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Biofiltration of High Loads of Ethyl Acetate in the Presence of Toluene

Marc Deshusses and Camdon T. Johnson
Department of Chemical and Environmental Engineering, University of California, Riverside, California

Gero Leson
Gero Leson Environmental Consulting, Berkeley, California

ABSTRACT

To date, biofilters have been used primarily to control dilute, usually odorous, off-gases with relatively low volatile organic compound (VOC) concentrations (<1 g m^{-3}) and VOC loads (<50 g m^{-3} hr^{-1}). Recently, however, U.S. industry has shown an interest in applying biofilters to higher concentrations of VOCs and hazardous air pollutants (HAPs).

In this study, the behavior of biofilters under high loads of binary VOC mixtures was studied. Two bench-scale biofilters were operated using a commercially available medium and a mixture of wood chips and compost. Both were exposed to varying mixtures of ethyl acetate and toluene. Concentration profiles and the corresponding removal efficiencies as a function of VOC loading were determined through frequent grab-sampling and GC analysis. Biofilter response to two frequently encountered operating problems—media dry-out and operating temperatures exceeding 40 °C—was also evaluated under controlled conditions. Microbial populations were also monitored to confirm the presence of organisms capable of degrading both major off-gas constituents. The results demonstrated several characteristics of biofilters operating under high VOC load conditions.

- Maximum elimination capacities for ethyl acetate were typically in the range of 200 g m^{-3} hr^{-1}.
- Despite the presence of toluene degraders, the removal of toluene was inhibited by high loads of ethyl acetate.
- Several byproducts, particularly ethanol, were formed.
- Short-term dry-out and temperature excursions resulted in reduced performance.

IMPLICATIONS

To date, biofilters have been used primarily to control dilute, usually odorous, off-gases with relatively low VOC concentrations. Yet there is a growing interest in expanding to higher concentrations the range of suitable applications for biological waste air treatment. However, this type of application has a high rate of system failure. Impediments for this application were reported in two recent case studies in which the treatment of high loads of ethanol from a foundry and a bakery, respectively, resulted in reduced percentage removal of the contaminants, formation of odorous acetic acid, and problems maintaining the proper moisture content because of the exothermic nature of the biodegradation process. The present study reports bench-scale operation of biofilters under high VOC loading conditions of mixed pollutant and the factors that lead to these adverse operating states

INTRODUCTION

It is generally thought that biofilters are suitable only for the treatment of dilute emissions of odors, volatile organic compounds (VOCs) and hazardous air pollutants (HAPs). The large majority of biofilters installed to date treat off-gases containing organic carbon compounds at concentrations of less than 0.5 g m^{-3}. Corresponding VOC loads and elimination capacities rarely exceed 50 grams of organic carbon per cubic meter of biofilter material per hour (g m^{-3} hr). Recently, however, industrial users, particularly in the United States, and vendors of biofilters have been attracted by the concept of treating higher concentrations and loadings of VOCs in biofilters to expand their range of applicability. The economic competitiveness of thermal and catalytic incineration systems, frequently the main competing technologies for complete pollutant destruction, has been a major obstacle to the biofilter treatment of higher concentrations of VOCs. However, additional fuel is needed for thermal or catalytic incineration of air streams containing less than about 20 to 50 g VOC m^{-3}, increasing both the operating costs and dependency on fossil fuels. Further, incineration generates nitrogen oxides, which require expensive off-gas treatment.

On the other hand, biological air pollution control does not require additional fuel and, under optimum conditions, complete degradation to carbon dioxide is achieved without the formation of secondary pollutants. Unfortunately, several technical obstacles have been reported when biofilters have been applied to highly polluted air streams. They include an elimination capacity that is limited to less than 200 g m^{-3} hr^{-1} for most VOCs,[1,2] the formation of potentially odorous or hazardous degradation byproducts,[3-5] and the increasingly difficult task of maintaining the proper moisture content of the biofilter medium.[6] Previous laboratory and field work had also indicated that biofilters may experience additional performance problems when treating mixtures of VOCs at high loads.[7-10]

The goal of this study was to investigate and quantify, under controlled conditions, several of the phenomena encountered in full-scale biofilters when treating high loads of two commonly used industrial solvents: ethyl acetate and toluene. Objectives for the study included establishing maximum removal rates for two commonly used biofilter media, assessing the inhibition of toluene removal in the presence of ethyl acetate, and evaluating the formation of byproducts. Simulations of two potentially catastrophic events—dry-out and temperature excursions in the media—were also conducted.

