solvents

Toluene (TOL), methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK), *n*-butyl acetate (*n*-BA), and acetone (Ac) were of analytical grade. Xylenes (XYL) was a technical-grade mixture containing all three isomers (o-, m-, and p-) in similar amounts. Table 1 shows the pertinent physico-chemical properties of the individual components of a paint solvent mixture.

Chromatographic analyses

The pollutants in the gas phase were determined using an Agilent 6890 N gas chromatograph equipped with an ultra alloy-5 (5% phenylmethylsilicone) capillary column of 30 m in length, with an inner diameter of 0.53 mm and film thickness of 1.5 μm (Quadrex Corp., UA5—30 V—1.5 F, New Haven, CT). The carrier gas was ultra high purity argon at a flow rate of 5.0 ml min^{-1}. The detection was carried out with a flame ionization detector (FID) with ultra high purity hydrogen and breathing quality air at flow rates of 28 ml min^{-1} and 315 ml min^{-1}, respectively. Operating conditions included a column temperature of 80°C and an FID temperature of 250°C.

Loading procedure

The loading of both reactors commenced while keeping the TOL/XYL ratio constant, at 1:1 (w/w). The inlet concentration of each aromatic substrate was varied within a range of 40–675 mg m^{-3}. The inlet concentrations of the polar components were kept constant (mg m^{-3}): MEK, 19; MIBK, 15; *n*-BA, 26; and Ac, 17. The empty bed retention time (EBRT) in both reactors was maintained at 43 s; the operation temperature was 22 ± 1°C. Thus, the combined loading rate of polar substrates, ketones and esters (OL_{POLAR}) was constant, at ca. 4 g m^{-3} h^{-1}.

Calculations

The following parameters were used to evaluate the system performance [12B]: Elimination capacity

$$EC = (C_{in} - C_{out}) \cdot \frac{Q}{V_b} (g\,m^{-3} h^{-1}) \tag{1}$$

Organic load

$$OL = C_{in} \cdot \frac{Q}{V_b} (g\,m^{-3} h^{-1}) \tag{2}$$

Removal efficiency

$$RE = \frac{EC}{OL} \cdot 100 = \frac{C_{in} - C_{out}}{C_{in}} .100 (\%) \tag{3}$$

Empty bed residence time

$$EBRT = 3.6 \cdot 10^3 \cdot \frac{V_b}{Q} (s) \tag{4}$$

where C_{in} and C_{out} are the inlet and outlet pollutant concentration (mg m^{-3}), respectively, Q is the air flow rate (m^3 h^{-1}), and V_b is the filter bed volume (m^3). The subscripts for OL, EC, and RE, W, AROM, and POLAR, refer to

Table 1 Chemical properties of selected solvents

Chemical	Formula	Molecular weight (g mol^{-1})	Water solubility (g l^{-1})[a]	Boiling point (°C)[a]	Log K^{b}_{ow}
Methyl ethyl ketone (MEK)	C_4H_8O	72	275	80	0.29
Methyl isobutyl ketone (MIBK)	$C_6H_{12}O$	100	20	117	1.4[c]
n-Butyl acetate (*n*-BA)	$C_6H_{12}O_6$	116	7	126	1.82
Acetone (Ac)	C_3H_6O	58	Unlimited	56	−0.24
Toluene (TOL)	C_7H_8	92	0.52	111	2.73
Xylenes (XYL)	C_8H_{10}	106	0.175	140	3.12–3.2

[a] Material safety data sheets, www.msds.com

[b] Spafiu et al. [27]

[c] Extrapolated from the value provided for methyl *n*-butyl ketone [27]

the whole mixture and its combined aromatic and polar components, respectively.

Results

Loading characteristics of the solvent mixture

Figure 3 shows the time courses of OL and RE of combined hydrophobic (aromatics, TOL + XYL) and polar/hydrophilic (ketones/esters) pollutants in the TBR. At day 59 (labeled as an A arrow in Fig. 3), extensive biomass backwashing was carried out to cause a drop of RE_{AROM}. The recovery of RE_{AROM} took ca. 3 weeks whereas the recovery of RE_{POLAR} took only 5 days. Following this treatment, the measurements were recorded at quasi steady state. A gradual increase of OL_{AROM} under a constant OL_{POLAR} of ca. 4 g m^{-3} h^{-1} caused a gradual drop of RE_{AROM} while RE_{POLAR} remained >98% as long as OL_{AROM} <36 g m^{-3} h^{-1}. Once this loading rate was exceeded (on day 118), the degradation rate of hydrophobic compounds began to decline as well.

