

Removal of H₂S from Synthetic Waste Gas Streams Using a Biotrickling Filter

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Abstract

The removal of hydrogen sulfide (H₂S) from airstreams was studied in a biotrickling filter packed with porous lava as a carrier of *Thiobacillus thioparus* (DSM5368) with counter current flows of the air and liquid streams. The effect of operating parameters on biotrickling filter performance was studied. Experiments were performed at different empty bed residence times (9-60 sec), and moderate H₂S concentrations (10-90 ppm) to assess the performance of biotrickling filter at different conditions of these parameters. The effect of superficial liquid velocity (0.98-1.95 m h⁻¹) on the performance of biotrickling filter was evaluated. Increasing superficial liquid velocity decreased removal efficiency of the BTF. The gradual change in the concentration of H₂S in different heights of the BTF was investigated. Decreasing empty bed residence time lead to a slight increase in the homogeneity of the removal at different heights of the BTF; however the effect of change in the inlet concentration was insignificant. Complete removal was achieved in the first 85% of the bed. To gain a brief insight into the robustness of the biotrickling filter, its performance was investigated after several upsets in the system.

Keywords: Biotrickling filter, Hydrogen sulfide, Lava rock, *Thiobacillus thioparus*

1- Introduction

Hydrogen sulfide is a colorless and flammable gas that is heavier than air. Its odor threshold is lower than 0.5 ppm and has a very typical smell of rotten egg. Many industrial activities produce hydrogen sulfide in considerable quantities such as waste water treatment, solid waste processing, food processing, petroleum refining, oil refining, natural gas treatment, drug manufacturing,

paper and pulp manufacturing and dye production. Hydrogen sulfide is highly corrosive and extremely toxic with a maximum allowable concentration (MAC) value of 10 ppm. At concentrations less than 50 ppm workers develop symptoms, around 150 ppm the odor of hydrogen sulfide disappears, and at concentrations of more than 500 ppm, leads to the "rapid knock down" of the exposed person [1]. Because of

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these characteristics several methods such as absorption, thermal and catalytic combustion, masking and scrubbing have been developed to eliminate the pollutant from airstreams. The physico-chemical methods have drawbacks of high energy requirement, secondary pollutant production and high chemical and disposal cost [2].

In biofiltration, microorganisms capable of degrading pollutants are immobilized on the bedding material while the treated fluid flows through it [3]. Biofiltration is a cost-effective process for a large flow of gases containing low concentrations of biodegradable compounds. Productions of harmless by-products and minimal operating costs are other advantages of this method compared to physicochemical ones.

Traditional biofilters date from 40 years ago with soil as their first bed, gradually changing to compost, wood barks, peat, etc. These supports have high water retention capacities, high buffering capacities, a high microbial population, and substantial adsorption capacity; additionally, they are nutrient rich environments and are inexpensive in comparison to synthetic supports [4]. However, they shrink and clog easily, have high pressure drop, high bed density, and their life-span is between 2-5 years. Since these supports host a diverse microbial population, after a period of growth the accumulation of microorganisms can contribute to the clogging of the biofilter [5]. The use of inorganic or synthetic support in BTFs makes them resistant to crushing that leads to little limitation in bed height, smaller footprints in BTF and much longer longevity than traditional biofilters. BTFs exhibit low pressure drop as a result of the high porosity of the bed. The existence of a free liquid phase makes it easy to control moisture, temperature, pH, accumulation of metabolites such as H_2SO_4 , and provides a continuous supply of nutrients for the microorganisms [6].

Lava rock can be a suitable support in BTFs

for the treatment of H_2S in airstreams. It is abundant and inexpensive, its porous structure gives a large surface area for biofilm attachment and growth, and also provides an acceptable level of bed porosity [7,8]. Chitwood and Deviny observed a considerable amount of weight loss in one kind of lava rock at low pH levels during 110 days of experiments [9]. However, this will not be a problem in BTFs that operate with cultures of *Thiobacillus thioparus* as the optimum pH, as the activity of these cultures is around neutral.

There are several reports of using lava rock in H_2S elimination in biofilters immobilized by activated sludge, *Acidithiobacillus thiooxidans*, and *Acidithiobacillus ferrooxidans* [7, 10], but the present study is the first report of the use of Lava rock to immobilize *T. thioparus*.