MATERIALS AND METHODS

Two bench-scale biofilter columns treated mixtures of ethyl acetate and toluene at a fixed mass ratio (3:1) and empty bed residence time (EBRT = 3 minutes) but varying concentrations of total VOC. Biofilter A was filled with an 80/20-by-volume mixture (medium A) of wood chips and compost, respectively. Biofilter B was filled with a commercially available medium (medium B) mixed from compost and polystyrene spheres (Bioton). Calcium carbonate (limestone) for pH buffering (10% based on dry weight) and essentially insoluble inorganic nutrients (0.4% N, 0.4% P_2O_5, 0.2% K_2O based on dry weight) from bone and blood meal were added to the wood chips medium mixture. To accelerate the acclimation process, both biofilters were inoculated with mixed bacterial cultures grown on ethyl acetate and/or toluene. All experiments described herein were performed over a two-month period.

The biofilters were made of clear PVC pipes, with an internal diameter of 15.2 cm. The biofilter bed was split into three 50-cm sections. The top segment was referred to as the first segment and the bottom segment was referred to as the third segment (see Figure 1). This segmented setup made it easier to collect gas samples at intermediary heights.

Biofilters were operated in a downflow mode. Using a metering pump (FMI, Inc., NJ), ethyl acetate and toluene were injected at specified rates into humidified air to produce the synthetic waste air stream. The air flow rate was controlled (mass flow controller, Porter Instruments, PA) to keep a constant empty-bed residence time of 3 minutes; the total VOC concentration ranged from 4 to 12.4 g m^{-3}.

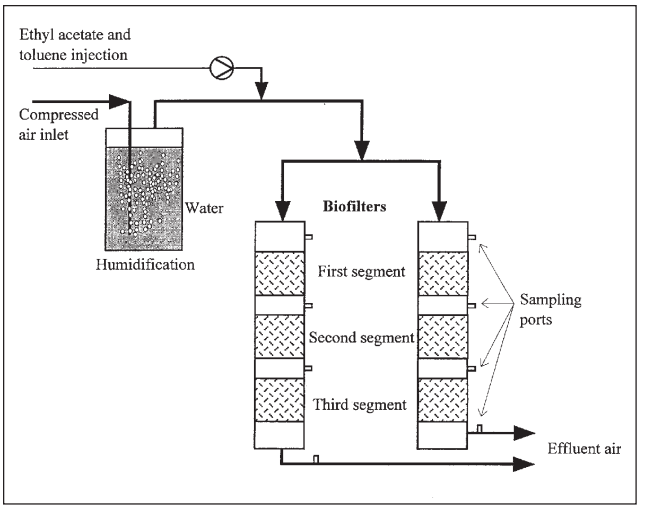

Figure 1. Schematic of the experimental setup.

Gas samples were collected and automatically injected into a Hewlett Packard 5890 Series II gas chromatograph fitted with a 30-m Supelcowax 10 column (0.53 mm, 1 µm film, Supelco, Bellefonte, PA), equipped with an flame ionization detector (FID) for the detection of ethyl acetate, toluene, and possible metabolites. During steady-state conditions, about one month after start-up, the population of heterotrophs on the biofilter media was estimated by plating serial dilutions of medium aqueous suspension on plate count agar (Difco, Detroit, MI). The relative numbers of ethyl acetate or toluene degraders were tracked in a similar manner on basal salt medium plates with ethyl acetate or toluene supplemented via the gas phase. Colony-forming unit (CFU) counts were made after 24 to 48 hours incubation at room temperature on the plate count agar, and 2 to 5 days on basal salt medium/VOC plates.

