WATER RESEARCH 40 (2006) 2629–2636

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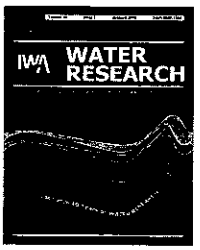

Biological treatment of H_2S using pellet activated carbon as a carrier of microorganisms in a biofilter

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ARTICLE INFO

Article history:
Received 19 December 2005
Received in revised form
9 May 2006
Accepted 12 May 2006

Keywords:
Hydrogen sulfide
Acidithiobacillus thiooxidans
Biological activated carbon
Biofilter

ABSTRACT

Biological treatment is an emerging technology for treating off-gases from wastewater treatment plants. The most commonly reported odourous compound in off-gases is hydrogen sulfide (H_2S), which has a very low odor threshold. This study aims to evaluate the feasibility of using a biological activated carbon as a novel packing material, to achieve a performance-enhanced biofiltration processes in treating H_2S through an optimum balance and combination of the adsorption capacity with the biodegradation of H_2S by the bacteria immobilized on the material. The biofilm was mostly developed through culturing the bacteria in the presence of carbon pellets in mineral media. Scanning electron microscopy (SEM) was used to identify the biofilm development on carbon surface. Two identical laboratory scale biofilters, one was operated with biological activated carbon (BAC) and another with virgin carbon without bacteria immobilization. Various concentrations of H_2S (up to 125 ppmv) were used to determine the optimum column performance. A rapid startup (a few days) was observed for H_2S removal in the biofilter. At a volumetric loading of 1600 $m^3 m^{-3} h^{-1}$ (at 87 ppmv H_2S inlet concentration), elimination capacity of the BAC (181 $gH_2S\, m^{-3} h^{-1}$) at removal efficiency (RE) of 94% was achieved. If the inlet concentration was kept at below 30 ppmv, high H_2S removal (over 99%) was achieved at a gas retention time (GRT) as low as 2 s, a value, which is shorter than most previously reported for biofilter operations. The bacteria population in the acidic biofilter demonstrated capacity for removal of H_2S in a broad pH range (pH 1–7). There are experimental evidences showing that the spent BAC could be re-used as packing material in a biofilter based on BAC. Overall, the results indicated that an unprecedented performance could be achieved by using BAC as the supporting media for H_2S biofiltration.

1. Introduction

Biological treatment processes are promising techniques and have wide applications in odourous air pollution control. The selection of an appropriate packing media is essential to the overall odor removal performance of a bioreactor system, which is generally believed to depend primarily on the type of packing medium (Luo, 2001). Packing medium should have a high surface area, high air and water permeability, and provide a good surface of microbial growth (Elias et al., 2002). It also plays an important role in air and water distribution, as well as mass transfer (Yang et al., 2000). So

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doi:10.1016/j.watres.2006.05.021

far, few examples were methodically reported about the relationship between the characteristics of packing material and bioreactor performance (Higuchi et al., 2000).

Packing materials used up to date include either natural materials (Cho et al., 2000; Smet et al., 1996; Yang and Allen, 1994a) such as soil, compost, peat, wood chips, and lava rock, or synthetic materials (Gabriel and Deshusses, 2003a; Kinney et al., 1996; Koe et al., 2001; Sorial et al., 1998) such as ceramic saddles, polyethylene pall rings, polyurethane foam, activated carbon, and extruded diatomaceous earth pellets. Natural materials such as compost and wood chips could be used but the surface-to-volume ratios are low which results in low volumetric reaction rate (Cho et al., 2000). Large footprint is generally required for natural materials and upgrading is difficult if flows increase due to the process expansion. For a long-term operation, settling of media might cause channeling thus reducing performance over time. Organic media also need to be replaced after 3–5 years, and they are difficult to be regenerated (Gabriel and Deshusses, 2003a). On the other hand, although humidified air must be used and nutrients must be supplied, the use of synthetic packing media has advantages, such as low head losses due to larger interstices between packing granules or pieces, larger specific surface areas, and solid phase adsorption of contaminants (Martin et al., 2002).

