ODOR AND VOC TREATMENT BY BIOTRICKLING FILTERS: PILOT SCALE STUDIES AT THE HYPERION TREATMENT PLANT

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ABSTRACT

A pilot-scale biotrickling filter was installed at the Hyperion Treatment Plant in Los Angeles to study H₂S/odor and VOC removal from the Headworks' waste air. The performance of the reactor was continuously monitored over a period of 10 months. At an average empty bed gas residence time of 24 s, 10-50 ppm H₂S was consistently removed at greater than 98% efficiency, corresponding to an average volumetric H₂S elimination capacity of 5.2 g/m³.h. Concentration profiles over the height of the reactor indicated near complete removal in the first section of the reactor and that elimination capacities up to 30 g/m³.h could be obtained. The odor reduction was 98%, which corresponded to the efficiency of removal of H₂S as the major pollutant. VOCs were present at concentrations up to 150 ppb. Low but significant removal of toluene and benzene was observed when the biotrickling filter was operated with pH-control to neutralize sulfuric acid production from H₂S oxidation. Xylenes and chlorinated VOCs were not removed, irrespective of experimental conditions in the reactor. The results lead to the conclusion that VOC removal is the limiting process in biotrickling filters for the simultaneous removal of H₂S and VOCs at POTWs.

KEYWORDS

Biotrickling filter, biological waste air treatment, hydrogen sulfide, odor, VOCs.

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INTRODUCTION

The University of California at Riverside (UCR), the University of California at Davis (UCD) and the Hyperion Treatment Plant (HTP) in Los Angeles have been testing pilot-scale biotrickling filters and biofilters as alternatives to chemical scrubbers for odor treatment and removal of Volatile Organic Compounds (VOCs) from odorous waste gases. The waste gas from the Headworks at HTP (100,000 cfm) contains 10-50 ppm H₂S as the principal odor-causing agent as well as a broad variety of chlorinated and non-chlorinated VOCs in concentrations ranging from 0 to 150 ppb. The current use of chemical scrubbers proves satisfactory with respect to odor/H₂S treatment. **However**, they fail to remove the VOCs. In some cases chemical scrubbers may even generate VOCs, which are subsequently emitted into the atmosphere (Witherspoon et al., 1995). Other drawbacks of chemical scrubbers include a high consumption rate of chemicals (hypochlorite, caustic soda) and relatively high operational costs.

Various pilot-studies with biotrickling filters and biofilters at POTWs have demonstrated that biological removal of H₂S and odor from waste gases is efficient and cost-effective (Iranpour et al., 2001). Additional reduction of VOC emissions is desirable, especially in the Southern California Air Basin where New Source Review Regulations for toxics have been effective since 1990. The upcoming regulations include the establishment of Maximum Achievable Control Technology standards as mandated under the Federal Clean Air Act Amendments of 1990.

Since biotrickling filters and biofilters host complex, mixed microbial populations (Deshusses, 1997; Cox and Deshusses, 1998), presumably capable of simultaneously treating a variety of pollutants, these reactors have the potential for effective co-treatment of H₂S and VOCs. This would further reduce the overall emission to the atmosphere, and without the need for post-treatment to remove VOCs, **as is needed for** chemical scrubbers. Initial studies performed by the UCR laboratory have demonstrated efficient co-treatment in biotrickling filters of H₂S and VOCs such as toluene and MTBE (Cox and Deshusses, 2000; Deshusses et al., 2001). Results obtained with larger scale reactors at POTWs, however, have this far been less successful, mainly because of lower than expected VOC removal efficiency (Iranpour et al., 2001). The reason for this discrepancy is unclear, perhaps because studies at POTWs mainly focused on **H₂S/odor** removal only.

Once an established and fully developed technology, biological waste gas treatment is expected to provide an environmentally friendly technique for waste gas cleaning at POTWs, at a cost lower than currently with chemical scrubbers. The objective of the present project is to further improve the potential of biotrickling filters in removing both H₂S and VOCs from waste gases from the Headworks and other facilities at POTWs. In this paper, results are presented of the performance of the biotrickling filter over ten months of operation. Performance of the biofilter unit is discussed in a separate paper (Converse et al., 2001).