RESULTS AND DISCUSSION

Inhibition of Toluene Removal

Concentration profiles of ethyl acetate, toluene and the byproduct ethanol at VOC inlet concentrations of 6.9 and 12.4 g m^{-3} using medium A are shown in Figures 2 and 3, respectively. Under both VOC inlet conditions, high elimination capacities for ethyl acetate, up to 180 g m^{-3} hr^{-1}, were noted in the first two segments. Little or no toluene removal occurred throughout the biofilter with the 12.4 g m^{-3} VOC inlet concentration, or in the first two segments at the 6.9 g m^{-3} VOC inlet concentration. Only in the third segment of the latter biofilter, where concentrations of ethyl acetate had fallen off to less than 0.5 g m^{-3}, did toluene removal commence. In this latter segment,

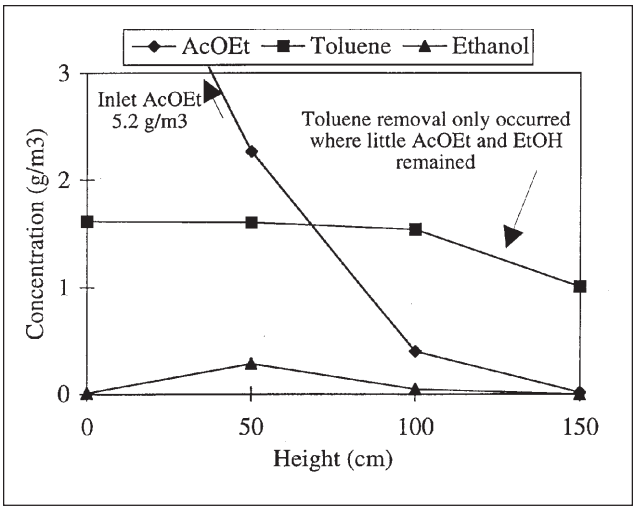

Figure 2. Typical concentration profiles in the biofilter reactor. Here, the medium A biofilter is shown with a total inlet VOC concentration of 6.9 g m^{-3}.

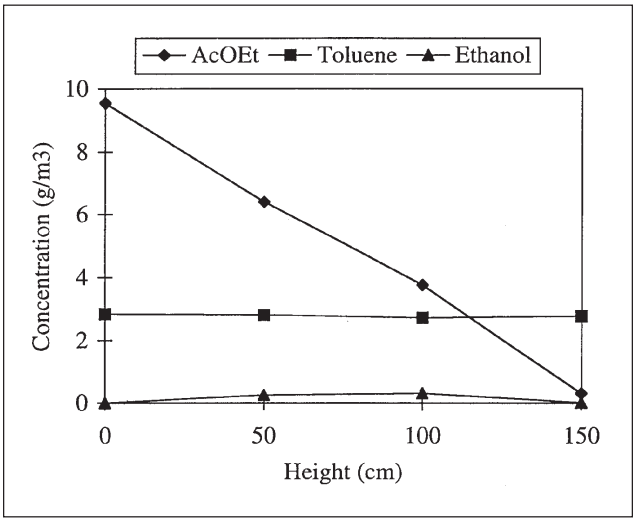

Figure 3. Concentration profiles in the medium A biofilter. Total inlet VOC concentration was 12.4 g m^{-3}.

the observed elimination capacity for toluene was about 20 g m^{-3} hr^{-1}, comparable to values reported for other, compost-based, biofilter systems.[7]

Consistent with previously reported data,[11] the concentration profiles illustrate that concentrations of ethyl acetate in excess of 0.5 to 2 g m^{-3}, apparently inhibit the concurrent removal of toluene. To assess whether this lack of toluene removal is due to the absence of toluene-degrading microorganisms, microorganisms extracted from the first segments of each column were plated onto purified agar plates and exposed to ethyl acetate and toluene vapors, respectively, as the sole source of organic carbon. Results are summarized in Table 1. Even in the non-toluene-degrading first segment of the biofilter, toluene-degrading microorganisms were present in sufficiently high quantities (10^8–10^9 CFU/gram of moist medium) to be able to remove the toluene transferred from the gas phase into the biofilm. Further testing indicated that all of the isolated strains of toluene degraders were also capable of degrading ethyl acetate if supplied as the sole carbon source. However, these facultative toluene degraders were outnumbered by the microorganisms capable of degrading only ethyl acetate.

Table 1. Log of the total number of heterotrophic and toluene-degrading microorganisms per gram of damp medium. Samples were taken at the top of the first segment.

