



Effects of anionic surfactant on n-hexane removal in biofilters

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HIGHLIGHTS

- The biodegradability of 3 surfactants by biofilm microorganisms was evaluated.
- SDS could be biodegraded by and was not toxic to biofilm microorganisms.
- The optimal SDS concentration for enhanced n-hexane removal in biofilters was 0.1 CMC.

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ABSTRACT

The biodegradability of three anion surfactants by biofilm microorganisms and the toxicity of the most readily biodegradable surfactant to biofilm microorganisms were examined using batch experiments, and the optimal concentration of SDS for enhanced removal of hexane was investigated using two biotrickling filters (BTFs) for comparison. Results showed that SDS could be biodegraded by microorganisms, and its toxicity to microorganisms within the experimental range was negligible. The best concentration of SDS in biofiltration of n-hexane was 0.1 CMC and the elimination capacity (EC) of 50.4 g m⁻³ h⁻¹ was achieved at a fixed loading rate (LR) of 72 g m⁻³ h⁻¹. When an inlet concentration of n-hexane increased from 600 to 850 mg m⁻³, the removal efficiency (RE) decreased from 67% to 41% by BTF2 (with SDS) and from 52% to 42% by BTF1 (without SDS). SDS could enhance hexane removal from 43% (BTF1) to 60% (BTF2) at gas empty-bed residence time (EBRT) of 7.5 s and an inlet concentration of 200 mg m⁻³.

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1. Introduction

Air pollution has recently been a main concern and become a crucial issue due to an increase of public consciousness about emissions of volatile organic compounds (VOCs). Governments and environmental agencies strictly regulate these VOC emissions (Muñoz et al., 2007). The development of viable and effective VOC emission control strategies has become a necessity. The most preferable option to control VOC emissions is an environment-friendly technology. Biofiltration systems designed and operated properly are considered a cost-effective and promising technique

for VOC and odorous gases control. Additionally, comparing with conventional VOC control technologies, biofiltration systems are more suitable for VOC removal (Sorial et al., 1997; Cox and Deshusses, 2002; Dixit et al., 2012; Xue et al., 2013). The biofiltration process is based on the ability of microorganisms to convert VOCs into carbon dioxide, water and biomass (Devinny et al., 1999).

However, biological process performs poorly when treating hydrophobic VOCs, because the low solubility and transfer rates of hydrophobic VOCs from gas phase to biofilm phase inhibit microbial activity (Yang et al., 2010). As a result, low removal performance of hydrophobic VOCs such as n-hexane and styrene has been recorded in biofiltration systems (Lebrero et al., 2014; Kim et al., 2005). Therefore, increasing the bioavailability of VOCs in biofilm phase will help to enhance the biodegradability of these compounds (Zehraoui et al., 2012).

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To enhance the bioavailability of hydrophobic VOCs, one method is that surfactants are applied in biofilters. It is attributed to that the addition of surfactants reduce the surface tension and form micelles and thus improving the solubility of hydrophobic VOCs in liquid phase (Böttger et al., 2012). In this regard, surfactants have been extensively studied in contaminated soils and sediments (Mulligan et al., 2001). Moreover, most studies have reported that surfactants have a crucial effect on gas–liquid mass transfer of VOCs (Anderson, 1992; Cheng et al., 2009; Yang et al., 2010; Galindo et al., 2011). Zhou and Zhu (2007) and Li et al. (2011) reported that surfactants increased the solubility of hydrophobic organic compounds, leading to an enhancement of the biodegradation rate of these compounds in contaminated environments. In biofiltration systems, chemical surfactants have been introduced and researched as means for enhancing solubility of VOCs in water in recent years (Chan and You, 2009, 2010). Moreover, nonionic surfactants are used and studied more widely in biofilters (Wang et al., 2014; Tu et al., 2015). However, utilization of anionic surfactants such as sodium dodecyl sulfate (SDS) in biofiltration systems is rarely reported in literature. Zeng et al. (2007) reported that SDS could not be poisonous to microorganisms and could also be biodegradable, which avoided secondary contamination due to its discharge directly with waste solution from the bottom of a biofilter.

