



# Investigations into the application of a combination of bioventing and biotrickling filter technologies for soil decontamination processes—A transition regime between bioventing and soil vapour extraction

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## ABSTRACT

Bioventing has emerged as one of the most cost-effective *in situ* technologies available to address petroleum light-hydrocarbon spills, one of the most common sources of soil pollution. However, the major drawback associated with this technology is the extended treatment time often required. The present study aimed to illustrate how an intended air-injection bioventing technology can be transformed into a soil vapour extraction effort when the air flow rates are pushed to a stripping mode, thus leading to the treatment of the off-gas resulting from volatilisation. As such, a combination of an air-injection bioventing system and a biotrickling filter was applied for the treatment of contaminated soil, the latter aiming at the treatment of the emissions resulting from the bioventing process. With a moisture content of 10%, soil contaminated with toluene at two different concentrations, namely 2 and 14 mg g soil<sup>-1</sup>, were treated successfully using an air-injection bioventing system at a constant air flow rate of ca. 0.13 dm<sup>3</sup> min<sup>-1</sup>, which led to the removal of ca. 99% toluene, after a period of ca. 5 days of treatment. A biotrickling filter was simultaneously used to treat the outlet gas emissions, which presented average removal efficiencies of ca. 86%. The proposed combination of biotechnologies proved to be an efficient solution for the decontamination process, when an excessive air flow rate was applied, reducing both the soil contamination and the outlet gas emissions, whilst being able to reduce the treatment time required by bioventing only.

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## 1. Introduction

In response to an increasing demand to address soil contamination by volatile organic compounds (VOCs), remediation technologies have been developed [1]. Of the available biological treatment methods, bioventing (BV), has emerged as one of the most cost-effective *in situ* technologies presently accessible to address vadose zone remediation of petroleum derivatives contaminated sites, including gasoline [2–5]. BV, integrating a volatilisation process with a bioremediation process [6,7], can be seen as an adaptation of soil vapour extraction (SVE) [3,8–13], which is a standard soil remediation technique that relies on the maximisation of VOC volatilisation via vapour extraction [14–17]. Lee et al. [18] and Malina et al. [19] investigated the feasibility of using SVE technology for a petroleum hydrocarbon-contaminated site, both reporting high initial mass removals. Malina et al. [19] observed that 4 mg g soil<sup>-1</sup> of toluene initially present in soil was reduced by 99% within 24 days of applying a constant gas flow of 40 cm<sup>-1</sup> cm<sup>-1</sup> h<sup>-1</sup>. Air-injection bioventing (AIBV) differs from

SVE in that AIBV is generally accomplished through the injection of air into the subsurface of the soil [14], enhancing the natural *in situ* biodegradation of aerobically degradable compounds in soil. By providing oxygen to existing indigenous microbial flora, the aerobic bioremediation process is favoured, thus minimising VOC migration or associated emissions [2,14,20]. Natural hydrocarbon-degrading microorganisms must be present in the soil at concentrations large enough to attain reasonable biodegradation rates [20]. Österreicher-Cunha et al. [2] evaluated the influence of BV or AIBV in gasoline contaminated soil, achieving, in 60 days, ca. 95% of gasoline removal (initial concentration of ca. 117 mg g soil<sup>-1</sup>), applying a 2 psi constant air pressure. Tsao et al. [20] have also reported high proportions of benzene, toluene and xylene (BTX) removal (52–68%), with maximal rates of mineralisation of 6.1–8.0% g of soil<sup>-1</sup> day<sup>-1</sup>. Other applications of BV or AIBV are also found in literature [5,8,21–23].