	Total Count	Toluene Degraders
	Log N	Log N (% of total)
Wood chips biofilter	9.36	8.45 (12.3%)
Bioton biofilter	9.07	8.89 (66.0%)

These microbiological results indicate that the lack of toluene removal in segments 1 and 2 was not due to the absence of suitable microorganisms but rather to some inhibitory mechanism preventing them from metabolizing toluene in the presence of high ethyl acetate concentrations. At this time, the exact mechanisms leading to the inhibition of the toluene metabolism remain unknown. Similar inhibitions have been reported for simultaneous treatment of MEK and MIBK in biofilters,[12] and butanol and toluene in a biotrickling filter.[10] However, inhibition was not expected a priori in this case, since ethyl acetate and toluene are known to be degraded by very different pathways. One potential reason for inhibition of toluene degradation is failure of the toluene degraders to compete for oxygen, which limits total VOC removal at high concentrations.[7] It is also conceivable that other mechanisms exist by which the presence of ethyl acetate or the accumulation of degradation byproducts may inhibit the expression of the inducible enzymes involved in the initial breakdown of toluene.[13] Further research is required to determine the actual mechanisms of inhibition in this case and whether they can be controlled.

Due to the minimal removal of toluene in the concentration range tested in our experiments, the following discussion will focus on the removal of ethyl acetate in both biofilters.

Elimination Capacity

Bulk ethyl acetate elimination capacities by mediums A and B were measured at various times during the project as a function of ethyl acetate influent concentration and load. Bulk loads, *L*, and elimination capacities, *EC*, for individual segments and for the entire reactor, singly, were calculated using the empty bed residence time in minutes (*EBRT*) as follows:

$$L = \frac{c_{in} \cdot 60}{EBRT} \quad (1)$$

$$EC = \frac{(c_{in} - c_{out}) \cdot 60}{EBRT} \quad (2)$$

where L and EC are measured in grams of pollutant per cubic meter of medium per hour (g m^{-3} hr^{-1}), inlet and outlet concentrations, c_{in} and c_{out} in g m^{-3}. The *EBRT* ranged from 1 minute for one segment to 3 minutes for the entire column. Thus, for a given inlet concentration of ethyl acetate, each concentration profile provided 3 *EC* versus *L* data points, namely for the first, the first two, and all three segments.

At the highest inlet concentration (~10 g m^{-3}), bulk elimination capacities for segment 1, segments 1 and 2, and all 3 segments of biofilter A were similar, 180 to 200 g m^{-3} hr^{-1} (see Figure 4). Because the bulk *EC* for a biofilter or biofilter segment is proportional to the average slope of the respective concentration profile, this corresponds to a more or less linear concentration profile across the entire biofilter. Such behavior has been modeled by Ottengraf[7] assuming full penetration of the active biofilm by pollutants at high concentrations and zero-order removal kinetics. This case is referred to as "reaction limited." The corresponding maximum elimination rate in the film may be limited by the limited transfer of oxygen into the biofilm, or any other event in the biodegradation scheme.

At the high inlet concentrations for ethyl acetate, more than 5 g m^{-3}, *EC* did not vary with inlet concentration. Nonvariability indicates that the biofiltration of ethyl acetate followed overall zero-order kinetics in this concentration range, in agreement with the "reaction limited" scenario.

In comparing the data shown in Figure 4 with the concentrations profiles reported in Figures 2 and 3, it can be seen that the slope of the concentration profile and the corresponding *EC* decrease as inlet concentrations to segments 2 or 3 fall below 2 to 3 g m^{-3}. In Ottengraf's model, this decline is due to "diffusion limitation" in the lower segments of the biofilter. There, the sufficiently low vapor-phase concentrations of ethyl acetate prevent full penetration of the biofilm, thereby reducing its per volume (of medium) elimination capacity.

The measured maximum elimination capacities agree closely with those achieved by similar bark-compost mixtures. Windsperger et al.[14] reported a maximum *EC* of about 175 g m^{-3} hr^{-1} for pure ethyl acetate at inlet concentrations of ~3 g m^{-3}. For comparison, representative data points taken from their results have been included in Figure 4. Similar to the present study, Windsperger et al. also indicated a gradual reduction in *EC* as inlet concentrations were lowered from 3 to 1.5 g m^{-3}, with a corresponding reduction in residence time at a constant pollutant loading.

In comparison, the removal of ethyl acetate by medium B (Bioton) is summarized in Figure 5. A maximum *EC* of ~300 g m^{-3} hr^{-1} was achieved at an inlet concentration of 8.4 g m^{-3}, compared to less than 200 for medium A (compost-wood chips). Improved performance was likely due to the high porosity and larger active surface of the medium. The greater variability in the performance of this medium, as seen in Figure 5, was primarily a result of some dry-out experienced, particularly in the first segment, when applying the highest inlet concentration (9.4 g m^{-3} ethyl acetate and 3.2 g m^{-3} toluene).