Among synthetic packing media, activated carbon has been the most extensively used material for physical adsorption of odor. However, its limited capacity and high cost prevents carbon from being a broader application. Biological systems that employ biological activated carbon (BAC) for the treatment of organic pollutants in water, wastewater, or air have been known to exhibit superior performances (Ehrhardt and Rehm, 1985; Li et al., 2002a; Liu and Barkley, 1994). The major function of activated carbon is to support the microorganisms and act as a buffer for fluctuating loading though biofilm might hinder the carbon adsorption. The enhanced performance may be manifest in higher removal efficiency compared to conventional biological systems, shorter acclimation periods of the microorganisms in the system, and lower pollutant concentration in the effluent during step increases in the influent pollutant concentration (Voice et al., 1992). Activated carbon as a filter material mixed with other materials (mostly compost) performed successfully in some biofilter applications (Abumaizar et al., 1998; Mohseni et al., 1998). Although some studies on organic pollutants treatment using BAC have been reported, further investigation is still needed using carbon as packing media for bacteria immobilization, particularly for the application of BAC in biological deodourisation processes. BAC should provide a more efficient odor treatment compared to other conventional media, but supporting data for this assumption is not sufficient yet.

In this research, a laboratory-scale cylindrical biofilter system was set up to investigate the performance of BAC (e.g., elimination capacity, removal efficiency). Various operating parameters were studied including H_2S inlet concentration, gas retention time (GRT), gas flow rate, and frequency of system irrigation, etc. Spent activated carbon (SAC; saturated with H_2S) was also investigated, targeting at the potential re-use or bio-regeneration. Scanning electron microscopy (SEM) was used to observe the biofilm development on the activated carbon.

2. Materials and methods

2.1. *Activated sludge acclimation*

An acclimated activated sludge was used in this work rather than the activated sludge itself for a quick system set up. It was prepared using a normal activated sludge acclimated to Thiosulphate (TS) medium for 5 days, and transferred to a fresh medium after the 5-day acclimation. The composition of TS medium is as follows: $Na_2S_2O_3 \cdot 5H_2O$, 10 g L^{-1}; KH_2PO_4, 1.5 g L^{-1}; K_2HPO_4, 1.5 g L^{-1}; NH_4Cl, 0.4 g; $MgCl_2 \cdot 6H_2O$, 0.8 g L^{-1}; $CaCl_2 \cdot 2H_2O$; 0.05 g L^{-1}; cycloheximide (anti-fungus reagent), 0.05 g L^{-1} (pH = 6.8 ±0.2). Sulfide oxidizing bacteria were obtained from a return activated sludge stream at the secondary sedimentation tank at a local wastewater treatment plant. After acclimatization for about 15 days (2 transfers), the bacteria seeds were ready for inoculating onto the activated carbon bed.

2.2. *Biofilter system*

A laboratory-scale biofilter system was designed and constructed. It consisted of parallel dual vertical columns, which could be operated simultaneously and controlled separately (Fig. 1). The packing material (Calgon AP460) was placed in a transparent and rigid Perspex tubing, which has an inner diameter of 3.6 cm and a height of 30 cm. The carbon bed was packed inside the tubing to a height of 20 cm and yielded 0.2 L of packing volume. The packed material was supported by a plastic sieve plate to ensure a homogeneous distribution of the inlet gas across the bed. In this work, columns A and B are distinguished by whether the carbon bed is immobilized with bacteria or not. The composition and physical properties of the columns are summarized in Table 1.

