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ORIGINAL PAPER

Effect of empty bed residence time on biotrickling filter performance: case study—triethylamine

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Abstract In this study, a laboratory-scale biotrickling filter (BTF) is used to remove *Triethylamine* (TEA) from gaseous wastes. The BTF is made of stainless steel with a height of 210 cm and an internal diameter of 21 cm packed with lava rocks. TEA elimination pattern was evaluated by changing empty bed residence times (EBRTs). The maximum elimination capacity (EC) has been determined to be 87 g/m^3/h. At all EBRTs 52, 31, 20, and 10 s, contaminant transferring from gas phase to liquid was more than the EC. Also, the removal efficiency was 100 % for a mass loading of 100 g/m^3/h. While the liquid recirculation velocity of 3.466 m^3/m^2/h was maintained, the flow rate was adjusted to 60, 100, 156, and 312 L/min. The results show that due to the high solubility of TEA in water for all the EBRTs, TEA can be solved in the circulated liquid and then be degraded gradually by microorganisms. Therefore, the least EBRT of 10 s is more appropriate.

Keywords Air pollution · Biotrickling filter · Empty bed residence time (EBRT) · Triethylamine

Introduction

Triethylamine (TEA) is an organic compound used as a catalytic solvent in chemical syntheses. It acts as an accelerator activator for rubbers, corrosion inhibitor, and propellant used in manufacturing of wetting, penetrating, and waterproofing agents of quaternary ammonium compounds as well as desalination of seawater (Budavari 1996; HSDB 2011). In addition, TEA is used as a curing and hardening agent for polymers. In this process, TEA is usually collected through air-conditioning hoods and ducts (Golbabai et al. 2003).TEA has a fishy ammonia-like odor with a threshold of 0.48 parts per million (ppm), discontenting workers and environmental habitants (Pohanish and Sittig 2012). Acute exposure of humans to TEA vapor causes eye irritation, corneal swelling, halo vision, and irritation in the skin and mucous membranes (Budavari 1996). Furthermore, it has harmful metabolic pathways in humans (Akesson et al. 1988).

Tertiary amines, such as TEA, are the main gaseous catalysts comprising the majority of nitrogenous emissions (Boger et al. 1997; Busca and Pistarino 2003). Nowadays, many industries such as Iranian car makers use TEA in a process called cold molten casting. TEA sampling in the PUCB unit (phenolic urethane cold box) of these companies showed the average concentration of 430 mg/m^3 in the exhausted gas. Due to low price of natural gas in Iran, the hot molten casting is preferred to the cold molten casting that has higher precision compared to the hot molten (Golbabai et al. 2003). Therefore, it is necessary to find simple and inexpensive methods that can be more instrumental to obtain higher efficiency in reducing this kind of pollution. Many methods have been studied for the reduction and elimination of air pollution. Biological process has been determined to be a highly efficient technology to remove odorous or toxic waste gas of volatile organic compounds (He et al. 2009; Kaosol and Pongpat 2012; Luvsanjamba et al. 2008; Wan et al. 2011a).

Biofiltration has been classified into three types: biofilters (BFs), biotrickling filters (BTFs), and bioscrubbers

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(Devinny et al. 1999). A biofilter is substantially a compost bed through which the waste gases can pass and be absorbed simultaneously, leading to the compost bed to be degraded by the immobilized microorganism on the packing materials. Although BTFs operate in a similar way to BFs, nutrimental solution that contains essential inorganic nutrients such as nitrogen, phosphorous, potassium, and some microelements is continuously trickled on the packing materials (He et al. 2009). The contaminants in the gas phase are dissolved into the liquid phase and then diffused into the biofilm phase to be degraded (Lee et al. 2010).

It has been proven that the BTFs are more efficient in purifying various odorous compounds compared to the BFs (Wan et al. 2011b). The continuous control of liquid phase parameters such as salinity, pH, pressure drop, and nutrient content in the BTFs allows for the excellent and higher performance compared to biofilters (Cox and Deshusses 2000). Furthermore, the BFs and BTFs are limited by a critical inlet load (IL), above which the removal efficiency of the bioprocess decreases. For the water-soluble volatile organic compounds (VOCs) such as TEA, the critical IL is smaller in the BFs compared to the BTFs. Continuous trickling of the aqueous nutrient solution in a BTF is probably the reason for a higher performance when a water-soluble VOC is treated. This can be due to the high solubility of TEA in water (Avalos Ramirez et al. 2009).