The treatment of soil contaminated due to accidental spills of petroleum derivatives, such as gasoline, requires high stripping rates to maximise volatilisation of the VOCs, similar to a SVE regime. It is known that one of the major drawbacks associated with BV or AIBV is the extended time of treatment, varying from a few months to years, depending on the specific site conditions [24,25]. When the injection of air is increased it may reach rates favouring stripping of

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the contaminating compounds, pushing the system into conditions which may not allow for the timeframe needed for biodegradation to occur. Nevertheless, AIBV can be adapted to a stripping mode by using an air flow rate higher than the lower aeration rate needed to promote biodegradation, resulting in the transition regime between AIBV and SVE, where volatilisation takes place but the biodegradation processes can also occur.

When high air flow rates are applied, the volatilisation process promotes the transfer of VOCs to the vapour phase, which then leads to the need to treat the resulting off-gas previous to the release into the atmosphere [26]. Biotrickling filters have proven to be a cost-effective and environmentally friendly technology for the control of large air streams contaminated with moderate concentrations of VOCs. Often, these type of emissions result from commonly used remediation techniques such as air sparging, SVE and air stripping [26,27–30]. The use of biotrickling filters for toluene treatment is found in the literature. Cox and Deshusses [31] observed, at a volumetric load of  $170 \text{ g m}^{-1} \text{ h}^{-1}$ , maximum elimination capacities of  $70 \text{ g toluene m}^{-1} \text{ h}^{-1}$ . Chou and Wu [32] indicated that, for a test period of 121 days, toluene removal efficiencies of over 90% were obtained at an OL  $30 \text{ g m}^{-3} \text{ h}^{-1}$ . As such, a combination of an AIBV system and a biotrickling filter applied for the treatment of contaminated soil has the advantage of the latter aiming at the treatment of the emissions resulting from the bioventing process. Such combined technologies could be potentially useful for situations when soil restoration of hydrocarbon-contaminated site needs to be promoted or accelerated.

This study aimed to illustrate how an intended AIBV technology can be used and transformed into a SVE effort when high air flow rates are preferential, thus leading to the need to treat the off-gas resulting from volatilisation. A combination of two biotechnologies, air-injection bioventing system followed by a biotrickling filter, was investigated at laboratory scale to treat a superficial soil artificially contaminated with toluene with different toluene loads, using injection of high air flow rates, similar to those reported for SVE, pushing the regime to a soil stripping mode.

## 2. Materials and methods

### 2.1. Microbial inoculum

A microbial inoculum able to biodegrade toluene has been previously enriched in the laboratories using batch methods, following procedures described in Bastos et al. [33], and was used to inoculate the biotrickling filter reactor.

### 2.2. Soil

The soil used was a natural soil, freshly collected from the top layer (10 cm under surface) of a lawn area at Escola Superior de Biotecnologia, Universidade Católica Portuguesa, Porto. Soil samples collected and used in the experiments had no history of hydrocarbon exposure and no toluene was detected. The porosity, bulk density, and pH of the soil were 0.2,  $756 \text{ kg m}^{-3}$  and 6, respectively. The soil was passed through a sieve with 1.8-mm-diameter holes, and mineral medium (MM) was added to bring it to a soil moisture of 10%. The MM used in these experiments was composed by the following components per  $\text{dm}^3$ :  $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$  (2.67 g; Merck, Germany);  $\text{KH}_2\text{PO}_4$  (1.4 g; Merck, Germany);  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  (0.2 g; Merck, Germany);  $(\text{NH}_4)_2\text{SO}_4$  (0.5 g; Sigma, Germany); Yeast extract powder (0.02 g; LABM, England); and  $10 \text{ cm}^3$  of a metal supplement solution. This solution contained the following compounds per  $\text{dm}^3$ : NaOH (2.0 g; Merck, Germany);  $(\text{Na}_2)\text{EDTA} \cdot 2\text{H}_2\text{O}$  (12.0 g; Pronalab, Portugal);  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  (2.0 g);  $\text{CaCl}_2$  (1.0 g; Merck, Germany);  $\text{Na}_2\text{SO}_4$  (10.0 g),  $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$  (0.4 g; Merck, Germany);

$\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$  (0.4 g; Merck, Germany),  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  (0.1 g; Merck, Germany),  $\text{NaMoO}_4 \cdot 2\text{H}_2\text{O}$  (0.1 g; Merck, Germany) and  $\text{H}_2\text{SO}_4$  conc. ( $0.5 \text{ cm}^3$ ).