Sampling ports are located along the column for gas sampling and pressure measurements. The individual sampling ports are identified based on their locations along the biofilter column as inlet, 5, 10, 15 cm and outlet ports. In this study, most of samplings were conducted at outlet, except for several points where 10 cm sampling port was used dealing with short GRTs (at 1 s). The humidified air stream was prepared by blowing air through a gas wash bottle that contains water (the humidification chamber). Moreover, the bed was irrigated twice a day by submerging the bed in culture medium for 10 min and then releasing the solution. The desired H_2S inlet concentration was adjusted by the needle valve at the outlet of the 5% H_2S gas cylinder (balanced in N_2, Linde Gas Singapore Pte Ltd). Foul gas (containing various concentrations of H_2S) flow rates were controlled and measured using AALBORG (Orangeburg NY, USA) flow meters with units of L min^{-1} located at the inlet of the wash bottle, blowing upward from the bottom inlet into the biofilter. Two additional pressure ports were installed at the top and bottom of the column in order to measure the pressure drop by a water manometer with a minimum reading of 1-mm water column. The column was sealed with fitted rubber stoppers. The rubber stopper on the top of the column is removable so that water and mineral sources can be introduced into the filter material to maintain sufficient moisture and mineral nutrient contents in the bed. All the gas lines were 1/4-in. diameter Teflon tubing. The system was operated at room temperature of about 25 °C throughout all the experimental runs.

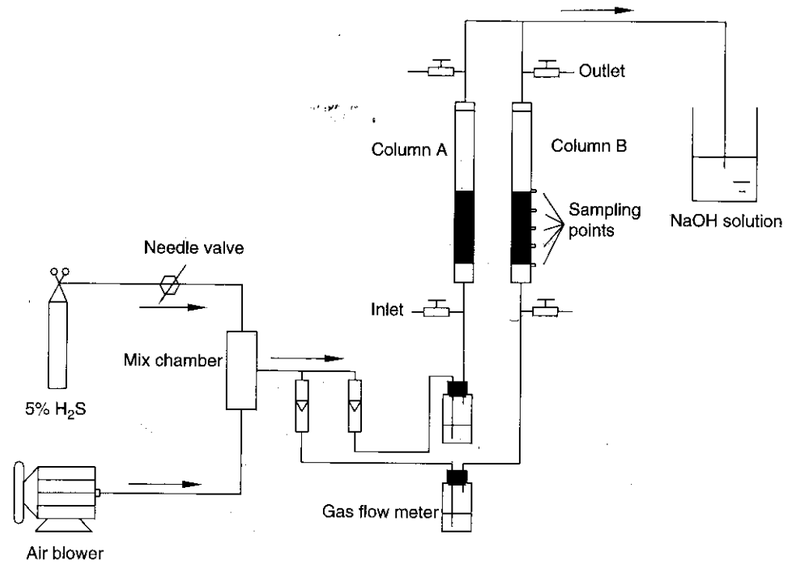

Fig. 1 – Schematic diagram of bench-scale biofilter system.

Table 1 – Physical properties of the biofilter columns

Physical property	Column	
	A	B
Bacteria immobilization	With	Without
Wet carbon weight, W_1 (g)	183.4	181.6
Dry carbon weight, W_2 (g)	105.6	106.9
Moisture content, $m = (W_1 - W_2)/W_1$ (%)	42.4	41.1
H_2S adsorption capacity, (wet weight basis, %)	20.08[a]	0.44[b]
Diameter of column (cm)	3.6	3.6
Carbon surface area, m^2 g^{-1}	807	807
Diameter of carbon pellet (mm)	4	4
Height of media in each column, L (cm)	20	20
Volume of packing, V (L)	0.2	0.2
Bulk density, $d = W_1/V$ (kg m^{-3})	917	908
Apparent density of activated carbon, ρ (kg m^{-3})	490	490

[a] H_2S consumption capacity for 100 days' biofilter running time.
[b] H_2S exhausted capacity.