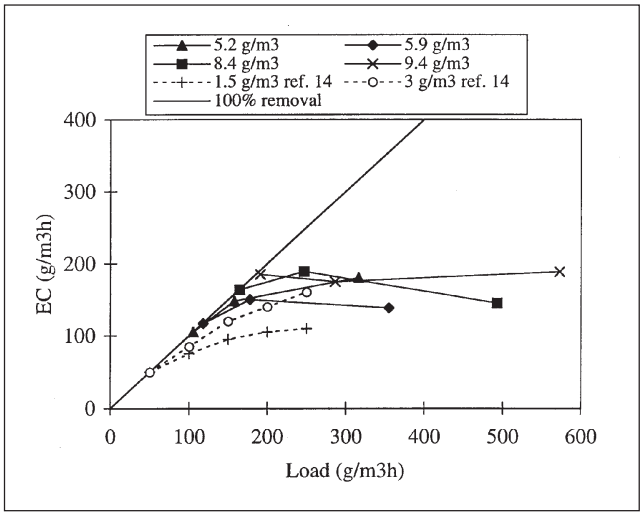

Figure 4. Ethyl acetate elimination capacity versus ethyl acetate loading for the wood chips biofilter in the presence of toluene. The legend shows the inlet ethyl acetate concentration. The dashed line represents data for pure ethyl acetate obtained by Windsperger et al.[14] at two different inlet concentrations.

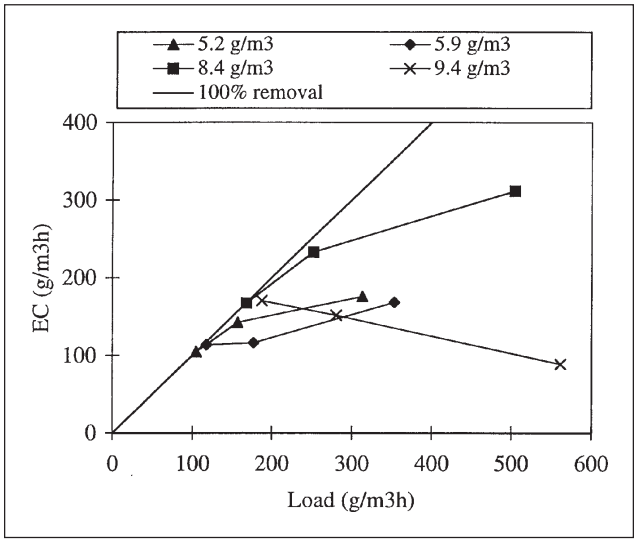

Figure 5. Ethyl acetate elimination capacity versus ethyl acetate loading for the Bioton biofilter in the presence of toluene. The legend shows the inlet ethyl acetate concentration.

Based on the data shown in Figures 4 and 5, it can be seen that both media achieved nearly complete removal of ethyl acetate at VOC loads up to 180 g m^{-3} hr^{-1}. However, comparison of these data with those of Windsperger et al. indicates that removal efficiency is not merely a function of loading rate, but also of residence time. For the same loading rate, reduced percentage removals occurred when the inlet concentration was lower and the residence time was decreased (as the result of higher air flow rate). Considering that large-scale biofilters are usually not economical if designed for residence times exceeding one minute, the overall percent removal will decrease as inlet loads of ethyl acetate exceed 50 g m^{-3} hr^{-1}.

Production of Ethanol and Other Degradation Byproducts

Another phenomenon commonly encountered in biofilters, but not widely investigated when treating high loads of polar VOCs, is the formation of degradation intermediates.[4,5,15] Ethanol, an intermediate of the biodegradation of ethyl acetate, was routinely formed in biofilter segment 1 and degraded in segments 2 and 3 (see Figure 6). Maximum concentrations of ethanol were 3 to 5% of the respective inlet concentration of ethyl acetate. At the highest inlet concentration, the rate of formation of ethanol in segment 2 exceeded its rate of degradation. Also, because there was a high degree of ethyl acetate removal in the first two segments and the amount of ethanol generated was low, nearly complete removal of ethanol was achieved by the end of the last segment in the biofilter.