n-Hexane is well known for its high hydrophobicity and low bioavailability due to the restriction on mass transfer from gas phase to biofilm phase. Several investigations have reported on the n-hexane biofiltration under different operating parameters (Cheng et al., 2015). Other researchers used other methods to address the bioavailability of n-hexane, like introducing ionic surfactants or biosurfactants (Hassan and Sorial, 2008; Tu et al., 2015), providing favorable conditions for fungi (Spigno et al., 2003; Zehraoui et al., 2013), using two-phase reactors (Lebrero et al., 2014), and utilizing hydrophilic compounds (Zehraoui et al., 2012).

This study was to examine the bioavailability of n-hexane after introducing anionic surfactant into a biofilter and the potential of anionic surfactant for enhancing the degradation of n-hexane from contaminated air streams. In this work, batch experiments were conducted to evaluate the biodegradability of SDS, Tween 20 and Triton X-100 and the toxicity of SDS on microorganisms. The optimal concentration of SDS which effected n-hexane removal performance was investigated. Processes for continuous degradation of n-hexane vapor under different influent concentrations and gas empty bed residence time (EBRT) have been carried out with BTF2 fed with SDS and BTF1 without SDS.

2. Materials and methods

2.1. Chemicals

n-Hexane (C_6H_{14}) with a purity of 99% was selected as the target contaminant to model hydrophobic waste gas. SDS was purchased from Acros Organics, with purity 98%. Triton X-100 and Tween 20 were obtained from Sigma Chemical Company. Their structure and properties are listed in Table 1.

The mineral salt medium used for batch experiments and BTFs was reported by Chen et al. (2012).

Table 1
Critical Micelle Concentration (CMC) of surfactants.

Surfactant	Molecular formula	MW	CMC (mg L ⁻¹)	HLB
Triton X-100	$C_{18}H_{37}O_2$	628	116.4	13.5
Tween 20	$C_{58}H_{113}O_{26}$	1225	60	16.7
SDS	$C_{12}H_{25}SO_4Na$	288	1580	40.0

2.2. Experimental setup and operation

2.2.1. Batch reactor

After the successful start-up of the biofilter, the removed biofilms from the medium bed second from the top were carried out for the batch experiments. The removed biofilm was put into a 250 mL glass flask with a 100 mL of the nutrient solution sterilized and shaking well. The cell concentration of suspension liquid is measured and calculated using protein content (unit: mgprotein L⁻¹). The detailed determination method for the protein concentration is provided by Zhong et al. (2014).

Biodegradability tests of surfactants (SDS, Triton X-100 and Tween 20) were carried out in duplicate in 135 mL glass flasks with a certain volume of the mineral salt medium, surfactants (prepared in water, at different concentrations of 0.1CMC and 1.0 CMC) and 1.0 mL of biofilms at 38 mgprotein L⁻¹. The total volume of the solution was 20 mL. The glass flasks were closed with butyl rubber stopper and tightened screw caps. Subsequently, these flasks were put on a rotary shaker at 150 rpm and 30 °C to incubate. Conditions of control flasks were similar to samples except not supplying with ether SDS, Triton X-100 or Tween 20 solution. A gas chromatograph was used to measure the headspace CO₂ concentrations of flasks every three days by extracting 100 μ L gas samples from these flasks with a 100 μ L gas syringe. If CO₂ content in control flasks without surfactants was lower obviously than that of flasks provided with surfactants, surfactants were considered to be biodegradable (Arriaga et al., 2006; Galindo et al., 2011).

Toxicity tests of the surfactant were conducted and the operating process was described as above. The difference was that easily available sources of carbon and energy (per liter in deionized water): 1.0 g glucose, 0.02 g yeast extract and 0.02 g peptone, respectively were applied in this experiment. However, control flasks were supplied with the nutrient solution lacking SDS. The produced CO₂ in the headspace of flasks were used for evaluating the toxicity of SDS to microorganisms and determined by a gas chromatography as described above. The SDS was considered to be toxic to microorganisms when produced CO₂ in flasks with SDS was apparently lower than that of control flasks.