During the past decade, numerous studies have been conducted concerning the treatment of contaminated gas stream with amines biofiltration (Chou and Shiu 1997; Moussavi et al. 2011; Torkian et al. 2005). Moreover, there are several studies on the elimination of waste gas containing TEA using the BFs (Belin et al. 1983; Busca and Pistarino 2003; Gandu et al. 2013; Torkian et al. 2005, 2007). However, no study has been published using an aerobic BTF system for the removal of waste gas containing TEA besides the authors' previous paper (Mehrdadi et al. 2010).

Investigation of TEA removed by the BTFs is more troublesome compared to many other materials for the plurality of samplings. The pollution on the gas stream output appears in the forms of TEA and ammonia as well as on the liquid phase in the forms of TEA, NH_3, NO_2, and NO_3. All these substances should be measured in each step, what has been happened in this study.

Among all parameters affecting biotrickling efficiency, in this study, empty bed residence time (EBRT) is investigated. The EBRT has a strongly direct correlation with plant sizes as well costs of construction, maintenance, and operation. Decreasing in the EBRT means increasing in mass and volume loadings, which causes the treatment plant's size to be minimized (Wan et al. 2011a). In this study, the removal efficiency (RE) and the elimination capacity (EC) are evaluated to minimize the EBRT with different inlet concentrations and flow rates. The experimental results obtained will provide useful information concerning the design criteria and operation in order to control the waste gas containing TEA.

TEA contaminant emissions are temporal in many industries. For example, in foundries, there is a high emission exhaust of TEA with 5-s duration for every 2 min. Therefore, it is highly practical to apply a method which is able to completely eliminate this pollution from the outlet and degrade it to similar substances during the remaining time till the subsequent exhaust. This condition is called noncontinuous condition that has been studied in this paper.

Materials and methods

Experimental setup

The experimental setup consisted of a laboratory-scale biotrickling filter and an influent gas supply system, as illustrated in Fig. 1. The biotrickling filter was made of stainless steel with an internal diameter of 21 cm. The spaces of the cylinder, without packed material at the top and bottom, had the height of 30 cm. The biotrickling column with a height of 150 cm was packed with lava rocks (size range of 2–4 cm with average diameter of 3 cm, initial bed porosity of 60 %, and a 52-L packing volume). The lava rocks were taken from the mountains near Tehran. They are comparatively inexpensive and easily obtainable in most parts of the country. Activated sludge for the seeding was obtained from Tehran plant of municipal wastewater treatment. The biotrickling filter was equipped with a liquid recirculation system, including a total liquid volume of 40 L. The main air stream was supplied by a compressor. A side stream of air was sent through a 1-L bottle containing pure liquid TEA. The rest was mixed with the side stream containing pollutant vapor. Air flow rates were properly controlled using pressure regulators, and flow meters were applied to produce feed air with required concentration. Temperature control of the bed was attained by placing the return liquid container in a water tank with the temperature maintained constant at 25 ± 1 °C. A heated element was used for the temperature control in the water tank.

Analytical methods

Gas samples were collected at the inlet, outlet, and in the 50 and 100 cm height of the bed by taking the gas through the impinger containing 20 mL methanol as TEA solvent. The gas flow rate was 340 mL/min for 15 min of sampling time. TEA amount in methanol was measured by UV