In this study the performance of a BTF using a pure culture of *T. thioparus* immobilized on a lava rock for the elimination of H_2S in airstreams at low concentrations was investigated.

2- Materials and methods

2.1 - Microorganisms and media

Thiobacillus thioparus (DSM5368) was obtained from the microbial collection of IROST. The strain was maintained on agar slant (medium DSM 486) containing (in $g L^{-1}$): KH_2PO_4 , 2; K_2HPO_4 , 2; NH_4Cl , 0.4; Na_2CO_3 , 0.4; $MgCl_2.6H_2O$, 0.2; $Na_2S_2O_3.5H_2O$, 5, plus vitamin and trace metals solutions. The inoculum for the biotrickling filter was prepared in liquid culture using a rotating shaker at 150 rpm and 30°C. The liquid medium used in the preparation of the inoculum was DSM-36 and had the following composition: (in $g L^{-1}$): $(NH_4)_2SO_4$, 0.10; K_2HPO_4 , 4.00; KH_2PO_4 , 4.00; $MgSO_4.7H_2O$, 0.10; $CaCl_2$, 0.10; $FeCl_3.6H_2O$, 0.02; $MnSO_4.H_2O$, 0.02; $Na_2S_2O_3.5H_2O$, 10.00; All media were sterilized by autoclaving at 115°C for 20 min. A vitamin solution was sterilized by filtration.

2.2 - Experimental setup

A laboratory scale BTF was constructed and used for the removal of hydrogen sulfide. A schematic of BTF is shown in Figure 1. The total bed height of the BTF was 120 cm with a diameter of 14 cm. The active height of the packed column was 65 cm, resulting in an active volume of 10 liters. Lava rock was used as the support for the microbial population. A Jaw crusher (Denver, Germany) was used to crush the lava rock to 12-25 mm pieces. The filter bed porosity was measured according to the method proposed by Hodge and Devinny [11]. The initial porosity of lava rock was 66%, which is considered to be quite acceptable [8]. The volume of recycled liquid in the vessel was 10 liters. The countercurrent mode of gas/liquid flow was the chosen mode of operation. A solution of 4M NaOH was used to control the pH of the recycled liquid with a peristaltic pump. The pH of BTF was kept constant at pH 6.0 during the experiments. Five sampling ports were located at 15, 31, 46, 55 and 62 centimeters of effective bed

height. 1 liter of recycled medium was replaced with concentrated fresh aqueous mineral medium every 12 hours.

The change in the temperature of air across the height of the BTF was not significant during the main part of the project. During normal steady state operation of the BTF the temperature of air was in the region of 28 -30 °C.

2.3 - Analytical methods

The hydrogen sulfide concentration was determined using a Drager Sensor (Drager Sensor Micropac Plus, Drager Safety, Germany). The pH was determined using a pH meter (Corning M120). Sulfate and thiosulfate analysis were carried out gravimetrically and iodometrically, respectively [12]. Microbial counts were done by serial dilutions of recycled liquid and biofilm samples in distilled water and subsequent plating on various media. Heterotrophs were counted on plate count agar and *T.thioparus* was counted using medium DSM-486 agar plates.