In addition to ethanol, the formation of which had been expected based on previous field experience, several other degradation intermediates of ethyl acetate were detected by gas chromatography (GC), albeit at much lower concentrations. Their GC retention times did not correspond to those of other potential degradation intermediates previously found, such as acetic acid and acetaldehyde. However, no further attempt at their identification was made, although one metabolite with a very short retention time on the GC may have been methane. The concentration of this unknown intermediate in the effluent air was approximately 30 to 80 mg m^{-3}, and it increased over the duration of the tests. Methane formation would indicate anaerobic activity within the biofilter. Whether such activity is related to pollutant elimination or to biodegradation of the biofilter medium is unknown.

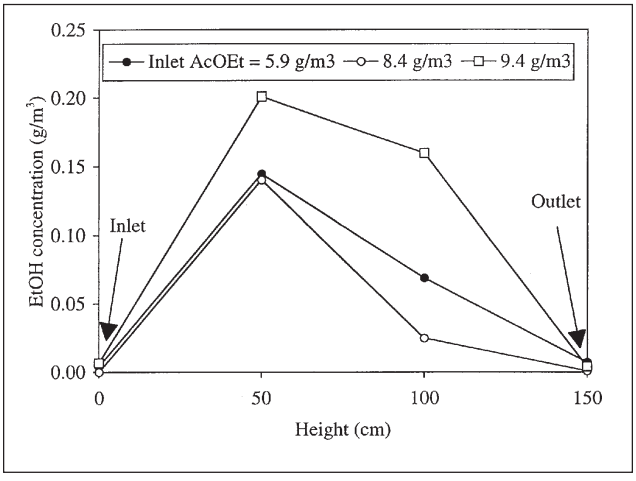

Figure 6. Ethanol concentration profiles at different VOC loadings for the Bioton biofilter.

These findings on the formation of intermediates during biodegradation of ethyl acetate are analogous to those from previous bench-scale research on the biofiltration of off-gases containing high concentrations of ethanol in which the formation and potential release of intermediates, notably acetic acid, acetaldehyde, and ethyl acetate was observed.[4] While maximum concentrations of these intermediates were found to be low, typically less than 500 mg m^{-3}, only acetic acid, with its low odor threshold of <0.5 mg m^{-3}, had the potential to generate an odorous off-gases. This problem was borne out in a study of two field biofilters treating ethanol loads that frequently exceeded 150 g m^{-3} hr^{-1}, in which odor problems caused by the release of acetic acid were experienced.[3]

While the ethanol produced during our tests from the degradation of ethyl acetate is not of concern because of its higher odor threshold (~30 mg m^{-3}) and its low toxicity, it is known to react with certain acrylates to form highly odorous reaction products. Thus, the formation of intermediates should be carefully assessed when attempting to treat high loads of polar VOCs in a biofilter.

Temperature Increase

Due to the exothermic nature of the biofiltration process, the removal of high concentrations of VOCs is accompanied by an increase in off-gas temperature. Temperature increases of 5 to 10 °C have been observed in several full-scale biofilters.[6] Because of its much higher surface-to-volume ratio, such effects are generally less pronounced in a bench-scale biofilter. However, because of the high loads at which our tests were conducted, temperature increases of as much as 20 °C were observed. A typical radial temperature profile taken in segment 2 of the wood chips biofilter is shown in Figure 7. This temperature increase was due to an internal process that resulted in some of the thermal energy gained from the biodegradation of ethyl acetate being lost to the environment. Further, because of the high radial temperature gradient, drying out of the media may occur at the center of the bed, whereas the media near the reactor wall may remain at the proper moisture content and look perfectly normal. This hypothesis was confirmed by observation of the media after the biofilters were open and has also been observed by other researchers.[16]

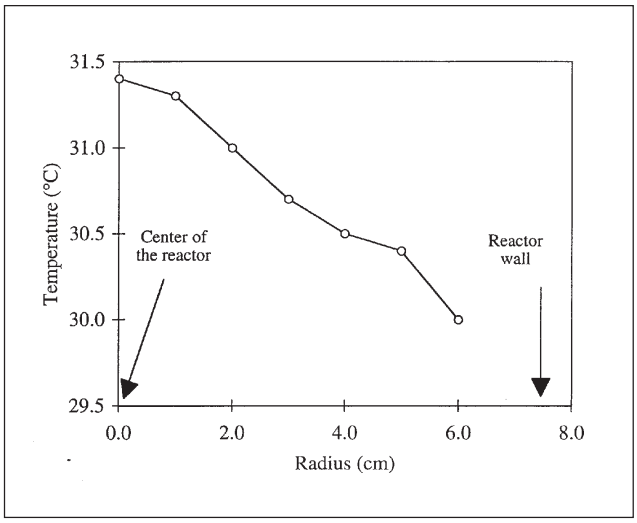

Figure 7. Radial temperature profile in the middle of the second segment of the medium A biofilter.