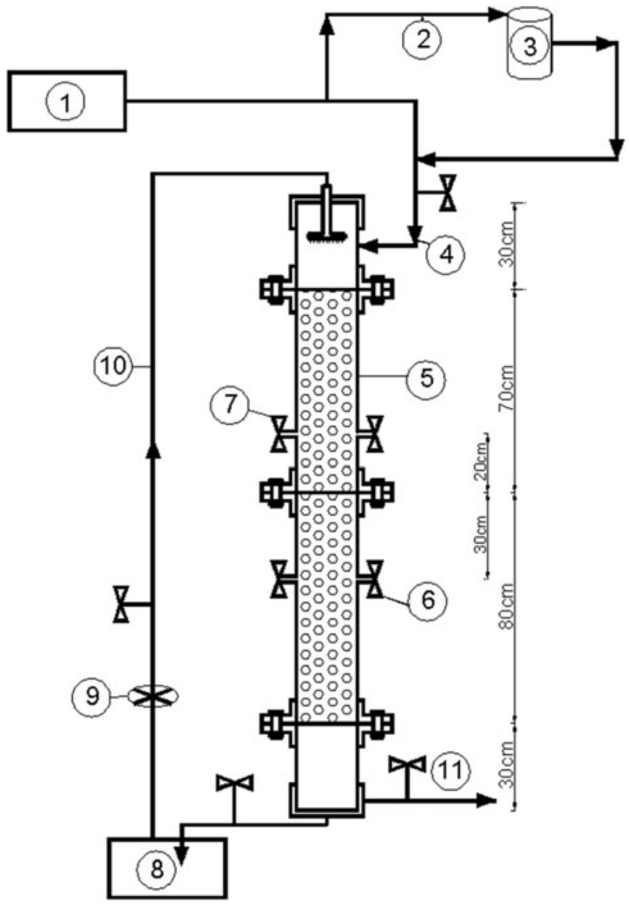

Fig. 1 Schematic diagram of the experimental system. *1* Air pump, *2* Needle valve, *3* Aqueous solution of TEA, *4* Rotameter, *5* Biotrickling filter, *6* Gas sampling port, *7* Liquid sampling port, *8* Recycled water chamber, *9* Liquid pump, *10* Recycled water line, *11* pure air

spectrophotometer (UV/VIS) at a wavelength of 215 nm (Torkian et al. 2007). Standards were prepared by introducing the known volumes of TEA into 5-L Tedlar bags. Appropriate volumes were selected based on vapor pressure calculations at room pressure and temperature. The equilibrium vapor pressures were calculated as 7.7 kPa (3,142 mg/m^3) at 25 °C and 101.325 kPa for the TEA.

In all concentration measurements, pollution was presented in the forms of TEA or ammonia in gas phase or TEA, ammonia, or ammonium in liquid phase, leading to the assumption that the complete degradation had not been occurred. Thus, for all the calculated EC, the amount of nitrogen in ammonia and ammonium was not assumed to be eliminated. After converting all ammonia and ammonium to nitrate and nitrite, TEA was assumed to be eliminated. Therefore, the EC means the amount of TEA that is converted to nitrate and nitrite. This approach caused the measurements to be a severe part of the study.

Although the instant mass loading of TEA has a theoretically direct correlation with the influent TEA concentration, in real condition having constant flow rate is impossible. Therefore, mass loading is obtained by dividing the total inlet of mass pollution into the total inlet of flow gas. Several parameters of biotrickling filter performance are defined in Table 1.

Startup and operation

The operation sequence shown in Table 2 is discussed in details by Mehrdadi et al. (2010). In the first phase, in order to expedite the commencement phase, the returned sludge from municipal waste treatment plant was kept and aerated in a separate tank for 55 days. After microorganisms were adapted, 15 L of the liquid was filtered and used as return liquid in recirculation liquid tank of the biotrickling filter pilot. In the second phase, during 65 days, the influent TEA concentrations were changed from 40 to 2,000 mg/m^3. A constant EBRT of 312 s and a recirculation liquid velocity

Table 1 Definition of biotrickling filter performance parameters

Parameter	Definition	Units	Explanations
Empty bed volume	V_f	m^3	–
Total air flow rate	Q	m^3/h	–
Porosity	θ	–	–
Recirculation liquid flow rate	V_L	m^3/m^2/h	–
TEA concentration	C_{in} and C_o	g/m^3	The subscript in indicates that the compound is in an inlet stream, and the subscript o indicates that the compound is in an outlet stream
Empty bed residence time	$EBRT = V_f/Q$	Sec	The time that a flow element need to receive from empty reactor inlet to the outlet
Real residence time	$T = V_f * \theta/Q$	Sec	The time that a flow element need to receive from cylinder with packed mat inlet to the outlet
Mass loading	$IL = (Q * C_{in})/V_f$	g/m^3/h	–
Removal efficiency	$RE = (C_{in} - C_o)/C_{in} * 100$	%	The fraction of contaminant removed by biofilter
Elimination capacity	$EC = Q(C_{in} - C_o)/V_f$	g/m^3/h	Mass of the contaminant degraded per unit volume of filter material per unit time

Table 2 The operation sequences and conditions

Phase	Purpose	Duration (day)	C_{in} (mg/m^3)	EBRT (s)	V_L (m^3/m^2/h)
1	Adaptation	55	–	–	–
2	Biofilm cultivation	65	0–828	312	3.466
3	Effect of empty bed residence time	20	900–3,400	52	3.466
		25	550–2,400	31.2	3.466
		22	400–1,600	20	3.466
		17	200–800	10	3.466

of 3.466 m^3/m^2/h (2 L/min) were maintained for the full test period. Gas samples at inlet and outlet column and liquid samples at liquid sampling port after liquid pump were taken everyday and analyzed for TEA and by-products concentrations.