2.2.2. Biofilter

The two equally BTFs (BTF1 and BTF2) were carried out in parallel in this work. Both biofilters were made of a closed plexiglas column containing an internal diameter of 10 cm and a total height of 78 cm. Four similar cylindrical polyurethane sponge media were packed in both BTFs. The property of packing medium had been reported in our previous work (Wang et al., 2014). Total bed volume of each biofilter was 3.14 L. Packing media before packed in both BTFs were soaked into the activated sludge taken from a wastewater treatment plant as seed source (Cheng et al., 2015) to inoculate BTFs. Nutrient sprayed on the filter bed at 4.5 L d⁻¹ from the top of the biofilter periodically and automatically using the timer to maintain the humidity of packing media. Both BTFs were fed with gas mixtures of n-hexane and the humidified air. The gas flow rates were adjusted by flowmeters. The gas flow was co-current with the nutrient. The schematic of the BTF setup is illustrated in Fig. 1 and had been previously provided (Cheng et al., 2015).

After successful start-up of both BTFs, SDS was added into the nutrient solution for BTF2 while the BTF1 fed without SDS. An average n-hexane inlet concentration of 200 mg m⁻³ and an EBRT of 30 s were set as a reference condition. To obtain an optimal concentration of SDS, experiments were conducted by varying concentration of SDS (0.05 CMC, 0.1CMC, 0.3 CMC and 0.5CMC) at a continuous hexane feeding of 72 mg m⁻³ h⁻¹. Subsequently, effects of n-hexane concentration (600 and 800 mg m⁻³) and EBRT (30, 15, 7.5 s) on BTF performance were also examined in presence of