Figure 4 shows the time courses in the BF for the same parameters as in Fig. 3. The recorded value of RE_{AROM} was lower than that in TBR throughout the entire period of operation. The other features of the BF performance were similar to those of TBR. The removal rate of aromatics continuously dropped with increasing OL_{AROM} while RE_{POLAR} remained constant. Upon exceeding an OL_{AROM} of ca. 52 g m^{-3} h^{-1}, the RE_{POLAR} also started to decline. These features are consistent with the expectations that polar components would be preferentially biodegraded than more hydrophobic aromatic hydrocarbons.

Impact of OL_{AROM} on the REs of individual components

The analysis of both reactors' performance was extended to the individual components. Figure 5 shows a comparison of the RE changes of toluene and xylenes with increasing loading rates (cf. Figs. 3 and 4). Toluene was degraded more efficiently than xylenes in both reactor systems. While comparing the reactors at the same loading rate (Table 2), the RE_{AROM} were consistently greater in the TBR whereas the RE_{POLAR} was slightly higher in the BF.

In TBR up to $OL_W = 55$ g m^{-3} h^{-1}, the RE of MEK, MIBK, and *n*-BA, i.e., of all the partially water-soluble polar compounds, were above 95%. Similar high values of RE_{POLAR} were also observed in the BF, up to $OL_W = 65$ g m^{-3} h^{-1}. Thus, even though the cells were overloaded with toluene and xylenes, the cell catabolism of polar compounds was not influenced within a wide range of substrate concentrations. Only with OL_{AROM} >80 g m^{-3} h^{-1}, some inhibitory effect of *n*-BA degradation in TBR started to show up (Fig. 6a).

The only exception was acetone, the most water-soluble component of the paint solvent mixture. The RE of acetone,

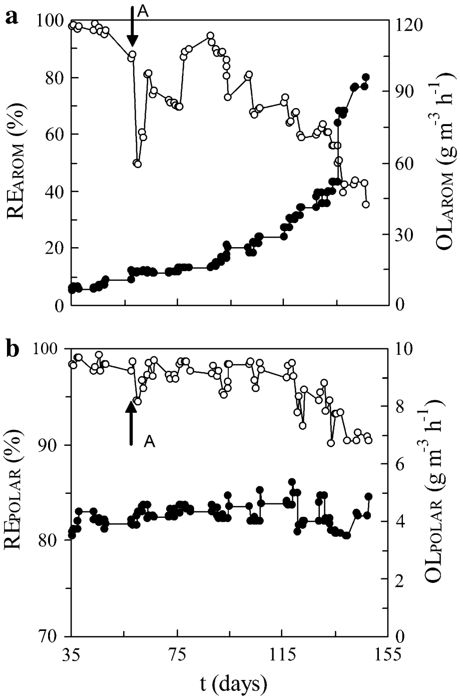

Fig. 3 Loading rates (*closed circles*) and their impact on RE (*open circles*) of hydrophobic (**a**) and hydrophilic (**b**) components in TBR

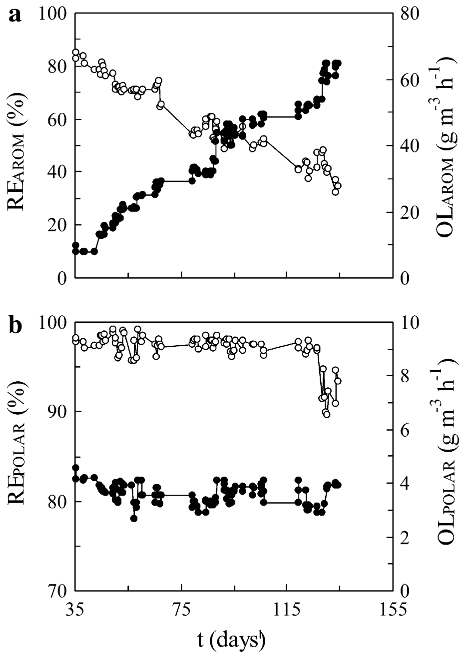

Fig. 4 Loading rates (*closed circles*) and their impact on RE (*open circles*) of hydrophobic (**a**) and hydrophilic (**b**) components in BF