METHODOLOGY

Equipment

The biotrickling filter was constructed of 304 stainless steel with a diameter of 1.5 m and a height of 3.4 m (Figure 1), containing seven layers of a PVC COOLdektm Munsters 12060 structured packing with a specific surface area of 230 m²/m³ and a porosity of 90-95%. The packed bed height was 2.1 m, resulting in a bed volume of 3.8 m³. Air from the Headworks was introduced into the bottom of the reactor (gas upflow) at an average flow rate of 600 m³/h, corresponding to an empty bed gas residence time (EBRT) of 24 s. The available differential pressure from the Headworks building was about 13 cm of water column, thus no blowers were required to operate the biotrickling filter at the specified gas flow rate. A 0.75 HP pump was used for continuous trickling of recycle liquid over the packed bed at a rate of 1.4 m³/h (superficial liquid velocity of 0.8 m/h). The liquid was collected in the base of the reactor, containing approximately 0.6 m³ of recycle liquid. Secondary effluent water from the plant was supplied as a source of nutrients and to purge produced sulfate. The feed rate was 6-12 L/h and a constant recycle liquid volume was maintained by an overflow outlet. Effluent feed and purge rates were comparable, indicating that evaporation losses were negligible. Control of pH in the recycle liquid was done with a Cole-Parmer stand-alone pH controller, which actuated the metering of 0.75-1.3 M NaOH to the base of the reactor when the pH in the recycle liquid dropped to less than 7.0.

Experimental set-up

Operation of the biotrickling filter started in April, 2000, after the addition of raw influent water from the plant and recycle liquid from a H₂S-degrading biotrickling filter as microbial seeds. Day 0 in graphs corresponds to the day of inoculation, after which

standard operation was started immediately. Results presented herein are over ten months of continuous operation. The pressure drop across the reactor remained always below 2.5 cm water column, and was on average 1 cm H₂O. On a regular basis, operational parameters such as the gas flow rate, liquid recycle rate and the secondary effluent feed rate were determined and adjusted if necessary. Biotrickling filter performance was assessed by frequent analysis of H₂S and VOC removal, as well as the removal of organic sulfur compounds and odor (both only after 254 days of operation). During the first 46 days of the experiment, the biotrickling filter was operated without pH control. A neutral recycle liquid pH was maintained after day 46, although failure of the pH control unit would occasionally cause the pH to drop. Over the course of the experiment, various attempts were made to stimulate the removal of VOCs. These included the addition of specific bacterial cultures as well as VOC spiking experiments. Details are presented below in the Results section.

Analyses

On a semi-daily basis, H₂S was determined by direct measurement in the inlet and outlet airducts using a Jerome 631-X analyzer (Arizona Instruments, Tempe, AZ), capable of measuring H₂S in the range of 1 ppb to 50 ppm. Two or three measurements were usually sufficient to obtain reproducible results. Continuous measurements were done with a Hydrogen Sulfide Monitor, Model 1176, from Interscan Corporation, connected to an Intech Instruments Ltd. Nomad Datalogger. For VOCs, inlet and outlet air was sampled in 10 L Tedlar bags for 3-4 minutes and analyzed the same day according EPA method TO-14 using a GC with photoionization and electrolyte conductivity detection. The method detection limit was 0.5 to 2 ppb depending on the VOC. Analyses were done on a weekly/monthly basis. Organic sulfur compounds were analyzed in triplicate on day 254 by Performance Analytical Inc. (Simi Valley, CA) in a GC equipped with a sulfur chemiluminescence detector. On the same day, samples were collected in Tedlar bags for odor analysis. ASTM methods E-679-91 and E-544-99 were used for quantification of the dilution-to-threshold ratio and the odor intensity (Odor Science & Engineering, Inc., Bloomfield, CT; odor panel of eight members). Gas flow rates to the biotrickling filter were regularly measured in the inlet airduct using a digital Omega anemometer model HHF300A (Stamford, CT), inserted into a straight PVC pipe (ID 14.5 cm, length 2 m).