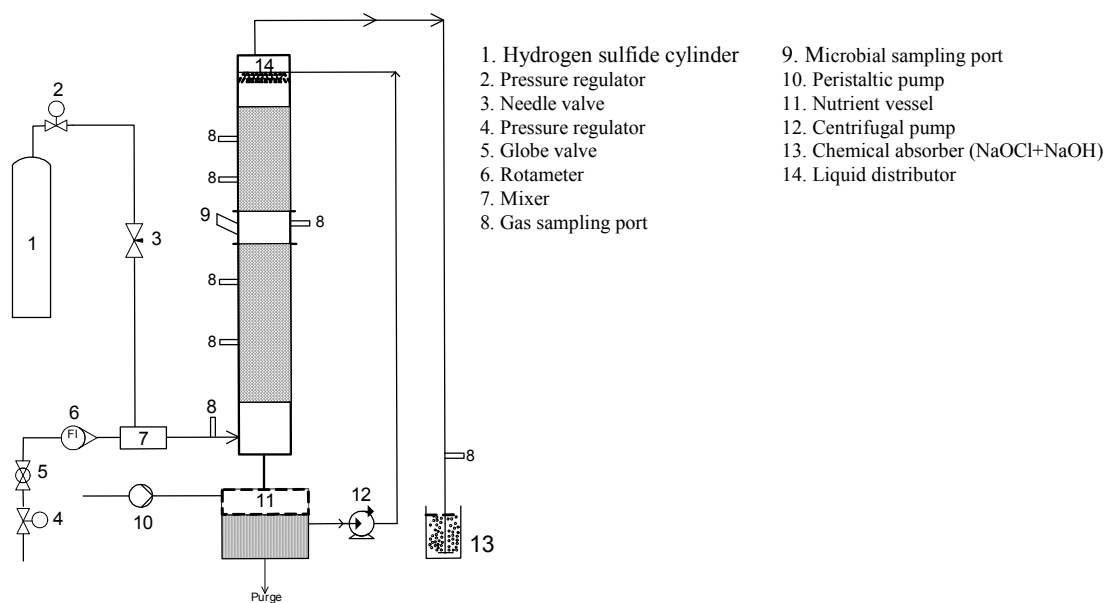


Figure 1. Experimental set up

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To assess the performance of the biotrickling filter, H₂S removal efficiency and elimination capacity were determined at different empty bed residence times and pollutant loadings according to the following equations:

$$EBRT = V/Q \text{ (s)} \quad (1)$$

$$L = \frac{C_{in}}{V} \times Q \text{ (g m}^{-3}\text{h}^{-1}\text{)} \quad (2)$$

$$R.E = \frac{C_{in} - C_{out}}{C_{in}} \times 100 \text{ (\%)} \quad (3)$$

$$E.C = \frac{(C_{in} - C_{out})}{V} \times Q \text{ (gm}^{-3}\text{h}^{-1}\text{)} \quad (4)$$

$$R.E_n = (C_i - C_s) / (C_i - C_x) \quad (5)$$

3- Results and discussion

3.1 - Immobilization and acclimatization

In previous reports the acclimatization of the microbial cultures to the pollutant to be degraded has usually been carried out in the BTF, whereas the immobilization stage has been carried out either in a separate bioreactor or inside the BTF [7,13,15,16].

In the cases where the immobilization step has been done in a bioreactor, the duration of this stage has been reported as 25-30 days; however, in such cases, acclimatization stages are reportedly short (around 3 days), especially for pure cultures [15,16]. For the case when both the immobilization and acclimatization has been carried out in the BTF, the immobilization stages as short as 2-3 days and acclimatization stages around 8 -20 days have been reported, although there have also been reports of combined immobilization-acclimatization as long as 80 days [17, 18].

In the present study both the immobilization and acclimatization were carried out in the BTF, but a slightly different procedure to those previously reported was adopted. The immobilization of BTF was started by

recirculating 10 liters of *T.thioparus* solution containing 1.1×10^8 CFU of bacterial cells at a liquid recirculation rate of 540 mlmin^{-1} (corresponding to SLV of 2.1 mh^{-1}). Since sodium thiosulfate was used in preparation of the inocula [17,18], this substrate, (instead of H₂S) used in some previous works, was supplied to the bacterial culture at this stage.

The consumption of thiosulfate and the pH change during the immobilization stage is shown in Fig. 2. The pH was adjusted to 6 every day. The complete consumption of 100 g of thiosulfate in 24 hours occurred from the second day onwards. After 3 days the acclimatization stage began with the elimination of thiosulfate from the recirculating media and the introduction of an air stream containing 72 ppm of H₂S at EBRT of 60 seconds. It took four days to reach steady state conditions in the outlet and at different heights of BTF. The removal efficiency changed from 69.4% on the first day to 100% on the fourth day and the pattern of H₂S removal did not change at the outlet and different heights of BTF after the fourth day.

Comparison of the present work with previous reports shows that the length of the immobilization stage was much shorter than most previous reports [7,13,15,16]. It was comparable to the works of Deshusses et al., while the acclimatization stage was comparable to the work of Cho et al but shorter than Deshusses' and other works [7,18]. Deshusses et al. used activated sludge in all of their experiments instead of pure cultures and this may be the reason for the longer duration of their acclimatization stage [18].

It has been shown that above a certain concentration sulfate has an inhibitory effect on H₂S elimination. Different minimum inhibitory concentrations of sulfate ion, in the range $1.9 - 7 \text{ glit}^{-1}$, have been reported [19,20]. In the present study the concentration of sulfate was not allowed to increase above 5 g/lit, and no inhibitory effect was observed.