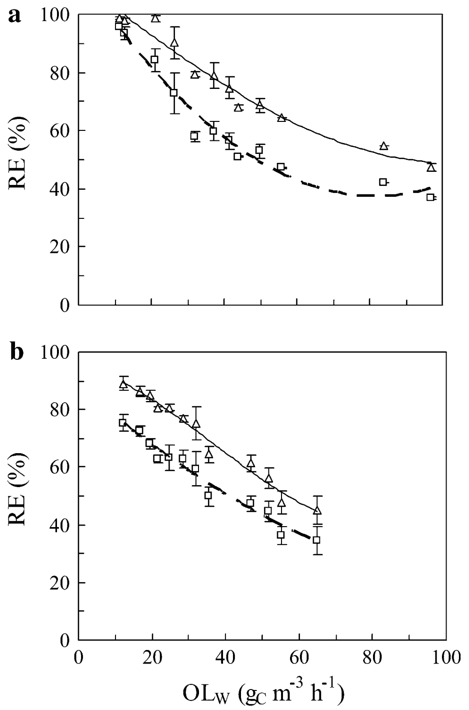

Fig. 5 The removal efficiencies (RE) of TOL (*open triangle*), and XYL (*open square*) at varied overall organic loading rates (OL$_W$) in TBR (**a**), BF (**b**)

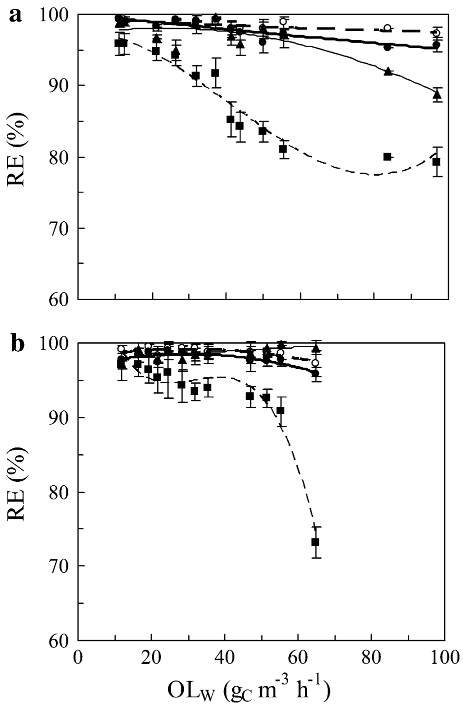

Fig. 6 The removal efficiencies (RE) of MEK (*open circle*), MIBK (*closed circle*), *n*-BA (*closed triangle*), Ac (*closed square*) at varied overall organic loading rates (OL$_W$) in TBR (**a**), BF (**b**)

RE$_{AC}$, started to drop below 95% at OL$_{AROM}$ >20 g m^{-3} h^{-1} in both reactors. While the drop of RE$_{AC}$ in BF was not pronounced up to an OL$_{AROM}$ of 50 g m^{-3} h^{-1} (remaining >90%), a further OL$_{AROM}$ increase resulted in a significant inhibitory effect on the acetone degradation (Fig. 6b). The TBR response to increased OL$_{AROM}$ was qualitatively similar, although, unlike in the BF, no clear threshold of OL$_{AROM}$ triggering a pronounced drop in RE$_{AC}$ was observed (Fig. 6a).

Overall performance characteristics

Figure 7 shows a comparison of the overall performance parameters of both reactor systems. The TBR was operated with RE$_W$ $\geq$ 90% up to OL$_W$ $\sim$ 25 g m^{-3} h^{-1} and the BF up to OL$_W$ $\sim$ 15 g m^{-3} h^{-1}. While treating the solvent mixture, the BF, unlike TBR, never yielded an RE$_W$ above 90%. Upon increasing the OL$_W$ above this level, the RE$_W$ continuously dropped in both reactors. However, the TBR still exhibited significantly higher total degradation rates and efficiencies. The maximum EC$_W$ values achieved were 43 and 28 g m^{-3} h^{-1} in the TBR and BF, respectively.

Dynamic response to a step-drop of the loading rate

Figure 8 shows the dynamic response of both reactor systems to a step-drop in the loading rate from higher, overloading values back to the non-overloading value applied prior to the loading tests, i.e., before day 35 in Fig. 3. Since the loading rate of polar components remained the same throughout the entire duration of experiments, (OL$_{POLAR}$ $\sim$ 4 g m^{-3} h^{-1}), it was OL$_{AROM}$ that was lowered by an order of magnitude, from 100 to 7 g m^{-3} h^{-1}. As a result, the RE$_{POLAR}$ increased to 98% in both reactors. However, prior to reaching the new steady state, the RE$_{POLAR}$ in the TBR exhibited damping oscillations for 4 h, whereas in the BF the response was gradual and took 6 h. As seen in Fig. 8 as compared to Fig. 3, the final overall RE$_W$ values were ca. 15 and 10% lower than those observed prior to the loading test in TBR and BF, respectively.