The contamination of soil was undertaken by adding toluene homogeneously to achieve the desired soil concentrations.

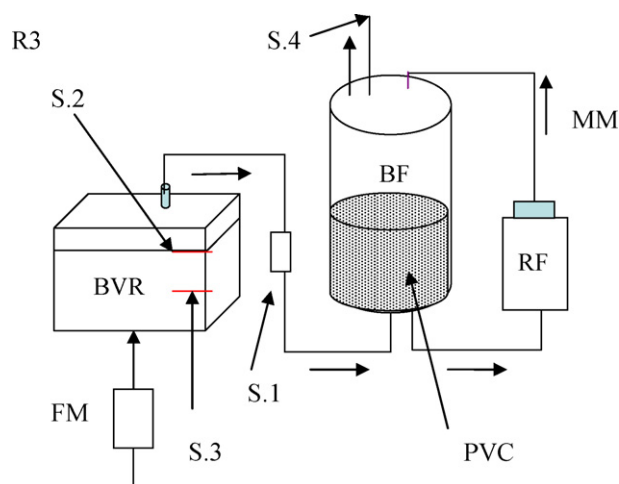
### 2.3. Investigation of biodegradation of toluene in soil

The presence of toluene degraders in the indigenous microbiota of the collected soil was investigated by incubating a small portion of soil samples (2 g) in flasks containing MM amended with toluene at concentrations of  $100 \text{ mg dm}^{-3}$ . Transfers were made every 2 days with fresh MM supplemented with toluene. After 6 days,  $10 \text{ cm}^3$  of the culture was added to two flasks containing MM with 100 and  $500 \text{ mg dm}^{-3}$  of toluene, each. Flasks were incubated at  $24^\circ\text{C}$  with gentle agitation. Monitoring of microbial growth was undertaken by periodically collecting samples from the flasks for the determination of the optical density at 600 nm. The experiments were carried out under aseptic conditions.

### 2.4. Experimental set-up of the remediation reactors

A schematic representation of the proposed combination of biotechnologies is shown in Fig. 1. The AIBV system set-up was composed of three micro-scale reactors consisting of an acrylic bucket with multiple perforations for supplying compressed air. The air flow rate was controlled by air flow meters (Hastings, UK). Each reactor, with a total volume of  $1236 \text{ cm}^3$ , was internally protected by aluminium foil to avoid the adsorption of the organic compounds to the walls. After the addition of ca. 514 g of contaminated soil in the reactors, each reactor was carefully sealed. The reactor cover was designed with two outlets, one to take soil samples and another to allow for the collection of gas samples.

Reactor 1 (R1) worked as a control, receiving no gas feed; reactor 2 (R2), an air-injection bioventing control, was ventilated with a controlled air flow to provide constant oxygenation of the soil. Reactor 3 (R3) was a combination of an air-injection bioventing reactor (BVR), having the same conditions of R2, with a biotrickling filter (BF) (Fig. 1) fed with the outlet gas of BVR.



**Fig. 1.** Apparatus of the proposed combination of the technologies bioventing and biotrickling filter (R3 reactors). BF: biotrickling filter; FM: air flux-meters HFM-60; PVC: growth support medium; BVR: bioventing reactor; RF: recycling flask (MM feed);  $S_i$  ( $i=1-4$ ): sampling points.