Table 2 – Operating conditions for both columns

Operating parameter	Range
Gas retention time (s)	2–21
Inlet H_2S concentration, C (ppmv)	5–100
Gas flow rate, Q_g (L min^{-1})	0.57–4
Superficial foul gas velocity, v (cm s^{-1})	0.95–10
pH of the column	1.0–2.0

2.3. *Bacteria immobilization*

An online immobilization was adopted to start the biofilter in this study, and the procedures as follows: Virgin activated carbon pellets were autoclaved and stuffed into both columns randomly. A 5 mL of concentrated microbial broth was added into the 45 mL fresh mineral medium (liquid medium without $Na_2S_2O_3 \cdot 5H_2O$). Then, 50 mL bacteria solution with a bacteria concentration of 1.96×10^8 cfu mL^{-1} was poured from the top of the column A to submerge the carbon bed with 5 mm water level above it. For column B, the control column, 50 mL distilled water was used instead of bacteria solution. At the same time, the synthetic foul gas with a low concentration of H_2S (about 20–50 ppmv) was blown into both packing columns. The feed of H_2S provided energy source for the bacteria to grow. Upon completing the immobilization and acclimatization stage (~1 week in this study), the system was deployed for performance evaluation of BAC. The operating conditions during this period are tabulated in Table 2. In Table 2, the non-inoculated column also has a low pH which is comparable of inoculated column, because of

the adsorption of H_2S gas by activated carbon, as reported previously (Yan et al., 2002).

2.4. *Analytical methods*

Periodical measurements of gas concentration from sampling ports, pressure drop in the columns, and gas flow rate of each column in the biofilter system were carried out using the following devices. Tedlar bags were used to collect the gas samples from various locations on the columns. H_2S concentrations were measured using Jerome 631-X Hydrogen Sulfide Analyzer (Arizona Instruments, USA). The analysis of each gas sample was carried out immediately after sampling to avoid the deterioration of H_2S sample. To eliminate any effect of residual H_2S in the Tedlar bags, all sample bags were flushed by clean air immediately after each analysis. Three analyses for each sample were carried out for consistency. Gas flow rates were controlled and measured using AALBORG (Orangeburg NY, USA) flow meters with units of L/min. Flow Injection Analyzer with Ion Chromatograph (FIA/IC, Lachat, QuickChem 8000) was used for sulfate analysis. SEM (Stereoscan 420, LEO, UK) was used to identify the biofilm development and BAC porosity. BACs were sampled periodically for microbiological examination by plate counting method on the TS medium at pH of 4. Dominant sulfur oxidizing bacteria that appeared on the TS medium were isolated and subject to a 16S rRNA partial sequencing for identification.

The pH of carbon suspension, which provides useful information about the average acidity/base of carbon surfaces, was measured in the following way. About 1 g of the selected exhausted carbon was soaked in 50 ml of ultra pure water and stirred in the auto-shaker for 24 h to reach equilibrium. The sample was filtered, and the pH of solution was measured using Horiba pH meter F-21 (Horiba International Corporation). The results were referred to as "pH of carbon" for simplification.

3. Results and discussion

3.1. *Performance during startup period*

Fig. 2a and b shows the performance of the two biofilters during the initial 21 days of operation. Analysis of the H_2S removal performance during the startup period phase (GRT was adjusted to 21 s, Fig. 2a) revealed that the removal efficiencies (RE) started to increase after 1 day of operation to reach a value of 90% for column A which contained the BAC, and 70% for column B which contained virgin carbon, about 6 days into the experimental run. The pH of the BAC declined to a value of 2 after 4 days of operation. The increase