After reaching the stable conditions, the removal efficiencies of TEA were evaluated by varying the influent TEA concentrations and the superficial gas velocities through the column in the phase three of the experiment. The EBRT was set to 52, 31, 20, and 10 s. Air inflow to the filter was adjusted at 60, 100, 156, and 312 L/min (EBRT decreasing below 10 s was not possible for the pump power limitation), and a liquid recirculation velocity of 3.466 m^3/m^2/h was maintained for this phase.

Noncontinuous inlet pollutant with high concentration

In the condition of continuous inlet pollutant, if mass loadings (ILs) are more than elimination capacity (EC), pollution concentration in the liquid phase will increase. It makes TEA to be detected on the gas phase outlet, leading to the reduction in RE. This prevents studying of the real effects of IL and C_{in} on the TEA elimination. To avoid this problem, the experimental condition was changed by altering the TEA injection trend, similar to the real condition of many industries with noncontinuous inlet pollutant. In this condition, high concentration pollutant of inlet gas enters the reactor and concentration in the liquid phase is kept below the limit of desorption using a temporary intercept of inlet gas. Both continuous and noncontinuous conditions are studied in this paper.

Results and discussion

Continuous inlet pollutant

The effect of inlet pollutant concentration on the RE in different EBRTs during 75 days of pilot operation is showed in Fig. 2. There is a threshold concentration for each EBRT below which the RE is 100 %, and by increasing the concentration to more than this threshold, the RE will decrease. In this condition, for all EBRTs, by increasing each IL in steps of 2.5 g/m^3/h, there is nearly 1 % decreasing on the RE. It is noteworthy that for all the EBRTs, this trend is very similar.

At the EBRT 52 s, the RE is 100 % for all concentrations below 1,375 mg/m^3 (IL = 88 g/m^3/h) and for a concentration of 1,829 mg/m^3 (IL = 120 g/m^3/h), the RE is 87 %. The maximum EC in this EBRT is 85 g/m^3/h. At the EBRT 31 s, the RE is 100 % for all concentrations below 833 mg/m^3 (IL = 96 g/m^3/h) and for a concentration of 1,159 mg/m^3 (IL = 128 g/m^3/h), the RE is 82 %. The maximum EC in this EBRT is 88 g/m^3/h. At the EBRT 20 s, the RE is 100 % for all concentrations below around 528 mg/m^3 (IL = 120 g/m^3/h) and for a concentration of 638 mg/m^3 (IL = 150 g/m^3/h), the RE is 85 %. The maximum EC in this EBRT is 85 g/m^3/h. Finally, at EBRT 10 s (Fig. 3), the RE is 100 % for all concentrations below around 265 mg/m^3 (IL = 120 g/m^3/h) and for a