### 2.5. Biotrickling filter set-up

The BF consisted of an 80 cm high and 8 cm internal diameter cylindrical stainless-steel column that was filled with 1500 cm<sup>3</sup> of polyvinyl chloride (PVC) pall-rings as a support medium (ca. 220 g). For biofilm immobilisation, the microbial inoculum pre-grown on toluene and further supplied with a toluene concentration of ca. 100 mg dm<sup>-3</sup> was fed (46 cm<sup>3</sup> h<sup>-1</sup>) continuously for 7 days, in a recirculation system. A water nozzle was used at the top of the BF. During BF operation, the gaseous effluent was continuously fed to the bottom of the reactor in a counter current mode with the liquid flow, and to allow a better distribution of the gaseous effluent through the reactor a perforated stainless steel plate was placed at the bottom of the column.

### 2.6. Experimental set-up

The operation of the proposed combination of technologies was investigated at two different toluene concentration scenarios.

In order to adapt the AIBV to a stripping mode to maximise VOC volatilisation, an air flow rate higher than the lower aeration rate needed to enhance biodegradation was applied, resulting in the transition regime between AIBV and SVE. Malina et al. [19] reported that 4 mg g soil<sup>-1</sup> of toluene initially present in soil was reduced by 99% within 11 days of AIBV application. Using the toluene degradation rate obtained in the latter study, and considering the respiration equation, the minimum aeration rate for biodegradation was estimated to be 6.67E-04 dm<sup>3</sup> air min<sup>-1</sup> (1.4E-04 dm<sup>3</sup> O<sub>2</sub> min<sup>-1</sup>).

In the present study the AIBV was conducted, in reactors R2 and BVR, with a constant air flow rate of 0.13 dm<sup>3</sup> min<sup>-1</sup> (2.5E-04 dm<sup>3</sup> min<sup>-1</sup> g<sup>-1</sup> soil) (AOM 1000, Intelligent Flow Meter, Germany), and BF was fed with MM at a flow rate of 24 cm<sup>3</sup> h<sup>-1</sup>. Throughout the experiments the toluene concentration was periodically monitored, at four sampling points, namely at the outlet gas sampling point (S.1), at the soil surface (S.2) and at a deeper level of the soil (4 cm) (S.3) of reactors R1, R2 and BVR (Fig. 1). In the BF reactor, the sampling port was located at the top of the column (S.4) (Fig. 1). The organic load (OL g d<sup>-1</sup> m<sup>-3</sup>-reactor) to the BF and the removal efficiencies (RE), expressed as percentage, were determined for each experiment. A global toluene mass balance in R1, R2 and R3, and a toluene mass balance in the BF were carried out.

### 2.7. Analytical methods

Toluene quantification was carried out using gas chromatography on a Varian Star 3400 CX model equipped with a flame ionisation detector (FID) detector and a CP-Wax 52 CB capillary column (Chrompack International B.V., Middelburg, The Netherlands), under temperature conditions starting at 40 °C for 2 min, increasing to 150 °C at a rate of 25 °C min<sup>-1</sup> and reaching a final temperature of 250 °C at a rate of 50 °C min<sup>-1</sup>. Injector and detector temperatures were 250 °C. Liquid samples were analysed by extracting a volume of 4.50 cm<sup>3</sup> with 3.00 cm<sup>3</sup> of diethylether (as extraction solvent), and vortexing that volume for 1 min at maximum speed. The diethylether layer was analysed by split injection of 1.0E-06 dm<sup>3</sup> in the gas chromatograph. The soil analysis was carried out by transferring a known amount of soil to an extraction tube into which a known volume of diethylether was added (1–10 cm<sup>3</sup>). The diethylether layer was analysed by split injection of 1.0E-06 dm<sup>3</sup> in the gas chromatograph. Gas samples were directly analysed by split injection of a known volume (0.1–0.5 cm<sup>3</sup>) in the gas chromatograph. Toluene concentrations were determined from previously prepared calibration curves.