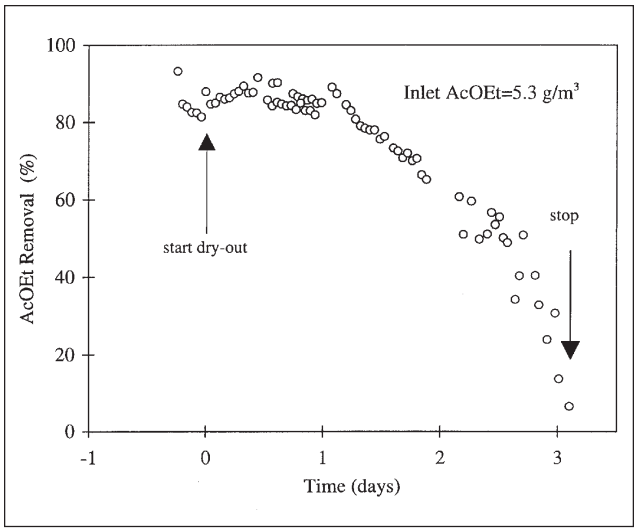

Figure 8. Removal of ethyl acetate after the first segment of the medium A biofilter during the dry-out experiment.

Medium moisture content and temperature are two of the most crucial operating parameters of a biofilter. For a "spot assessment" of how rapid changes in these parameters may affect performance of a biofilter receiving high organic loads, the bench-scale reactors were, at the end of the project, subjected to sequential dry out and overheating tests.

Dry-Out Test

The medium from the first segment of each biofilter was removed and homogenized. Following gravimetric determination of its bulk moisture content, the medium was reinstalled. Following reassembly, total VOC concentrations were kept at 7.0 g m^{-3}. Subsequent bypassing of the prehumidifier produced a dry off-gas (25 °C, 15% RH), which resulted in a gradual removal of moisture from the medium, starting with the first segment of the biofilter. The columns were operated until performance monitoring of the first segment indicated that biological activity and pollutant removal had ceased. The medium was then removed again, and the final moisture content was determined.

The loss of ethyl acetate removal in biofilter A is shown in Figure 8 as its bulk moisture content drops from 65 to 55% (Figure 9). The initial reduction in moisture content did not immediately affect performance. However, further dry-out caused rapid and nearly complete loss of biological activity and ethyl acetate reduction after a total of 3 days. These results confirm the detrimental impact of medium dry-out on performance that can be caused, for example, by the failure of the prehumidification system.

Under conditions of rapid dry-out, as in this experiment, the bulk moisture content is probably not a good measure of moisture available to microorganisms. Particularly in a medium containing large proportions of wood chips, much of

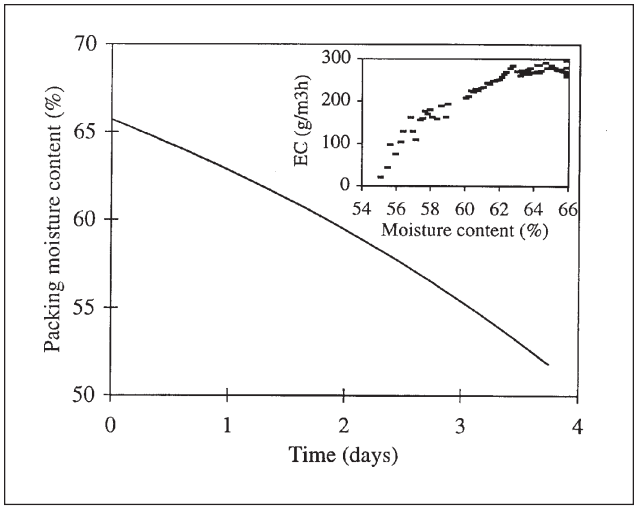

Figure 9. Moisture content for the medium A biofilter calculated assuming a constant drying rate. The inset shows the elimination capacity of ethyl acetate versus moisture content of the packing.

the medium moisture is stored in the chips and removed only gradually. The rate of moisture loss in the active biofilm covering the surface of chips and particles will be higher than suggested by Figure 9. Thus, the medium used in this experiment may well perform effectively at a bulk moisture content of 55%, as long as it is in equilibrium with respect to moisture distribution. Rather than indicating an "optimum moisture" content of medium A, this dry-out test merely demonstrates the detrimental impact of rapid changes in the biofilm's moisture content.