Table 2 Comparison of performance parameters achieved at identical loading rate

	OL$_W$ (g m^{-3} h^{-1})	RE$_{AROM}$ (%)	RE$_{TOL}$	RE$_{XYL}$	RE$_{POLAR}$	RE$_W$	EC$_W$ (g m^{-3} h^{-1})
TBR	55	56 ± 1	65 ± 1	47 ± 2	93 ± 1	58 ± 1	32 ± 2
BF		42 ± 3	48 ± 4	36 ± 3	97 ± 2	45 ± 3	25 ± 2

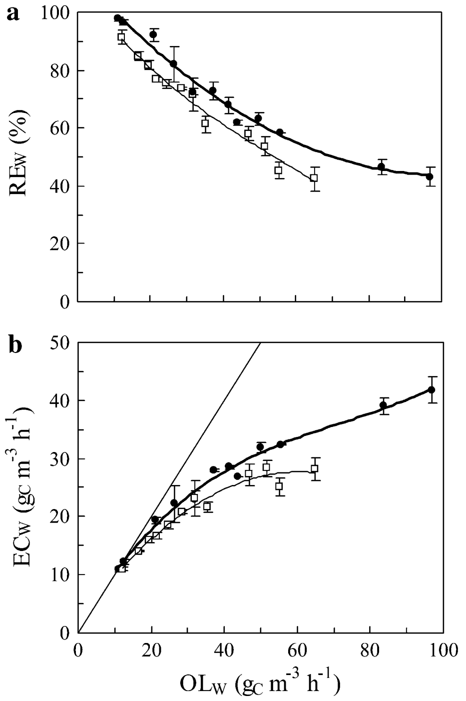

Fig. 7 Comparison of the overall performance characteristics in TBR (*closed circle*) and BF (*closed square*): **a** overall removal efficiency (RE_W) and **b** overall elimination capacity (EC_W)

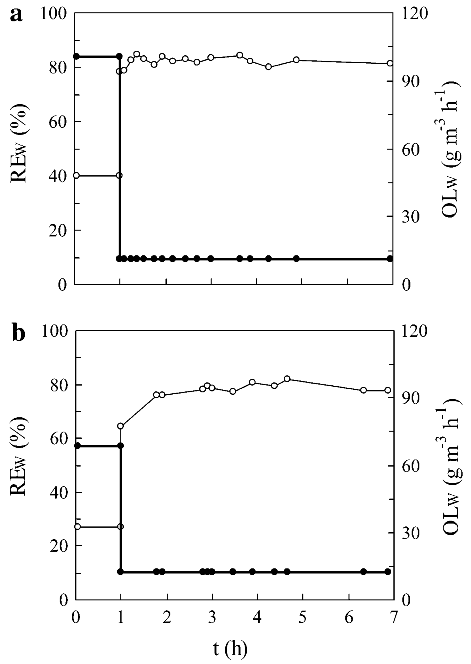

Fig. 8 The overall dynamic response of BTF (**a**) and BF (**b**) *closed circle*—OL_W; *open circle*—RE_W

As for the individual components, toluene and xylene RE profiles showed similar oscillating characteristics in two reactors (not shown). The new steady-state values were achieved in 5 and 7 h in TBR and BF, respectively.

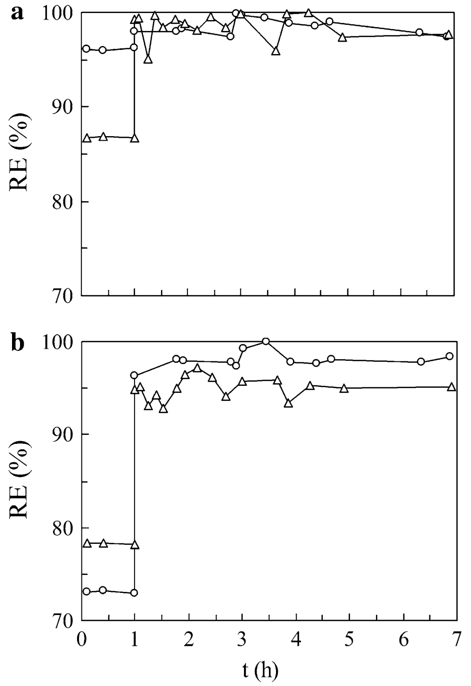

Fig. 9 The dynamic responses for *n*-BA (**a**) and acetone (**b**) *open triangle*—TBR, *open circle*—BF

Figure 9 shows the dynamic responses to a step-drop of OL_{AROM} for two polar components, *n*-BA and Ac, from high OL_{AROM}. A fast return of RE, within 1 h, to the original values obtained prior to the loading tests was observed. However, some minor oscillations continued to be observed for 4 h, especially in the TBR.