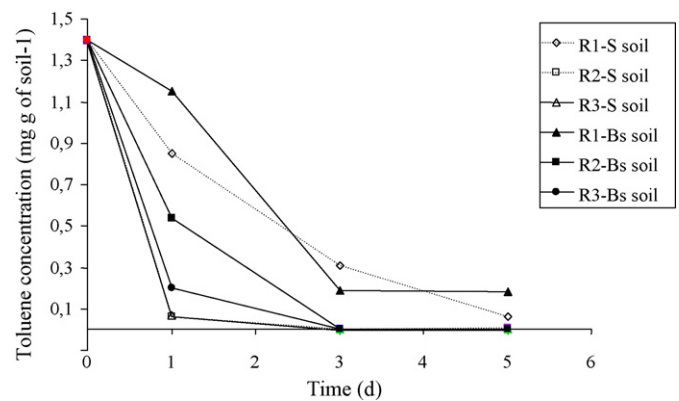


Fig. 2. Experiment 1: Toluene concentration remaining in soil at the surface (S) and below surface (Bs) (4 cm) in reactors R1, R2 and R3. (—◇—) R1: surface soil; (—□—) R2: surface soil; (—△—) R3: surface soil; (—▲—) R1: below surface soil; (—■—) R2: below surface soil; (—●—) R3: below surface soil.

## 3. Results and discussion

### 3.1. Extent of soil remediation at different toluene concentrations

After the first week there was an evidence of toluene degradation in batch cultures, noted by increases in OD, indicating the existence of toluene degraders in the soil samples and from this the possibility of biodegradation in the soil reactors.

In Experiment 1, with an initial toluene concentration of 2 mg g soil<sup>-1</sup>, toluene depletion from the soil was evident, particularly in the ventilated reactors, R2 and BVR, which presented a similar toluene removal profile. In these reactors toluene depletion from the surface soil was nearly complete within the first day (6.5E-02 and 6.8E-02 mg g soil<sup>-1</sup> remaining in the soil, respectively, corresponding to 4.6% and 4.9% of the initial toluene concentration), contrasting with reactor R1, which only by the end of the fifth day presented a toluene level, 6.4E-02 mg g soil<sup>-1</sup>, similar to that of R2 and BVR (Fig. 2).

In Experiment 2, with an initial toluene level of 14 mg g soil<sup>-1</sup>, a similar profile of toluene concentration in soil was observed. Both reactors R2 and BVR, after the first day of treatment, presented a toluene level in the surface soil (4.8% and 5.5%, of the initial toluene concentration, respectively) slightly lower when compared with that of R1, which presented 8% of the initial toluene concentration. After 5 days, toluene concentration was much lower in reactors R2 and BVR (3.2E-03 and 3.1E-03 mg g soil<sup>-1</sup>, respectively) than that observed in R1 (2.38 mg g soil<sup>-1</sup>; Fig. 3).

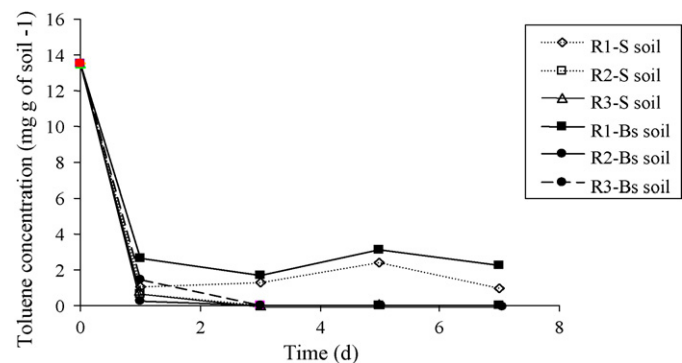
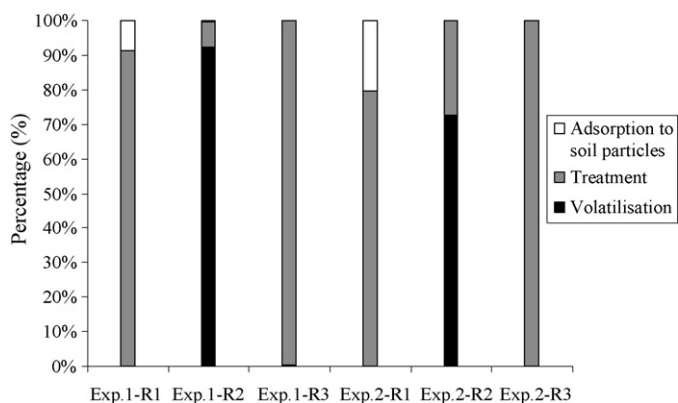