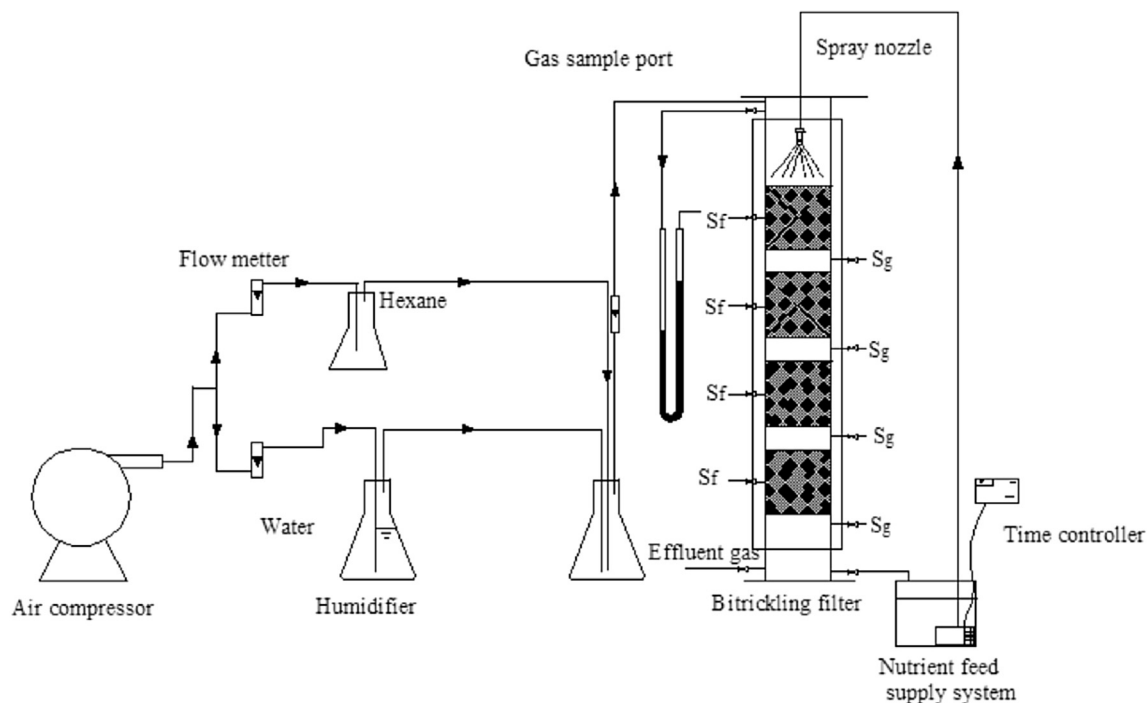


Fig. 1. Schematic diagram of the biotrickling filter (Sf: Biofilm sampling ports; Sg: Gas sampling ports.).

optimal concentration of SDS. In order to ensure pseudo steady state of BTf, both BTf were resumed at reference condition mentioned above for days before and after each operating parameter changed (Song et al., 2012).

2.3. Analytic methods

The measurement of n-hexane concentration in gas samples used a gas chromatography (Agilent 6890), and detailed description had been previously provided by Cheng et al. (2015). The concentration of CO₂ was detected by a gas chromatography (SP7820, Hongtu, China) equipped with a thermal conductivity detector (TCD) and a TDX-01 column (HRBY Inc., China). Detailed description of the analytical method is provided by Liu et al. (2015).

3. Results and discussion

3.1. Biodegradability of surfactants

After the start-up of both BTf, biofilms could be observed on the surface of the packing medium. Biodegradability of Triton X-100, Tween 20 and SDS were evaluated and compared. The results are presented in Fig. 2. It can be seen that the content of CO₂ produced in flasks with SDS was more than that of the controls without SDS. And more importantly the ratio of the CO₂ in flasks with 1.0 CMC of SDS and in control flasks without SDS was much higher than that of 0.1 CMC. It illustrated that SDS was a readily degradable carbon source by the microorganism. From Fig. 2, it can also be seen that the CO₂ had not been produced in the presence of the Triton X-100 concentration of 0.1 CMC or 1.0 CMC, suggesting that microorganisms could not use Triton X-100 as carbon source. In other words, Triton X-100 was difficult to be degraded by microorganisms. A possible reason is that Triton X-100 molecule has hardly degradable parts with aromatic ring and polymeric ethylene oxide structure. From Fig. 2, it was also observed that the CO₂ production in the presence of another nonionic surfactant Tween 20 of 1.0 CMC

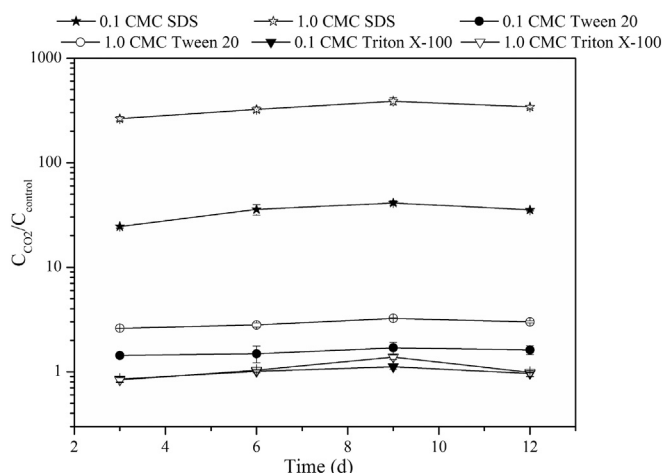


Fig. 2. Biodegradability of surfactants.

was greater than that in the controls, suggesting that Tween 20 could also be utilized by microorganisms at a high concentration. The reason for this may be that the microorganism can ingest the enough carbon and produced more CO₂ in the presence of a high concentration of Tween 20. Similar results had been obtained by Zeng et al. (2007). The authors had proved that Triton X-100 could hardly be degraded by microorganisms while SDS could be biodegraded as carbon source. Woertz and Woertz (2004) found that *E. lecanii-corni* could grow better using Tween 20 as its sole carbon source, which further suggested that Tween 20 could be degraded by the microorganism. The degradability of these compounds is affected by properties of themselves, abilities of microbial degradation and environmental conditions. Surfactant properties (electric property, source and molecular structure) and microorganism activity are key factors. Several investigations suggested that anionic surfactants were generally degradable (Lee et al., 1997;

Scott and Jones, 2000).