Discussion

The comparison of the reactors' pollutant removal efficiencies is provided in Table 2; the data shown were taken at the same loading rate. One pronounced feature of the system studied was that toluene exhibited a higher RE than xylenes in both reactors. This effect was consistent within a large range of OL_{AROM} (Fig. 5). Since mass transfer is presumed to be the process' rate-limiting step, toluene, as a more water-soluble substrate than xylenes (Table 1) was expected to be removed with a higher efficiency.

However, the second general observation, that the TBR consistently exhibited a higher removal rate than BF for aromatics, particularly toluene (Fig. 5; Table 2), was not expected. This observation was also valid for the overall reactor performance as shown in Fig. 7. The TBR consistently showed higher overall degradation rates and efficiencies, at both low and high OL_W, the difference becoming more pronounced at higher loads (Fig. 7b).

This specific feature of the system studied was unusual because the mass transfer conditions affecting the presumed rate-limiting step should be superior in biofilters, compared to TBRs, due to inherently lower amounts of the aqueous medium. Thus, a consistently *higher* RE_{AROM} in TBR demands a different explanation. Lower TBR propensity to the inhibition of cell catabolic activity by acid and by-product accumulation may not adequately explain this effect because, first, the pH was thoroughly controlled and, second, no organic intermediates of substrate oxidation were detected in the mineral medium (not shown).

A higher TBR efficiency for the removal of aromatics may still be explained by lower accumulation of toxic chemicals, i.e., a well-known specific feature of TBRs. However, these toxic chemicals may not only be the products of their metabolism but also the hydrocarbon substrates themselves [19–21]. The occurrence of this unusual toxicity effect, primarily caused by the aromatic hydrocarbon substrates, may be due to the presence of relatively high amounts of water-soluble polar organic solvents leading to an increase of the solubility of hydrophobic aromatic hydrocarbons in water, which is otherwise low (Table 1). The increased hydrocarbons' partitioning into the aqueous phase and, thus, their higher availability for microbial cells, would enhance their toxic effects.

Competitive effects observed between the aromatics and acetone may be explained by catabolite competition. Similar preferential degradation of ketones (acetone and MEK) versus aromatic hydrocarbons have been reported [9, 11, 22]. The inhibitory effect of toluene on acetone removal has also been observed [5]. Chang and Lai [23] found that the biodegradation of acetone was inhibited by MEK.

However, the proposed higher toxicity of aromatics at higher loads might also play a role because these effects were more pronounced in the BF, while *n*-BA degradation was inhibited by high OL_{AROM} only in the TBR. The observed two-phase response of the acetone removal rate to intermediate and then high OL_{AROM} values (Fig. 6b) indicates that both effects, those of substrate competition and enhanced hydrocarbon toxicity, could be operative under different substrate loadings.

It is noteworthy that in the BF (but not in TBR) *n*-BA was degraded with a higher RE than those of MEK and MIBK (Fig. 6a, b). A similar microbial preference for esters, e.g., *n*-BA, was observed earlier by Mathur and Majumder [24]. The other minor differences between the TBR and BF performance may be caused by a different microbial composition of the biofilms developed upon a long-term adaptation in the two reactors.

The dynamic tests showed faster response to the OL sudden drops in the TBR than in BF (Figs. 8 and 9). This effect is consistent with our hypothesis of the aromatics' enhanced toxicity in the presence of esters and ketones. Perhaps toxic hydrocarbons partitioned into the aqueous medium can be diluted by large amounts of water in the TBR.

Note that after a return to the original OL_W value, the original RE_W value was not attained, particularly in the TBR. A similar impact of hydrocarbons was reported by Bastos et al. [4] after 16 days of loading, and by Moe and Qi [8] after an even shorter period of loading. An inhibitory effect was also observed in our previous study [25] while using a similar solvent mixture (only without acetone). Ryu et al. [26] offered an explanation of such a drop in biodegradation rates during long-term bioreactor operation, ascribing it to a significant increase of the pressure drop. However, this effect may also be explained by a long-lasting inhibitory effect (more than 2 months) by hydrocarbons, thus being consistent with the hypothesis proposed in this study.

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