Fig. 3. Experiment 2: Toluene concentration remaining in soil at the surface (S) and below surface (Bs) (4 cm) in reactors R1, R2 and R3. (—◇—) R1: surface soil; (—□—) R2: surface soil; (—△—) R3: surface soil; (—▲—) R1: below surface soil; (—■—) R2: below surface soil; (—●—) R3: below surface soil.



**Fig. 4.** Toluene mass balance in bioremediation reactors in Exp. 1 to Exp. 2 after 5 days. (□) Adsorption to soil particles; (■) treatment; (■) volatilisation.

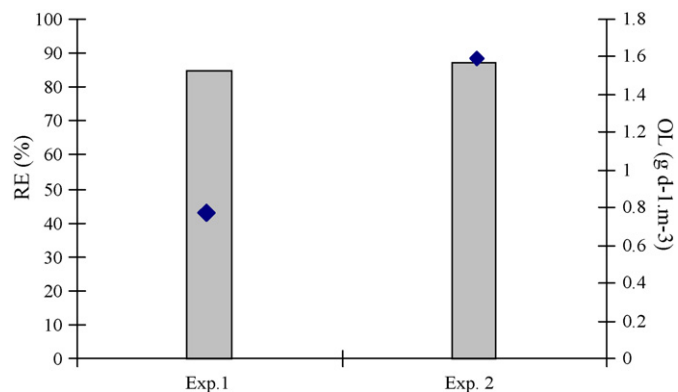
### 3.2. Global toluene mass balance in remediation reactors R1, R2 and R3

The global toluene mass balance for the non-ventilated control reactor (R1), for the ventilated control reactor (R2) and for the combination of biotechnologies (R3), was carried out from data obtained in a period of *ca.* 5 days, for each scenario of toluene contamination load (2 and 14 mg g soil<sup>-1</sup>). The mass balance carried out considered three different fractions: the toluene adsorbed to the soil particles, and therefore remaining in the soil; the toluene removed from soil through volatilisation but not treated; and finally, the toluene fraction that was removed from soil but treated, either by the BF and/or by bioremediation in the soil (Fig. 4).

The mass balance indicated that R1 presented the highest levels of toluene adsorption to soil particles and that toluene removal might have occurred mainly due to biodegradation process, carried out by the microbiota present in soil (in the range of 80–91%) (Fig. 4).

Prenafeta-Boldú et al. [34] reported bioremediation of soil contaminated with BTEX hydrocarbons by indigenous bacteria and the fungus *Cladophialophora* sp. strain T1.

In the present study, the constant air flow rate of 0.13 dm<sup>3</sup> min<sup>-1</sup> (2.5E–04 dm<sup>3</sup> min<sup>-1</sup> g soil<sup>-1</sup>) supplied to R2 significantly contributed to reducing the time required for soil remediation, with a high toluene removal being achieved within the first day of treatment (in a range of *ca.* 82–95%, in surface soil), contrasting with R1 (in a range of *ca.* 6–92%, in surface soil) (Figs. 2 and 3). Due to both volatilisation and biological treatment, reactor R2 removed *ca.* 99% of toluene after 5 days (Fig. 4). As expected for a SVE regime [18], the high air flow rate applied to R2 enhanced the volatilisation process (*ca.* 92% and 73%, in Experiments 1 and 2, respectively), compared



**Fig. 5.** Toluene removal efficiencies and organic load values in the biotrickling filter reactor in Exp. 1 to Exp. 2. RE (filled bars); OL (◆).