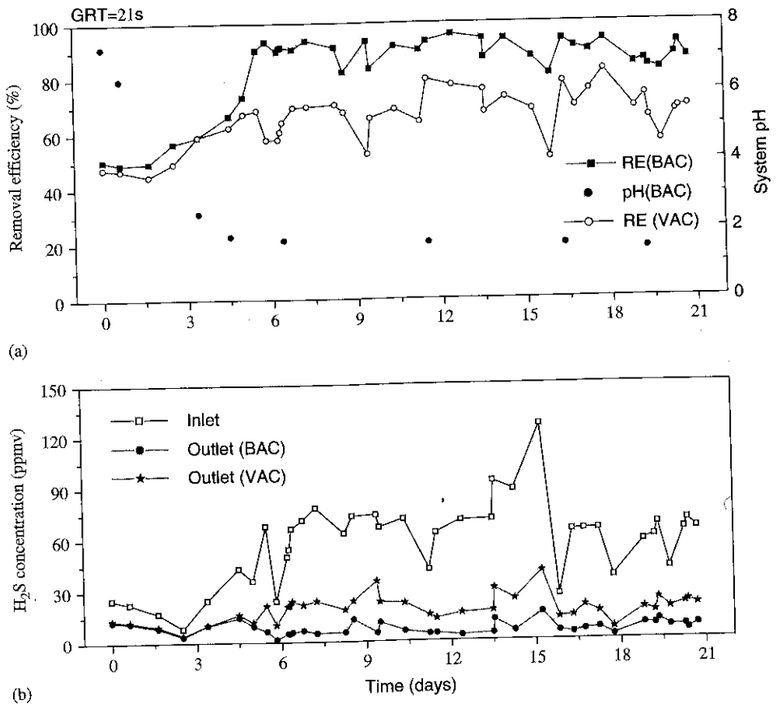

Fig. 2 – Performance of biofilter bed during the start-up period. (a) Removal efficiency and pH vs. time; (b) Inlet and outlet concentrations vs. time.

Fig. 3 – BAC surface after online immobilizing for 16 days in a biofilter.

in RE correlated with the decline of pH, due to the production of H^+ and sulfate from the oxidation of H_2S. Acclimation lasted around 6 days, after which the H_2S RE remained high for the remaining duration of the study. H_2S inlet concentrations ranged from 10 to 125 ppmv during the experimental run. Based on the data obtained, it can be considered that the biofilter with the BAC was ready for use after 6 days of startup period (Fig. 2). A longer start up time may be necessary if BACs is applied to a larger scale biofilter.

In Fig. 2b, although a highly fluctuating inlet concentration was repeatedly observed during the 21 days of running time, the biofilter system was robust enough to consistently absorb and treat incoming H_2S with variable concentrations of 10–125 ppmv. In order to confirm the formation of a suitable layer of biofilm on the carbon surface, a sample pellet of BAC was taken out from the middle of the media bed for analysis on the 16th day of operation. The surface characteristics of the sample was determined by SEM. From the SEM photograph shown in Fig. 3, it can be seen that a biofilm containing rod shape bacteria is formed on the carbon surface. Dominant sulfur oxidizing bacteria species in this system was identified as *Acidithiobacillus thiooxidans* by partial rRNA partial sequencing. On the other hand, column B is irrigated by distill water, growth of bacteria in distill water is thus impossible. SEM pictures were also taken from column B, with no biofilm but only chemical crystal shown.

During the startup period, the activated carbon pellets used in both column A and B were soaked in a liquid medium (either bacterial solution for column A or water for column B) for some time intervals. The water content of the wet carbon pellets averaged about 56 wt%. As the existence of a water layer around the medium used in a biofilter would inhibit mass transfer of contaminants from the gas phase to the biofilm (Li et al., 2002a), it is hence not surprising that the average removal efficiency of the BAC biofilter in column A is less than 94%.