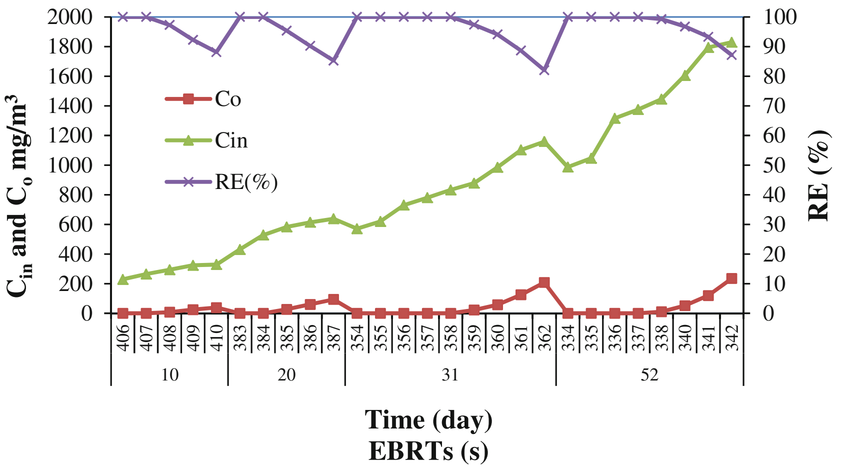

Fig. 2 C_{in} and C_o and RE at different EBRTs for continuous inlet pollutant

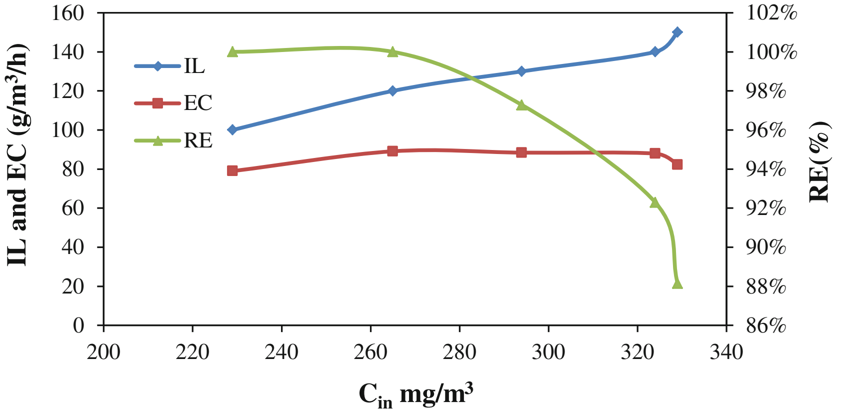

Fig. 3 IL, EC, RL, and RE versus C_{in} at EBRT 10 s

concentration of 329 mg/m^3 (IL = 150 g/m^3/h), the RE is 88 %. The maximum EC in this EBRT is 89 g/m^3/h.

In the condition of continuous inlet pollutant, when ILs are more than EC, by increasing the pollution concentration on liquid phase, the TEA transfer from gas-to-liquid phase and the desorb process will decline, resulting in the increase in outlet gas concentration. Table 3 shows the relation of the TEA concentration in liquid phase and in gas phase outlet at EBRT 52, 31, and 20 s, respectively. For example, at the EBRT 52 s and C_{in} of 919 mg/m^3, when concentration on liquid phase is 6,100 mg/L, C_o is 79 mg/m^3, but by increasing the concentration on liquid phase to 7,150 mg/L, for the C_{in} of 919 mg/m^3, the C_o decreases to 140 mg/m^3.

The maximum EC in the EBRTs investigated in this study is 87 ± 2 g/m^3/. In the condition of having continuous ILs higher than this EC, microorganisms will not be able to eliminate TEA effectively and thus, the TEA's concentration in liquid phase will increase. High solubility of TEA and its by-products' degradation in liquid phase lead to the accumulation of recirculation liquid; therefore, the RE relevant to its definition with C_{in} and C_o is 100 %, but with respect to the IL and the EC, it is not 100 %.

Noncontinuous inlet pollutant

It is remarked that in this condition, inlet gas with a high concentration pollutant enters the reactor and the concentration in liquid phase is kept below the limit of desorption. The TEA solubility in the liquid phase is a key point for this method prosperity. In the noncontinuous conditions, higher concentrations with better REs are attained. For example, an input with concentration 3,000 mg/m^3 will be degraded with the RE of 92 % at the EBRT 52 s. The results of this condition are presented in Fig. 4.

At the EBRT 52 s, when liquid phase concentration is higher than 1,629 mg/L, TEA on gas phase outlet will be detected. In this condition and during days 344–353 of pilot operation, when liquid phase concentration was lower than 1,629 mg/L, with making a break on inlet pollutant, simultaneous gas sampling from inlet and outlet was performed. The RE is 100 % for all concentrations below 1,493 mg/m^3

Table 3 TEA concentrations in liquid phase (mg/L) and gas phase output (mg/m^3)