The selection of an appropriate and cost effective surfactant is essential to the performance of the bioreactor. The most important characteristic of the surfactant is able to be rapidly removed from the environment to prevent secondary pollution, which will be beneficial for surfactants to be applied more safely and widely. SDS observed from the Fig. 2 has an advantage over other two nonionic surfactants in the biodegradability. Thus, considering this virtue, SDS was chosen to use for the biofiltration of n-hexane in BTFs.

3.2. SDS toxicity

Toxicity experiment of SDS was conducted together with biodegradability tests. Fig. 3 illustrates the toxicity of different SDS concentrations on microorganisms in the presence of different carbon sources. As shown in Fig. 3, the ratio of the CO_2 in experimental group and control group observed was more than 1 when supplied with peptone and yeast extract in flasks as easily available carbon and energy sources while the ratio of the CO_2 was near 1 when the glucose was provided in flasks, indicating that SDS had no inhibition for the microorganism activity. It also illustrated that SDS had no toxicity to the microorganism. Similar result was reported by the other author (Zeng et al., 2007). These significant variations in Fig. 3 depended on the properties of the carbon sources. The peptone and yeast extract can provide not only easily available carbon sources, but also nitrogen sources and other nutrients for the microorganism compared with the glucose which can only be as carbon source. Moreover, these compounds can be rapidly consumed by the microorganism. Thus more CO_2 can be produced in flasks.

Overall, SDS has no toxicity to microorganisms, which is another important characteristic. SDS was chosen to apply to the follow-up experiment.

3.3. Optimizing of SDS concentrations

Both BTFs were fed with nutrient solution where there was not SDS for startup, and an n-hexane average influent concentration of 200 mg m^{-3} and EBRT of 30 s were set, which resulted in an n-hexane loading rate (LR) of $24 \text{ g m}^{-3} \text{ h}^{-1}$. The performance of BTF2 was evaluated under steady-state conditions by varying SDS concentration in nutrient solution at a constant LR of $72 \text{ g m}^{-3} \text{ h}^{-1}$. The BTF1 as the control group was operated under the similar condition, but was not supplied with SDS in the nutrient solution. The

concentration of both BTFs with respect to n-hexane was measuring every day. Fig. 4 shows performances of BTF1 and BTF2 for treating n-hexane during 84 days. The n-hexane removal efficiency (RE) of both BTFs was lower than 60% during first 5 days. Subsequently, the RE of the two BTFs increased gradually and was over 80% on day 11 and 12, respectively. On day 15, 85% of RE for both BTFs was obtained with a corresponding elimination capacity (EC) of $20.4 \text{ g m}^{-3} \text{ h}^{-1}$ and in the next few days, the n-hexane RE in BTF1 and BTF2 remained stable at about 88% and 89%, respectively. It was considered as the successful start-up of BTFs which lasted 20 d.

The BTF2 was fed with the nutrient solution where there was SDS since day 21. The performance of BTF2 at various SDS concentrations ranging from 0.05 to 0.5 CMC is presented in Fig. 4(b) and the results from Fig. 4 were given in Table 2. The highest RE of 70% and a n-hexane EC of $50.4 \text{ g m}^{-3} \text{ h}^{-1}$ were obtained in the BTF2 at a SDS concentration of 0.1 CMC while the RE and EC in the control group BTF1 were lower than 58% and $41.76 \text{ g m}^{-3} \text{ h}^{-1}$ during the whole period of running, respectively. However, it is worth to note that the removal performance of BTF2 was lower than that of BTF1 when the concentration was higher than 0.1 CMC, indicating that an emulative restrain could possibly occur between the high concentration of SDS and n-hexane because of the biodegradability of SDS. As a result, there was an adverse effect on the biodegradation performance of BTF2 for the treatment of n-hexane (see the Section 3.1). In addition, our previous study on the effect of saponins on biofiltration of n-hexane had found this emulative restrain between