Elevated Temperature

Following the dry-out test, the medium in the first segment was re-wetted, inoculated with a small amount of fresh, hot compost extract likely to contain thermophilic and thermo-tolerant microorganisms, and reinstalled. The

columns were subsequently kept in a heated enclosure and the medium temperature, which had been kept between 30 and 37 °C during the previous experiments, was raised to 45 to 50 °C (see upper graph Figure 10). Ethyl acetate concentrations in the inlet of biofilter A and after each of its three segments, following the rise in temperature, are presented in Figure 10. Segments 1 and 2 lost biological activity and ethyl acetate removal within 3 days. Yet, segment 3 continued to achieve considerable removal of ethyl acetate (>100 g m^{-3} hr^{-1}) at temperatures of 50 °C, that is, above the maximum temperature tolerated by most mesophilic organisms (42 °C). Subsequent medium removal revealed that the decrease of ethyl acetate reduction through sections 1 and 2 was most probably caused by extensive medium dry-out rather than elevated temperature. The continued performance in segment 3 indicated that the removal of ethyl acetate in a biofilter may be sustained at temperatures of 50 °C, as long as thermophilic or thermo-tolerant microorganisms are present. However, the long-term feasibility of this approach, particularly when using organic media which may be subject to rapid degradation, requires further investigation.

CONCLUSIONS

The findings from this study highlight several major problems encountered when treating high loads of VOC or HAP mixtures. HAPs, such as toluene, which are targeted by regulatory requirements, may not be removed effectively in the presence of high loads of less volatile, polar VOCs, such as ethyl acetate. Bulk elimination capacities of ~200 g m^{-3} hr^{-1} may be achieved by a medium that is porous and contains sufficient moisture and nutrients. However, when attempting to remove such high organic loads consistently in a biofilter, the potential for causing temperature increase, rapid moisture loss, and the release of undesirable byproducts must be evaluated in a pilot test.

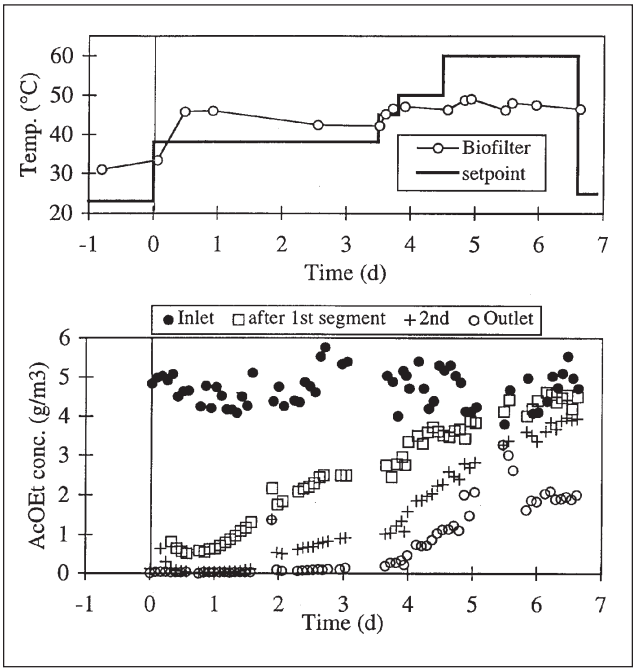

Figure 10. Ethyl acetate concentration in the inlet, after the first segment, the second segment, and in the outlet air stream during the tests at elevated temperatures. The upper graph shows the temperature at the center of the second segment, and the temperature in the enclosure built around the biofilters.

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About The Authors

Dr. Marc A. Deshusses is assistant professor of Chemical and Environmental Engineering at the University of California, Riverside. At the time of this study Camdon T. Johnson was an undergraduate student in the Chemical and Environmental Engineering Department at the University of California, Riverside, and is now a technical staff member of Treasure Chest Advertising. Dr. Gero Leson is an independent environmental consultant specializing in biological air pollution control technology. Correspondence should be addressed to Dr. Deshusses (mdeshuss@engr.ucr.edu).