EBRT = 52 s			EBRT = 31 s			EBRT = 20 s		
TEA on:								
Gas phase input	Gas phase output	Liquid phase	Gas phase input	Gas phase output	Liquid phase	Gas phase input	Gas phase output	Liquid phase
804	0	1,623	552	0	1,866	431	0	85
820	5	2,040	555	12	2,682	528	0	985
860	9	2,690	567	20	3,345	583	27	2,092
878	16	3,400	560	34	3,987	614	45	3,652
894	28	4,100	586	40	4,542	638	74	5,634
911	41	4,890	590	46	5,015	–	–	–
924	57	5,566	616	56	5,385	–	–	–
919	**79**	**6,100**	–	–	–	–	–	–
915	100	6,500	–	–	–	–	–	–
919	**140**	**7,150**	–	–	–	–	–	–

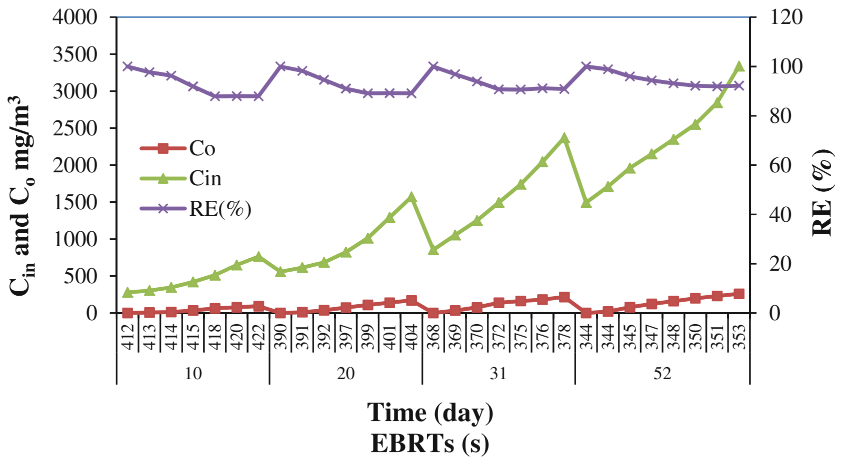

Fig. 4 C_{in} and C_o and RE at different EBRTs for noncontinuous inlet pollutant

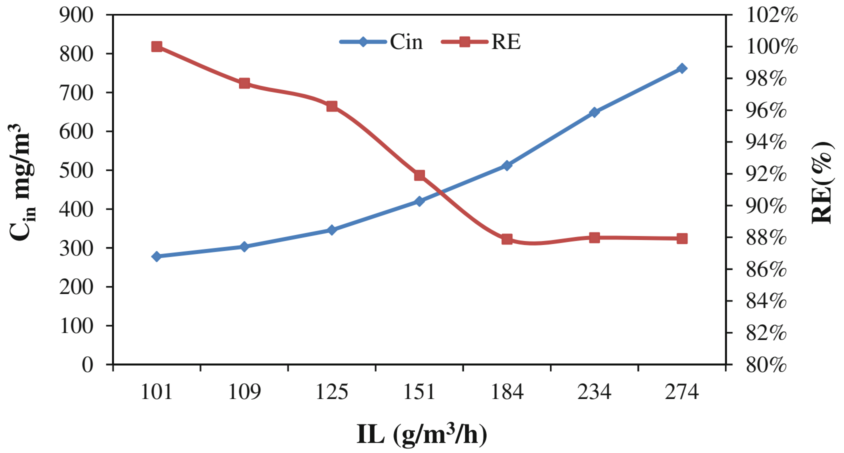

Fig. 5 IL versus RE and C_{in} at EBRT 10 s for noncontinuous inlet pollutant

Table 4 TEA IL and RE at EBRT 52, 31, 20, and 10 s

EBRT = 52 s		EBRT = 31 s		EBRT = 20 s		EBRT = 10 s	
IL	RE	IL	RE	IL	RE	IL	RE
103	100	98	100	100	100	101	100
118	99	121	97	110	98.2	109	97.7
135	96	144	94	137	96.6	125	96.2
149	94	172	90.7	161	92.8	151	91.9
162	93	200	90.6	197	89	184	88
176	92	235	91	231	89	234	88
196	92	273	90.9	281	89	274	88

(IL = 135 g/m^3/h) and for a concentration of 3,337 mg/m^3 (IL = 231 g/m^3/h), the RE is 92.2 %. At the EBRT 31 s, the RE is 100 % for all concentrations below 854 mg/m^3 (IL = 98 g/m^3/h) and for a concentration of 2,370 mg/m^3 (IL = 273 g/m^3/h), the RE is 91 %. At the EBRT 20 s, the RE is 100 % for all concentrations below 558 mg/m^3 (IL = 100 g/m^3/h) and for a concentration of 1,570 mg/m^3 (IL = 281 g/m^3/h) the RE is 89 %. Finally, at EBRT 10 s (Fig. 5), the RE is 100 % for all concentrations below 278 mg/m^3 (IL = 101 g/m^3/h) and for a concentration of 762 mg/m^3 (IL = 274 g/m^3/h), the RE is 88 %.