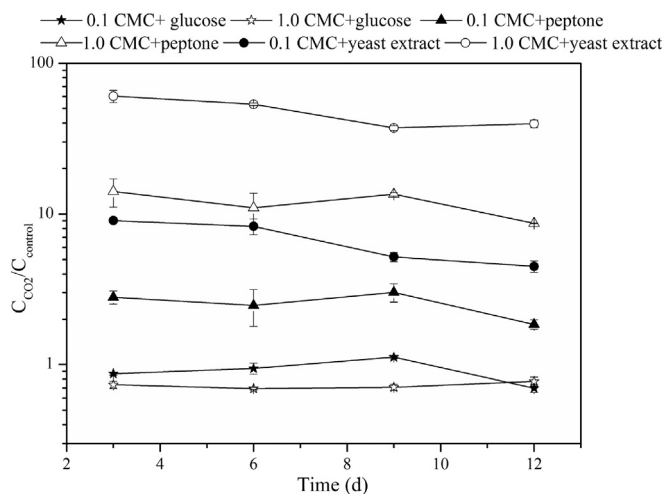


Fig. 3. Effect of SDS on the respiration of microorganisms.

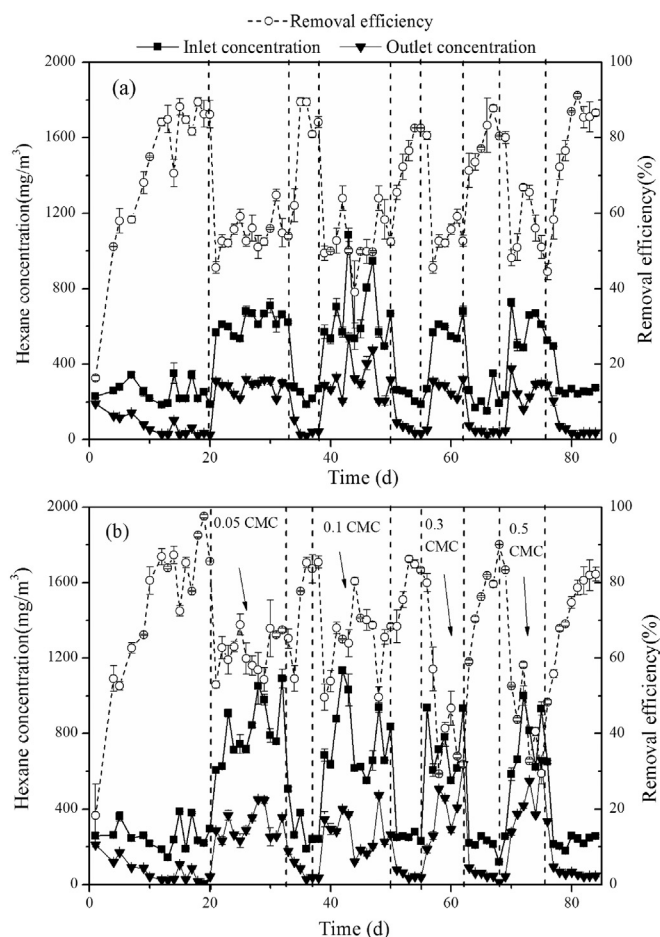


Fig. 4. Effect of SDS concentration on the performance of both BTFs with time for degrading n-hexane. (a) BTF1 without SDS; (b) BTF2 with SDS.

Table 2
Comparison of performance parameters of BTFs achieved.