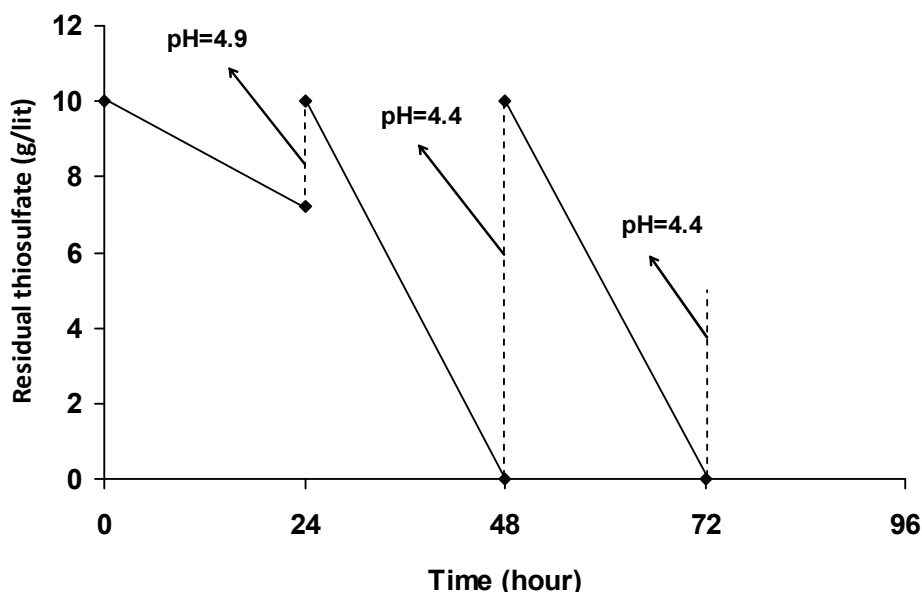


Figure 2. Thiosulfate consumption and pH change during immobilization

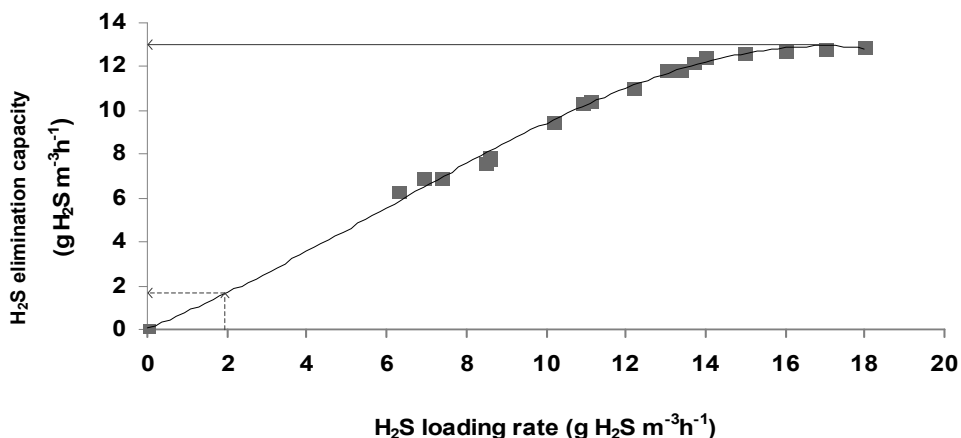
3.2 - Effect of loading on hydrogen sulfide removal

In order to increase the loading of a BTF either the inlet concentration of the pollutant was increased or EBRT was decreased. Figure 3 shows the effect of increasing the loading rate by decreasing EBRT on H_2S elimination capacity at constant H_2S concentrations of 35 (Fig. 3a) and 65 ppm (Fig. 3b). The effect of increasing the loading rate by increasing H_2S inlet concentration on H_2S elimination capacity at constant EBRT of 10 seconds and 12 seconds is presented in Figure 4. All of the curves can be divided into three regions:

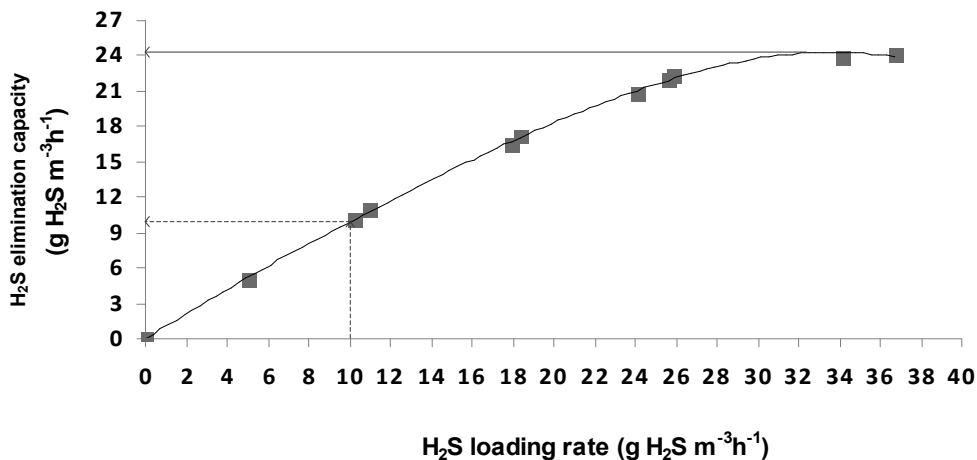
In the first region or first order regime the elimination capacity and loading of H_2S are the same and the removal is complete. In the second region, the breakthrough of H_2S occurs (for 35 and 65 ppm it starts around 2 and 10 $gm^{-3}h^{-1}$, respectively). In this region, the elimination capacity of H_2S increases to a lesser extent than the loading and the removal efficiency starts to decrease below 100%. In the third region or zero order

regime, the BTF reaches its maximum elimination capacity (EC_{max}) and an increase in loading does not lead to increase in elimination capacity (for 35 and 65 ppm it is around 13 and 24 $gm^{-3}h^{-1}$, respectively).

Fig. 3 shows that EC_{max} increases with an increase in H_2S concentration from 35 ppm to 65 ppm. The same trend was observed in the experiments at H_2S concentrations of 25, 45, 55 and 75 ppm (results not presented); EC_{max} increased from 12 g of $H_2S m^{-3} h^{-1}$ at the inlet concentration of 25ppm to 28.5 g of $H_2S m^{-3} h^{-1}$ at the inlet concentration of 75 ppm. Results presented in Figure 4 show that the EC_{max} for EBRT of 10 and 12 sec is around 32 and 27 $gm^{-3}h^{-1}$, respectively. These are in line with the findings of Martin et al [21] and Deshusses [6]; however, Deviny et al. stated that maximum elimination capacity is essentially constant for a wide range of concentrations and EBRTs, but these authors employed very high inlet pollutant concentrations in their work [21, 22].



(a)

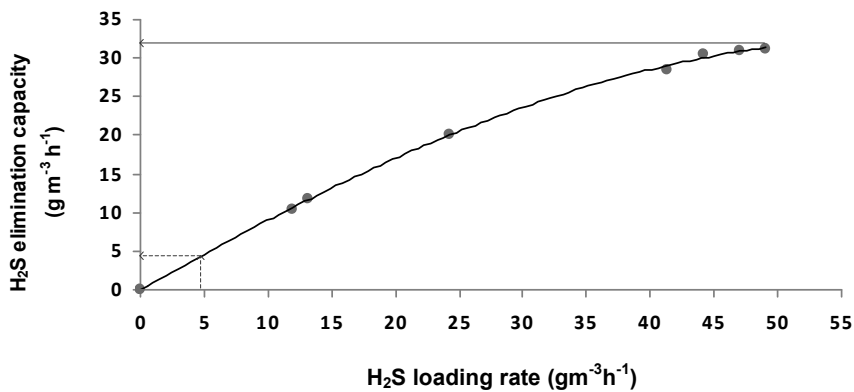


(b)