RESULTS

Pollutant composition of the Headworks' air

The air from the Headworks contained a complex mixture of H₂S, organic sulfur compounds, aromatic VOC's, and chlorinated VOC's. Table 1 presents a summary of the range of concentrations of individual compounds observed during ten months of operation of the biotrickling filter. H₂S was the major pollutant, present in concentrations ranging from 10 to 50 ppm. Organic sulfur compounds were also detected, but only at the ppb level. Toluene, xylenes, dichlorobenzenes, methylene chloride, chloroform and tetrachloroethylene were the major VOC's with concentrations up to 200 ppb. The VOC's analyzed for but consistently present in concentrations below the detection limit (0.5 or 2 ppb) were: vinyl chloride, vinylidene chloride, carbon tetrachloride, 1,1dichloroethane, 1,2-dichloroethane, benzylchloride and methyl chloride. The ranges reported in Table 1 indicate wide fluctuations of observed concentrations of the individual pollutants. For H₂S, continuous measurements revealed that the concentration was the lowest at the end of the morning, after which a rapid increase was observed during the afternoon (Figure 2). Identical trends were observed both on weekdays and during the weekend. We also noticed that H₂S concentrations were relatively high during the summer, which could be the result of stimulation of sulfate reduction in the collection system by elevated temperatures. Concentrations of individual VOCs also greatly varied, but without apparent correlation with the H₂S concentration or amongst each other.

H₂S removal

After start-up of the reactor, H₂S biodegradation started immediately and the biotrickling filter removed H₂S at the maximum elimination capacity after 1 month of operation. As the reactor was initially operated without pH control, a rapid decrease of the recycle liquid pH was observed down to a value of 1.5. After 46 days of operation, a neutral pH was maintained in the recycle liquid by metered addition of caustic soda. As presented in Figure 3, the H₂S removal efficiency was consistently greater than 98% over ten months of operation and irrespective of the recycle liquid pH. The average H₂S outlet concentration was 0.44 ppm, and with two exceptions was always less than 1 ppm, which is the current limit for H₂S emissions required by the South Coast Air Quality Management District.

The $\rm H_2S$ elimination capacity of the biotrickling filter (Figure 4), expressed as the amount of $\rm H_2S$ removed per $\rm m^3$ of packed bed per hour, ranged from 1 to 13.8 g/m³.h,

with an average capacity over 280 days of 5.2 g/m³.h. Higher elimination capacities are reported in the literature, but it should be noted that the biotrickling filter was not operated at its maximum elimination capacity. This is further demonstrated in Figure 5, which shows H₂S concentration profiles over the height of the reactor. These were determined both at low-pH operation of the biotrickling filter (the first 46 days) as well as during operation with pH control. An important conclusion of the concentration profiles is that a high H₂S elimination can be obtained at high (30-50 ppm) H₂S concentrations. The H₂S elimination capacity in the first section of the packed bed (0-0.65 m) was 30 g/m³.h, which demonstrates the applicability of biotrickling filters in removing H₂S at a fast rate. On the other hand, removal of low H₂S concentrations occurred at a much slower rate, as observed in the second (0.65-1.3 m), third (1.3-1.95 m) and fourth (1.95-2.6 m) section of the packed bed. This could be due to mass transfer and/or biological limitations at low H₂S concentrations. Figure 5 also demonstrates that the pH did not have a significant effect on the H₂S concentration profiles, hence, H₂S removal is feasible over a wide range of pH.

Fluctuating H₂S concentrations are the rule in field applications, which requires a fast response of the biotrickling filter to consistently keep emission levels below the required limit. On day 126, during a week of continuous H₂S measurements, a sharp increase of the inlet concentration occurred with a **temporary** maximum of 65 ppm at 10:00 am (Figure 6). The origin of this surge could not be identified. Although a slight increase in the outlet concentration was observed, the biotrickling filter effectively removed the H₂S peak. This demonstrates the fast response of the biotrickling filter to rapidly changing inlet concentrations. Such a response may be attributed to two factors. First, the biotrickling filter was operated below the maximum elimination capacity, as indicated by near complete removal of 40 ppm H₂S in the first two sections of the packed bed (Figure 5). Second, the large volume of recycle liquid in the base of the reactor may act as a sink for absorption of H₂S at temporarily high concentrations. As the outlet concentration did not show a tailing effect when the inlet concentration returned to a normal level, biological removal rather than absorption seems to be the primary cause of eliminating the H₂S surge.