The TEA REs for different ILs and the conditions in which concentration in liquid phase is below the desorption limit are summarized in Table 4. As shown in the table, in all EBRTs, the RE is 100 % for IL 100 g/m^3/h. Hence, if IL is around maximum of EC (87 g/m^3/h), TEA certainly will not be detected in outlet. In addition for IL around doubled EC at EBRT 10 s, the RE is nearly 88 %. It is noteworthy that in all studied EBRTs, contaminant transferring from gas phase to liquid phase is excessive and higher than the EC, and the microorganism's activity determines the EC.

By increasing the IL from the EC to around 170 g/m^3/h, the RE is decreased linearly ($R^2 = 0.99$). The slopes of this linear relationship for four tested EBRTs are approximately constant. For fixed IL, each 10 s growth in EBRT causes 1 % growth on the RE. It is considerable that for ILs above 170 g/m^3/h, the RE is nearly constant for the high TEA solubility in liquid phase. The RE-measuring results for different ILs in studied EBRTs, when the concentration in liquid phase is below desorption limit, are shown in Fig. 6.

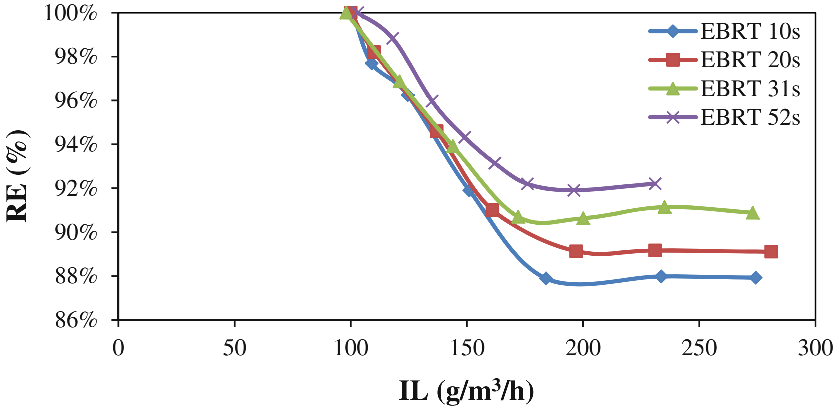

Fig. 6 RE versus IL at EBRT 52, 31, 20, and 10 s for noncontinuous inlet pollutant

For comparing the results, a successful study about trimethylamine (TMA) (Wan et al. 2011b) was selected. According to that study, maximum EC was 13.95 g/m^3/h with RE = 78.1 % at a TMA concentration of 0.42 mg/L, whereas for TEA, as it was mentioned, the maximum EC was 87 ± 2 g/m^3/h (RE = 100 %) at a TEA concentration of 2.0 mg/L. In that study, 100 % removal efficiency was acquired for EBRT larger than 110 s at an inlet concentration of 0.30 mg/L, whereas for TEA, as it was mentioned, the RE was 100 % for all concentrations below 1.493, 0.854, 0.558, and 0.278 mg/L at the EBRTs 52, 31, 20, and 10 s, respectively. Therefore, the inlet concentration of TEA in general can be higher than the inlet concentration of TMA.

Conclusion

A biotrickling filter has an appropriate output for TEA treatment. The maximum EC 87 ± 2 g/m^3/h was obtained at the EBRTs 52, 31, 20, and 10 s, and for all these EBRTs, the RE was 100 % for an IL 100 g/m^3/h. Due to the TEA high solubility in water, even contaminant concentrations higher than EC can be solved in recirculation liquid and are gradually eliminated. In spite of fivefold flow rate at the EBRT 10 s than 52 s, in the same IL, the REs are not very different and increasing EBRT for better RE is not highly effective; therefore, the least EBRT of 10 s is more appropriate than the other EBRTs.

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