Concentration of SDS	BTF2 (LR:72 –96 g m ⁻³ h ⁻¹)		BTF1 (LR:72 g m ⁻³ h ⁻¹)	
	RE (%)	EC (g m ⁻³ h ⁻¹)	RE (%)	EC (g m ⁻³ h ⁻¹)
0.05 CMC	64%–67%	46.08–48.24	52%–55%	37.44–39.6
0.1 CMC	68%–70%	48.96–50.4	49%–58%	35.28–41.76
0.3 CMC	32%–46%	27.43–39.44	52%–59%	37.44–42.48
0.5 CMC	32%–48%	22.6–33.91	44%–58%	31.68–41.76

saponins and the target VOC (Tu et al., 2015). However, other authors explained that an increase of surfactant concentration led to an increase in its viscosity of the solution, so mass transport n-hexane and oxygen from the gas phase to the biofilm phase was limited (Galindo et al., 2011), and consequently removal performance of n-hexane in biofilters dropped. The results obtained from Fig. 4 and Table 2 indicated that the higher concentration of SDS in nutrient solution was not always better, but the removal performance of n-hexane in the BTF could be improved by adding SDS at below 0.1 CMC. Hassan and Sorial (2010) achieved n-hexane RE of 59 ± 18% at a LR of 13.40 g m⁻³ h⁻¹ when a surfactant was used in a bifilter.

In this experiment, a maximum RE of 70% and EC of 50.4 g m⁻³ h⁻¹ were obtained at 0.1CMC of SDS under the test conditions. So, 0.1 CMC of SDS was selected in the following experiments.

3.4. Performances of BTFs at optimum SDS concentration

In this stage, removal performances of BTF1 and BTF2 were investigated by varying the n-hexane average inlet concentration from 600 to 850 mg m⁻³ and the EBRT of 30, 15, 7.5 s. Both BTFs restarted up with the same operating condition as the Section 3.3. Recovery periods of both BTFs were performed in the reference condition before the operating condition was changed every time (Chen et al., 2012). The RE of both BTFs reached about 85%, suggesting that BTF1 and BTF2 were operated at a pseudo-steady state during all the recovery experiments.

The inlet and outlet concentrations as well as the RE for both BTFs at each concentration and EBRT are presented in Fig. 5. It can be seen from Fig. 5 that the BTF1 and BTF 2 reached high n-hexane RE of 88% and 89% since day 11, respectively, and both BTFs remained stable in the next few days. So, both BTFs were started up successfully. Fig. 5 also shows that no adverse affects on n-hexane biodegradation were observed in BTF2 which was supplied with SDS, because BTF2 achieved higher RE than BTF1. When average inlet n-hexane concentration was increased from 600 to 850 mg m⁻³ at a constant EBRT of 30 s, the corresponding LR was increased from 72 to 102 g m⁻³ h⁻¹, and n-hexane RE of BTF2 decreased from 67% to 40%, while the RE of BTF1 decreased from 52% to 43%. The enhanced performance for BTF2 at inlet concentration of 600 mg m⁻³ could probably be explained by the fact that the surfactant increased the solubility of pollutants, consequently a reduced mass transfer limitation from the gas phase to biofilms phase was led, thus VOCs were more bioavailable and biodegradable (Wang et al., 2014). The behavior of BTF2 was very similar to data reported by Song et al. (2012) where surfactant Triton X-100 enhanced the removal performance of styrene in BTFs. In addition, surfactants increasing bioavailability and biodegradation of VOCs have already been reported in other literature (Hassan and Sorial, 2008; Tu et al., 2015). However, it is worthwhile to note that n-hexane RE of BTF1 was slightly higher than that of BTF2 at the inlet concentration of 850 mg m⁻³. This phenomenon may be explained