Removal of organic sulfur compounds

Organic sulfur compounds removal was determined 254 days after the start-up of the biotrickling filter. Methyl mercaptan was removed at an average efficiency of 70%, which may indicate that H₂S-oxidizing microorganisms in the biotrickling filter were capable of also metabolizing this compound. A reduction of the *tert*-butyl mercaptan

concentration was also observed, although an accurate estimation of the removal efficiency was not possible because *tert*-butyl mercaptan was present in a concentration close to the detection limit (2 ppb). Carbonyl sulfide, dimethyl sulfide and carbon disulfide were not removed.

Removal of VOCs

Average VOC removal with and without pH control in the biotrickling filter is presented in Table 2. In some cases VOC concentrations were higher at the outlet than at the inlet (negative removal efficiency). This believed to be due to a combination of fluctuating VOC concentrations in the Headworks' air, a 5-10 min delay in sampling between the inlet and outlet, and possibly absorption/desorption effects. Throughout the entire experimental of 280 days, removal of VOCs was poor, irrespective of experimental conditions. No removal of xylenes or any of the chlorinated compounds was observed. Moderate but significant removal of toluene and benzene occurred, but only when a neutral pH in the biotrickling filter was maintained (Table 2). This emphasizes that pH control is required for VOC removal, but other factors play a role as well because relatively biodegradable compounds such as xylenes and dichloromethane were not removed at a neutral pH.

One factor we considered was the possibility of VOC-degrading microorganisms not being present in sufficiently high numbers to catalyze significant VOC removal. At first, a few liters of laboratory cultures of pure species grown on toluene and dichloromethane were added to the biotrickling filter. This had no effect on the removal of either compound over a period of several weeks after the addition. Therefore, it might tentatively be concluded that the conditions prevailing in the biotrickling filter did not allow establishment and growth of a VOC-degrading population. In a subsequent experiment, it was decided to artificially increase the VOC loading in order to enhance the growth of VOC-degrading microorganisms. The concentrations of toluene, benzene and xylenes (added as gasoline), dichloromethane and trichloroethylene were temporarily increased by continuous injection of the liquids into the inlet airstream for a period of several weeks. Although this procedure raised the concentration of the spiked VOCs from the ppb to ppm level, no improvement of removal was observed (Table 3), and although the pH was controlled at a neutral value without interruption. At this point it is not clear why the spiking experiment failed in enhancing VOC removal.

Odor removal

The average reduction of odor as determined in the morning, afternoon and evening of day 254 was 97-98.8%. The odor panel characterized the smell of the samples at various dilutions. The smell of both treated and untreated air was described as 'sewage, rotten eggs, rotten garbage, and mercaptan.' The characteristic smell of H₂S was specifically mentioned for untreated samples only.

The observed odor reduction fully correlates with the removal of H₂S. Although VOCs and especially the organic sulfur compounds may have odor thresholds less than that of H₂S, concentrations of these compounds in the Headworks' air are apparently too low to significantly contribute to the odor. Poor removal of VOCs and organic sulfur compounds did therefore not interfere with the odor-reducing performance of the biotrickling filter in this study.

DISCUSSION

The present study shows that 10-50 ppm H₂S can effectively be removed at an EBRT in biotrickling filters of 10-15 s, which is close to the gas residence time in the chemical scrubbers currently employed at Hyperion. As H₂S was the major odorous compound in the Headworks' air, near complete elimination of odor was observed as well. These findings confirm the applicability of biotrickling filters for H₂S and odor removal at POTWs and other industries, as reviewed by Iranpour et al. (2001).