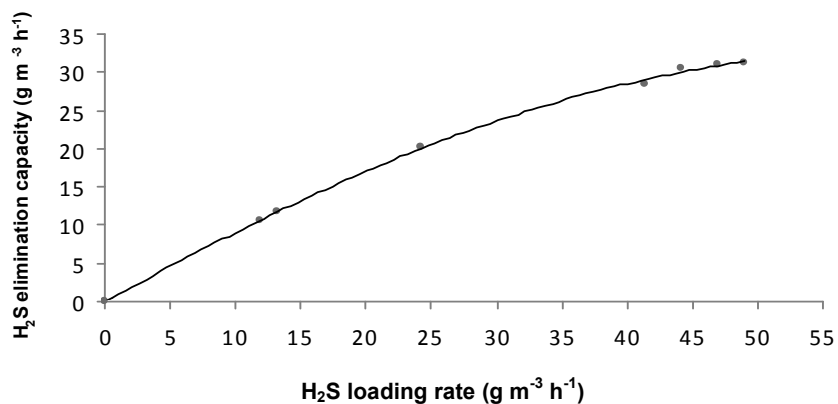
Figure 3. Effect of change in H₂S loading rate (as a result of change in EBRT) at a constant H₂S inlet concentration of a) 35ppm and b) 65ppm on E.C in the BTF

Figure 5 shows the removal efficiency of H₂S as a function of loading at different H₂S concentrations. It can be seen that the removal efficiency decreases more rapidly at low H₂S concentrations. This highlights the effect of EBRT on the elimination of H₂S; at loading of 12 g of H₂S m⁻³ h⁻¹ the removal efficiency for 25 ppm H₂S (EBRT of 10.5 sec) is 88.5%, whereas for 65 ppm H₂S

(EBRT of 27 sec) at the same loading removal efficiency is equal to 98.3%. It has previously been demonstrated that increasing the loading rate by decreasing EBRT decreases removal efficiency more than if the same increase in loading rate was achieved by increasing the inlet concentration of H₂S [15].



(a)



(b)

Figure 4. Effect of increase in H₂S inlet concentration at EBRT of a) 10 seconds and b) 12 seconds on E.C. in the BTF

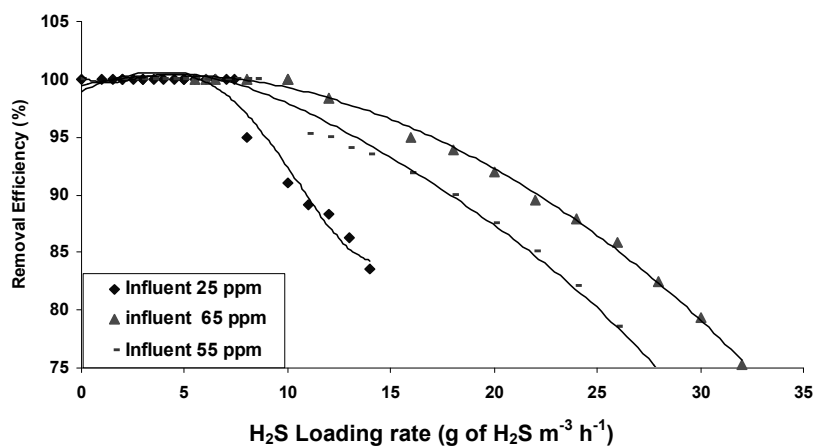


Figure 5. Removal efficiency of H₂S as a function of H₂S loading rate in the BTF

In Figure 6 the results obtained in the present BTF are compared with those obtained by Chung et al. [23], who used calcium alginate beads to immobilize *T. thioparus*. It can be seen that higher R.E.s were obtained in the present study at lower EBRTs and equal H₂S concentrations (60 ppm). Different methods of immobilization might be the reason for the different performances.

In Figure 7 the elimination capacity versus loading rate for the present study is compared with the study by Jin et al. [19], who used polypropylene pall rings as the microbial support and activated sludge as their microbial population. At the same H₂S inlet

concentration (55 ppm) and higher EBRTs, slightly higher R.E. have been reported by Jin et al [19], whereas higher R.E. has been obtained in this study at lower EBRTs and the same inlet concentrations.

On the other hand, the R.E. reported in several other investigations [7 ,13 ,10 ,24] is considerably higher than those obtained in the present work, which could be related to the use of better strains of *Thiobacillus* and/or synthetic supports like polyurethane foam with higher specific surface area and appreciable bed porosity, and/or higher concentrations of CO₂ in the air stream.