3.2. *Long-term performance*

After 21 days startup period, the excess culture liquid medium was drained off the biofilter, and another 79 days was taken to evaluate its long-term performance. During this period, the biofilters were irrigated twice per day to maintain the system moisture. Long-term performance of the biofilter trial is reported in Fig. 4, expressed in the form of elimination capacity vs. loading rate. Pollutant loading rate is an important variable in a biofilter design. In this study, the loading rate changes were the result of the fluctuations in H_2S inlet concentration and the velocity of the influent gas when flow rates were varied. The biofilter containing the BAC consistently degraded more than 97% of the incoming H_2S loading when loading rate was less than 100 $gH_2S\,m^{-3}h^{-1}$ (Fig. 4). At a loading rate above 100 $gH_2S\,m^{-3}h^{-1}$, the breakthrough of the BAC occurred, whereas a quasi-zero-order degradation regime was observed at loadings over 150 $gH_2S\,m^{-3}h^{-1}$, with RE gradually decreasing. The elimination capacity of BAC can reach 100 $gH_2S\,m^{-3}h^{-1}$ at a GRT of 2 s, comparable to the maximum value (110 $gH_2S\,m^{-3}h^{-1}$) previously reported at GRT around 2 s (Gabriel and Deshusses, 2003b). At a volumetric loading of 1600 $m^3\,m^{-3}h^{-1}$ (87 ppmv H_2S inlet concentration), a maximum elimination capacity of the BAC (181 $gH_2S\,m^{-3}h^{-1}$) at a RE of 94% was achieved.

The relationship between the inlet H_2S concentration, GRT, and H_2S removal efficiency is shown in Fig. 5. The H_2S concentrations varied from 20 to 100 ppmv and at each H_2S setting, the GRT for the biofilter was changed from 6 to 1 s. BAC can work efficiently at a GRT of 4 s or above in spite of the changes in the influent concentrations of H_2S. Reducing GRT further (<4 s) as expected resulted in lower H_2S removal. Even so, RE of 98% were commonly reached for inlet H_2S concentrations as high as 30 ppmv when the system was operated at GRT as short as 2 s. Such performance is exceptionally high compared to other biofilters removing a

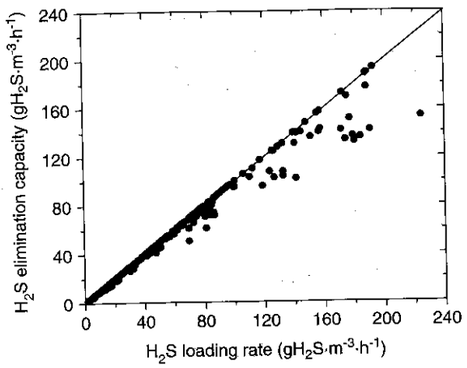

Fig. 4 – H_2S elimination capacity with loading rate at various gas retention times (from 2 to 21 s).

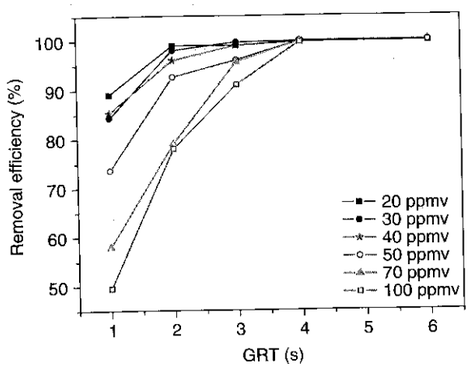

Fig. 5 – Biofilter performances under various H_2S inlet concentrations and gas retention times.

low concentration of H_2S even at longer GRTs (Burgess et al., 2001; Koe et al., 2001; Sublette and Sylvester, 1987; Wu et al., 2001). For most local wastewater treatment plants, H_2S concentration in the sewage air stream is typically less than 30 ppmv. Hence, biofilters packed with BAC should be able to perform efficiently and can even remove H_2S effectively at short GRTs of as low as 2 s.

3.3. BAC vs. virgin activated carbon

H_2S RE of the VAC (the control column B) dropped sharply after working for 34 days, while the RE of the BAC could be maintained at a high level of over 99% for most of the time. H_2S removal capacity of VAC was then calculated based on 34 days of control column operation, and it was found that VAC had removed about 0.49 wt% (wet weight basis) of H_2S in this control column. This value is close to the H_2S breakthrough capacity (0.44 wt%, wet weight basis) of the VAC. For comparison with the capacity of the BAC to remove H_2S, the results indicated that the BAC had removed H_2S of 20.08% (wet weight basis) after operating for about 100 days and the biofilter was still capable of removing additional H_2S. The results preliminarily proved that BAC has an excellent performance on H_2S removal, and also, BAC could extend the life span of activated carbon.