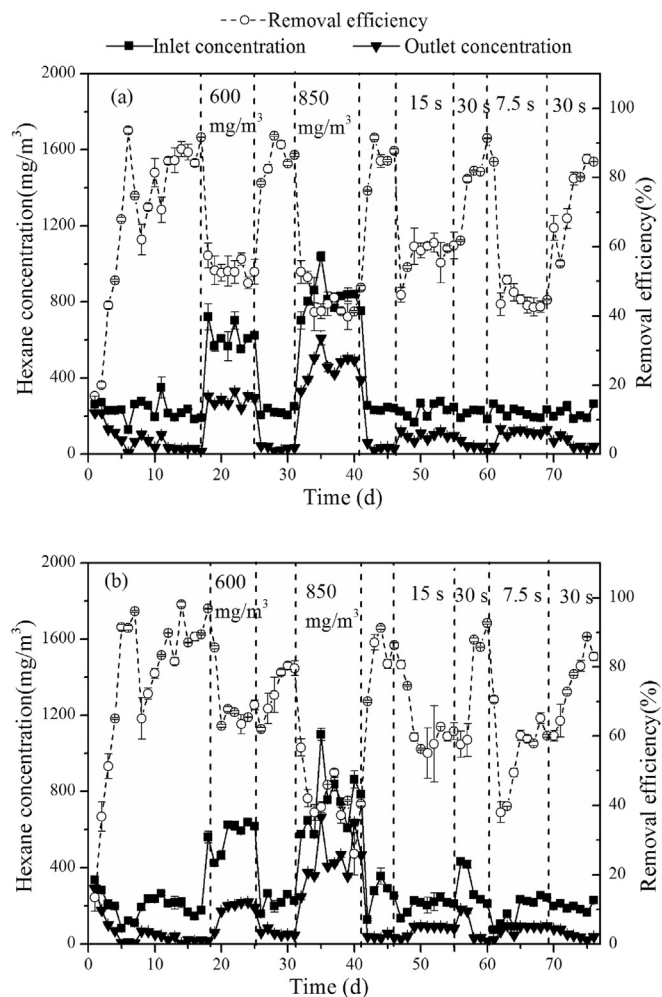


Fig. 5. Performance of both BTFs in the presence of SDS. (a) BTF1 without SDS; (b) BTF2 with SDS.

by the fact that the tolerance of microorganisms to substrate toxicity was reduced and microbial activity was inhibited under the condition of higher VOC concentration (Zehraoui et al., 2013), as a result, more chances are provided for SDS to be degraded by the microorganism, leading to the slightly lower RE of BTF2 than that of BTF1.

When varying the EBRT from 30 s to 15 s under a constant concentration of 200 mg m⁻³ (Fig. 5), it was observed that n-hexane RE of both BTFs suddenly dropped to about 60%, suggesting that high VOC concentration could greatly reduce the extent of n-hexane biodegradation. This result was consistent with the data reported by Yang et al. (2008). It is worth noting that the n-hexane RE still remained about 60% for BTF2 while the RE of BTF1 decreased to 43% when the EBRT was decreased to 7.5 s at an n-hexane concentration of 200 mg m⁻³, which shows that SDS enhanced the performance of the biofilter. Similar results were reported by other researchers (Liu et al., 2007). In addition, some researchers found that other surfactants such as Triton X-100 and saponins could also enhance the performance of biofilters for n-hexane removal (Hassan and Sorial, 2008; Tu et al., 2015). Wang et al. (2014) reported the RE of ethylbenzene decreased from 78% to 61% in BTF with surfactant and from 59% to 37% in BTF without surfactant EBRT decreased from 30 to 15 s at an inlet concentration of 1650 mg m⁻³, respectively.

4. Conclusions

SDS could be biodegraded by microorganisms and had no toxic to microorganisms at all the tested concentration. The optimal concentration of SDS for treating n-hexane in BTFs was 0.1 CMC and a maximum RE of 70% and EC of $50.4 \text{ g m}^{-3} \text{ h}^{-1}$ were obtained at a fixed influent concentration of 600 mg m^{-3} and an EBRT of 30 s. The RE of BTF1 and BTF2 decreased with the increasing inlet concentration at a constant EBRT or the decreasing gas EBRT at a constant inlet concentration under 0.1 CMC of SDS.

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