An additional feature of biotrickling filters would be the elimination of VOCs. Although some removal of toluene, benzene and dichlorobenzene was observed, the overall performance was less than expected. Similar experiences have been reported by others (Torres et al., 1996; Devinny et al., 1997; Chitwood et al., 1999; Webster et al., 2000). Effective VOC removal requires the presence of VOC-degrading microorganisms, as well as conditions that facilitate their growth and stimulate their activity. In this respect it should be noted that most H₂S-degrading microorganism are autotrophic, i.e., they use CO₂ as the carbon source for growth, while oxidation of H₂S provides energy for the cell. On the other hand, VOCs are used as a source of carbon and energy by heterotrophic microorganisms. Co-treatment of H₂S and VOCs in one reactor therefore requires the existence of a mixed consortium containing sub-populations with different requirements for growth and energy. Our laboratory-scale experiments prior to this study have shown that VOCs such as toluene and methyl *tert*-butylether (MTBE) can be removed at fast rates in biotrickling filters treating H₂S (Cox and Deshusses, 2000; Deshusses et al., 2001). No cross inhibition was observed between H₂S removal and the removal of

toluene or MTBE, as long as a neutral pH in the biotrickling filters was maintained. It can therefore be concluded that biotrickling filters can indeed host mixed populations that simultaneously remove H₂S and VOCs. Poor VOC removal observed in the present study must then be attributed to conditions in the biotrickling filter being not favourable for heterotrophic microorganisms, especially since the addition of pure cultures specialized in removing toluene and dichloromethane did not have any effect. At this point, one can only speculate about the reasons for poor or non-existent VOC removal. Laboratory experiments have demonstrated VOC removal in biotrickling filters at rates of 10 to >100 g/m³.h of most the VOCs found in the Headworks' air (eg., Diks and Ottengraf, 1991; Baltzis and Mpanias, 1998; Pol et al., 1998; Cox and Deshusses, 1999; Fortin and Deshusses, 1999; Lu et al., 1999). Such removal rates are orders of magnitude higher than required for complete removal of VOCs at the concentrations in the Headworks' air at the gas residence time employed in this study. One possible explanation could be that VOC concentrations at the ppb level, depending also on the air to water partitioning coefficient of the particular compound, were too low to sustain an actively growing heterotrophic population (Alexander, 1999). This was the reason for conducting spiking experiments, but VOC removal remained low after increasing concentrations by 2-3 orders of a magnitude. A toxic effect of the VOCs can be ruled out as spiked concentrations were still well below the concentrations often applied in biotrickling filter laboratory experiments (eg., Iranpour et al., 2001). One other explanation for poor VOC removal could be the accumulation of inhibitory concentrations of sulfate in the recycle liquid. From conductivity measurements we estimated an average Na₂SO₄ concentration in the recycle liquid of 10.5 g/L. This concentration appears to be too low to cause inhibition, as previous experiments with biotrickling filters simultaneously removing MTBE and H₂S have indicated that MTBE removal was not affected by Na₂SO₄ in a concentration up to 60 g/L (Deshusses et al., 2001). At present, we are investigating alternative explanations for poor VOC removal. These include the nutrient composition of the secondary effluent feed and the possible accumulation of metabolites other than sulfate that could inhibit VOC biodegradation.

Overall, the present study demonstrates the feasibility of biotrickling filters in removing H_2S and reducing odor at POTWs. H_2S removal is fast and operation can be sustained over the long run without concern for excessive biomass build-up and increasing pressure drops as sometimes is observed in biotrickling filters receiving high loads of VOCs (Smith et al., 1996; Alonso et al., 1997; Laurenzis et al., 1998). Economical evaluations have also indicated that biotrickling filters at POTWs are about 30% more cost effective in removing H_2S than chemical scrubbers (Deshusses et al., 2001). Poor VOC removal

in POTW biotrickling filters remains however a point of concern, as is the case for chemical scrubbers. This study and others, as discussed by Iranpour et al. (2001), show that VOC removal is the limiting factor in designing biotrickling filters for simultaneous H₂S and VOC removal at POTWs.