**Table 1**

The outlet gas toluene concentrations from biotrickling filter (BF).

Days	Toluene concentration (mg dm <sup>-3</sup> )	
	Exp. 1	Exp. 2
0	–	–
1	8.8E–03	4.1E–03
2	2.5E–03	4.3E–04
3	2.5E–03	3.1E–04
4	3.0E–04	1.1E–02
5	3.1E–04	6.4E–03
6	4.9E–03	6.4E–03
7	1.2E–03	6.3E–03

to biodegradation (*ca.* 7% and 27% in Experiments 1 and 2, respectively), which was not a significant contributor to toluene removal (*ca.* 7% and 27% in Experiments 1 and 2, respectively) (Fig. 4). This contrasts with the situation in reactor R1 where toluene volatilisation was very low and biodegradation by the microbial flora was the dominant mode of toluene removal. In R2, even though the volatilisation process resulted in an efficient soil remediation, it led to a significant non-treated toluene fraction being emitted to the atmosphere (Fig. 4).

The combination of biotechnologies was investigated in reactor R3, which presented the most effective arrangement to remediate soil contaminated with light hydrocarbons, with toluene concentrations ranging from 2 to 14 mg g soil<sup>-1</sup>, and to treat the toluene gas emissions that emerged from the stripping mode AIBV (Fig. 4). For all the experiments set up in R3, by the end of the day 5, *ca.* 99% of the contaminant was removed from soil by volatilisation and biodegradation. Similar values were observed in reactor R2, which was exposed to the same air flow rate (Figs. 2 and 3). As expected, similarly to R2, the fraction of toluene removed from soil through volatilisation needed to be treated.

The main difference between reactor R2 and R3 was that the percentage of treated toluene was higher (*ca.* 99%, in both experiments) in R3 than in R2 (*ca.* 7% and 27% in Experiments 1 and 2, respectively), as a consequence of the additional treatment of the fraction of the off-gas, which resulted from the contaminant removal through volatilisation, by the BF (Fig. 4). Fig. 5 shows that the BF presented, on average, for all experiments, a high RE (*ca.* 86%), with OL ranging from *ca.* 0.8 to 1.6 g d<sup>-1</sup> m<sup>-3</sup> reactor (0.033–0.083 g h<sup>-1</sup> m<sup>-3</sup> reactor) (Fig. 5, Table 1). BFs have been successfully used for toluene treatment. Cox and Deshusses [31] observed, at a volumetric load of 170 g m<sup>-3</sup> h<sup>-1</sup>, maximum elimination capacities of 70 g toluene m<sup>-3</sup> h<sup>-1</sup>. Chou and Wu [32] indicated that, for a test period of 121 days, with no excess biomass removal, toluene removal efficiencies higher than 90% were obtained with an OL of 30 g m<sup>3</sup> h. These results shows the potential of the combination of these technologies when higher loads are to be treated as it has been proven that both AIBV with SVE regime and BF reactor configurations are adequate for the treatment of such soil pollution.

## 4. Conclusions

The use of AIBV operated under a soil striping regime mode, has been shown to be an efficient process for the treatment of soil contaminated with toluene as it promoted high removal (up to 99%) of the contaminant (from initial contamination of *ca.* 2 and 14 mg g soil<sup>-1</sup>). However, in such a system a high percentage of the contaminant VOC was transferred from the soil to the atmosphere. Nevertheless, this disadvantage can be overcome by combining this technology with a BF for the treatment of gaseous emissions. Removal efficiencies in this reactor reached an average of *ca.* 86%, however there is a need to test this combination of biotechnologies when exposed to higher OLs.

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## Glossary

- AIBV: air-injection bioventing  
 BF: biotrickling filter  
 BVR: bioventing reactor  
 BV: bioventing  
 OL: organic load  
 RE: removal efficiency  
 SVE: soil vapour extraction  
 VOC: volatile organic compounds