3.4. Some factors influencing performance

There are some limitations observed in this system. First, the accumulation of sulfate and excess biomass in the packing bed are problems frequently encountered. During the operation of the biofilter, white deposits were observed on the carbon surface, and the color of the bed progressively changed from black to whitish yellow from the bottom of the bed to the upper carbon layers. The rate of deposition seemed proportional to the rate of H_2S loading. A sudden increase in H_2S loading over a wide concentration range and prolonged operation at high H_2S loading rates caused the white-colored material to accumulate rapidly. This discoloration of the bed was companied by a rapid drop of the bed pH.

Extremely high system acidity was found to be harmful to the microorganism niche. In this study, active cell number in the microorganism niche dropped quickly when the system pH was below 1. Although low pH does not assist H_2S adsorption, it provides favorable conditions for the sulfide oxidization. The optimum pH for *A. thiooxidans* is generally between 1.0 and 1.5. (Gabriel and Deshusses, 2003b). Further studies are needed to understand better the effect of low pH in BAC biofilters. The accumulation of biomass and sulfide biooxidation products, including elemental sulfur, sulfate, TS, etc., may inhibit the system performance. Both biomass and elemental sulfur are most likely to cause fouling. Most other sulfur intermediates are soluble. The fouling could block the carbon surface pores and the pathway of gas stream thus causing the biofilter to be less efficient.

Secondly, the bed was found to dry out easily and need to be washed periodically as the correct moisture content of biofilter media is another key parameter necessary for its good performance. Too high a moisture level will inhibit the mass transfer from the gas phase to the biofilm or adsorption surface of the carbon. On the contrary, drying out of the media will definitely harm the healthy growth of microorganisms that are immobilized on the supporting media surface (Morales et al., 2003). Control of moisture requires an understanding of the drying of the packing media due to the changes in inlet air temperature and relative humidity, and also from metabolic heat production by pollutant oxidation.

The accumulation of excess biomass and oxidizing products (e.g. sulfur) could be reduced via periodic washing the media bed by water irrigation. This irrigation could also help maintain the moisture content of the packing bed for the development of a healthy biofilm. Washing the system with 50 mL deionized (DI) water for 10 min could remove about 40% of sulfate accumulated in the carbon bed, but it can only increase the pH by 0.1–0.2 units. Mohseni et al. (1998) reported that the activated carbon could become fouled with biomass after the startup of the biofilter system for a mixture of wood chips and spent mushroom compost amended with activated

carbon. Similar phenomenon was also found in our peer study that applied *Pseudomonas* sp. on BAC to treat toluene (Koe and Liang, 2005). Nevertheless, such phenomenon was not found in this study, due to possibly the appropriate washing frequency chosen. Another reason for reduced fouling is the lower amount of biomass in the column removing H_2S. Autotrophs (*A. thiooxidans*) produce much less biomass than heterotrophs (toluene degrading isolates).

3.5. Using SAC as packing material

Spent carbon might be re-used and bio-regenerated by immobilized bacteria through the consumption of the H_2S molecules previously adsorbed in the activated carbon. One H_2S-exhausted carbon column was used to verify this hypothesis. Similar bacteria immobilization procedure was used for the exhausted carbon, followed by the biofilter performance evaluation. The results are shown in Fig. 6. The initial pH of the SAC is 2.5 compared to a pH of 7.96 for the VAC. After an initial immobilizing period of 6 days (which is the same immobilization period as used for VAC) at a GRT of 24 s, the GRT was reduced to 12 s and then 6 s by increasing the gas flow through the biofilter. It was found that the BAC derived from the SAC work almost as well as that from the VAC. H_2S loading can reach as high as 125 $gH_2S\,m^{-3}\,h^{-1}$ at the 98% removal efficiency level. However, it needs at least a 6 s GRT to achieve a RE over 98%. The BAC generated from SAC is far from exhausted after 55 days of operation. Periodically the RE of the biofilter drops when the media bed dries out or clogging occurs. The results indicate that it is possible to develop BAC based on SAC. This could be a potential solution for the re-use of activated carbon in industrial application.