CONCLUSIONS

Biotrickling filters are very effective in removing H₂S and odors from waste gases at POTWs while avoiding the drawbacks of chemical scrubbing. An additional feature of biotrickling filters is the potential for VOC removal. This research demonstrated simultaneous removal of H₂S and some of the VOCs present in POTW waste gases, however, VOC removal was in general poor and less than expected. Stimulation of VOC removal in biotrickling filters would first require identification of the cause for poor VOC removal in order to further reduce the overall toxicity of the waste gas.

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Table 1 – Composition of the headwork's air.

Pollutant ¹	Concentration	Pollutant ¹	Concentration
H ₂ S	10-50 ppm	Benzene	0.5-2.5 ppb
Carbonyl sulfide	19-52 ppb	Toluene	10-153 ppb
Methyl mercaptan	149-165 ppb	Xylenes	12-125 ppb
Dimethyl sulfide	8-12 ppb	Dichlorobenzenes	1-210-ppb
Carbon disulfide	6-8 ppb	Methylene chloride	4-120 ppb
Tert-butyl mercaptan	2-3 ppb	Trichloroethylene	1-15 ppb
		Chloroform	16-102 ppb
		Tetrachloroethylene	15-225 ppb

¹ Concentrations of H₂S and VOCs were the average of a large number (>7) of samples taken over one year. Concentrations of organic sulfur compounds were the average of three determinations done 254 days.

 $\label{eq:control} Table~2-VOC~removal~efficiency~(RE)~in~the~biotrickling~filter~with~and~without~pH~control.$

VOC	No pH control ¹		pH control ²	
	Inlet conc.	RE (%)	Inlet conc.	RE (%)
	(ppb)		(ppb)	
Benzene	1-22	0	8-13	35
Toluene	10-153	-4	38-73	36
Xylenes	13-120	-4	19-124	-2
Dichlorobenzenes	1-9	-19	11-17	-14
Dichloromethane	4-43	-2	11-63	-3
Chloroform	16-76	-2	58-102	-4
Tetrachloroethylene	15-89	-2	33-151	4
Chlorobenzene			1-2	25
Trichloroethylene	1	-4	4-15	-5

¹ Measurements over day 14-30, average of 12 determinations, operation without pH control, pH 1.5-2.

² Measurements over day 57-133, average of 7 determinations, operation with pH control, pH 7-9.

Table 3- Removal of VOCs during the spiking experiment (day 204-226, pH control).

VOC ¹	Inlet concentration ² (ppm)	Removal efficiency (%)
Benzene	0.18-0.36	6
Toluene	0.90-2.31	6
Xylenes	1.12-2.97	8
Dichloromethane	4.07-9.88	-8
Trichloroethylene	1.91-4.20	-6

¹ Continuous feed of gasoline as source of toluene, benzene and xylenes. Dichloromethane and trichloroethylene were fed as the pure compounds.

² Average of 6-10 determinations.

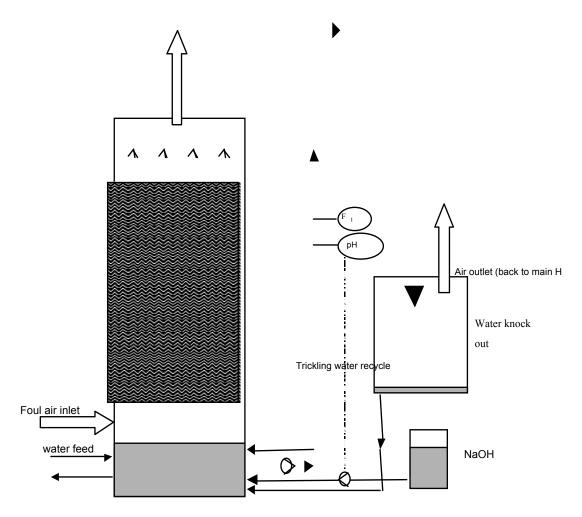
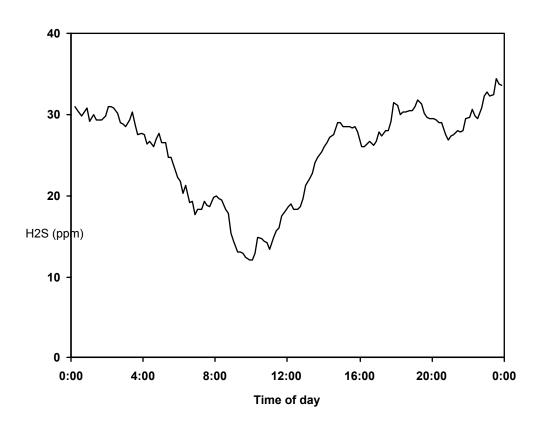


Figure 1. Schematic of the UC Riverside Biotrickling filter



 $Figure\ 2-Continuous\ measurement\ of\ the\ H_2S\ inlet\ concentration\ on\ day\ 125.$

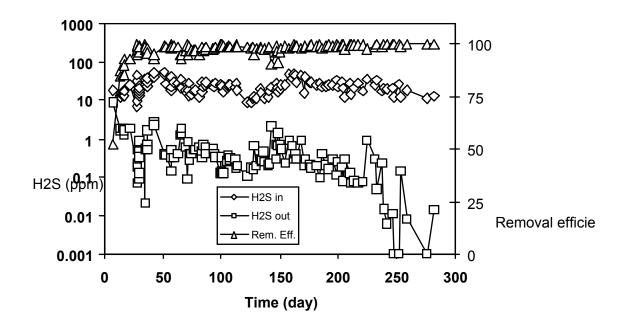


Figure $3-H_2S$ inlet and outlet concentrations and the removal efficiency over ten months of operation of the biotrickling filter; average EBRT of 24 s.

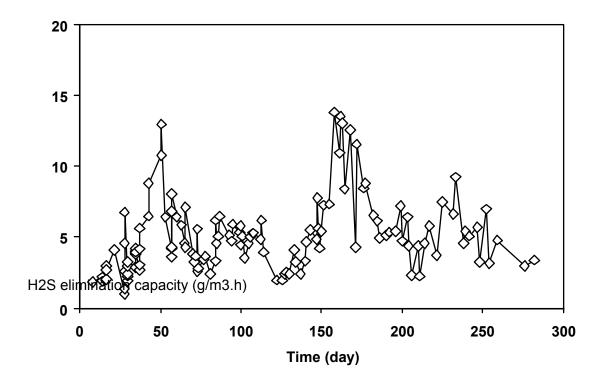


Figure $4-H_2S$ elimination capacity in the biotrickling filter over ten months of operation.

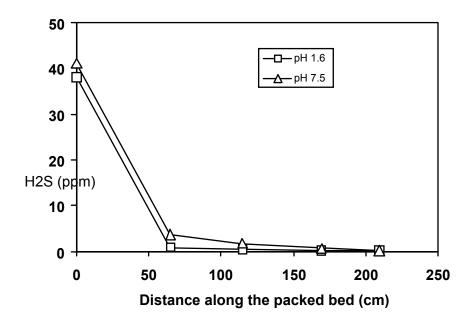


Figure $5 - H_2S$ concentration profiles over the height (inlet = 0 cm) of the packed bed; profiles were determined during operation with and without pH control.

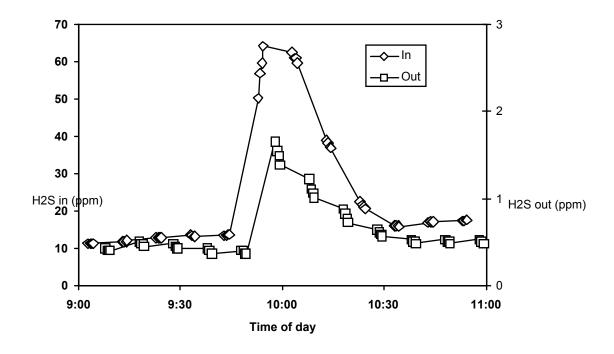


Figure 6 – Response of the biotrickling filter to a ${
m H}_2{
m S}$ surge (day126).