4. Conclusions

The performance of the bench scale biofilter trial shows that the BAC demonstrated a better performance than non-bacterial activated carbon as an odor adsorbent. Activated carbon was an excellent microorganism carrier in biofiltration suitable for the treatment of H_2S. Microorganisms immobilized on activated carbon were capable of extending the activated carbon's capacity and life span. Most of earlier studies on non-BAC biofilters require 10–30 s of GRT to achieve efficient removal of 20–100 ppmv of H_2S, resulting in the elimination capacity of 110 $gH_2S\,m^{-3}\,h^{-1}$ or less. In this biofilter study, BAC could achieve a better performance with a short online immobilization period of 6 days, and a low GRT of up to 2 s particularly for low inlet concentration. Elimination capacity as high as 181 $gH_2S\,m^{-3}\,h^{-1}$ is achievable and the high performance could be maintained for at least 100 days of biofilter run. The dominant bacterial population is *A. thiooxidans* identified by 16S rRNA sequencing. In addition, the use of spent activated carbon is also a possible medium that can be used as a bioreactor packing material. This could possibly be a potential solution for the reuse of spent carbon in the industry.

From the above results, it can be seen that although both of the H_2S adsorption capacity and adsorption rate of the activated carbon were affected by the biofilm formation, the BAC could still provide an attractive, nutrient-rich environment for bacteria growth because of its good water holding capacity, large external surface area and H_2S adsorption capacity. The combined effects of carbon adsorption and biofilm degradation of H_2S enabled the BAC biofilter to achieve a better performance than those packed with non-bacterial activated carbon alone.

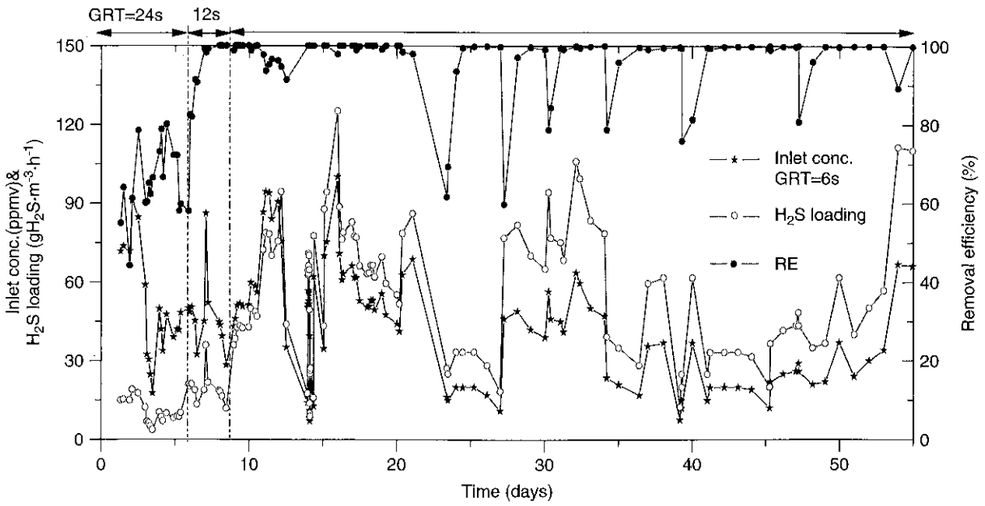

Fig. 6 – Performance of BAC developed from spent activated carbon (H_2S saturated carbon).

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