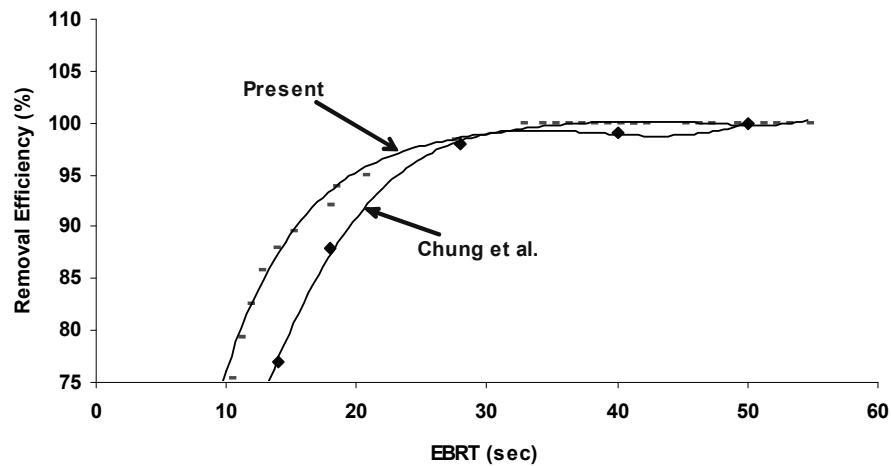


Figure 6. Comparison of present work with the study of Chung et al. [15] at constant C_{Gi} of 60 ppm

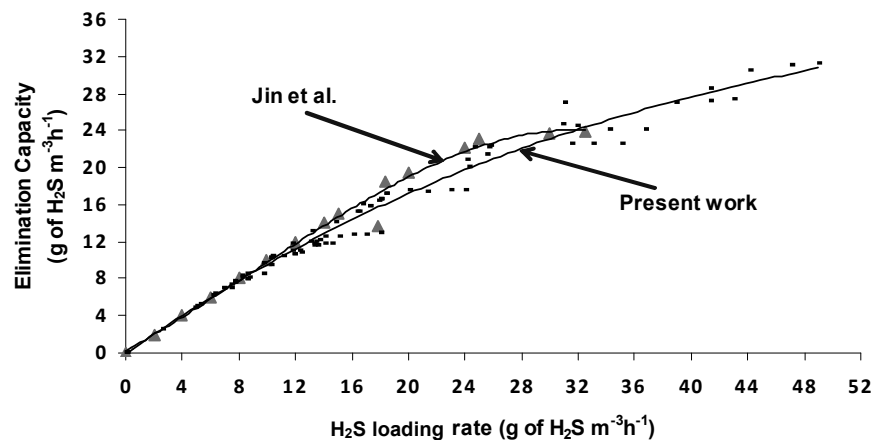


Figure 7. Comparison of the results of present work with the study of Jin et al. [19]

3.3- Effect of superficial liquid velocity on H_2S removal

Figure 8 shows the removal efficiency versus EBRT of pollutant at two different superficial liquid velocities (SLV). It can be seen that increasing SLV from 0.98 m h^{-1} to 1.95 m h^{-1} distinctly decreases removal efficiency of H_2S under otherwise similar conditions. Chou and Huang concluded that for low and average concentrations of soluble pollutants (like H_2S) SLV has no effect on the elimination capacity of the pollutant [25]. This has been observed in the study by Gabriel and Deshusses, who used very low EBRTs of around 2 seconds in their BTF [24]. However, in the current study and several other investigations the negative and positive effect of SLV on the removal of H_2S has been demonstrated [20,19]; Kim and Deshusses [20] encountered the positive effect of SLV on the elimination of H_2S , most probably due to the extremely low EBRTs used in their work. The positive effect of SLV on the elimination of other pollutants like dichloromethane has been demonstrated [22,24,26,27]. The negative effect of SLV on R.E. observed in the present

work is probably related to the fact that with increasing SLV the thickness of the liquid film surrounding the biofilm increases, and therefore the liquid film resistance, increases.

3.4- Effect of EBRT and C_{in} on the elimination of H_2S at different bed heights

Choosing the bed depth is important in designing a BTF. The height and cross sectional area of the bed determine the volume of the bed and consequently the EBRT. The removal of the pollutant would not be perfect without sufficient EBRT.

In this study the removal of H_2S at different heights of BTF was determined. Figure 9 shows normal removal efficiency versus height of the bed at a constant inlet concentration of 57 ppm and different EBRTs of 30, 17, 9 seconds. At EBRT of 30 sec, 86% of elimination occurred in 71% of bed height, but when EBRT was decreased to 9 sec, 67% of elimination occurred in the same height of the bed. This trend was observed for the other inlet concentrations of pollutant (data not reported).

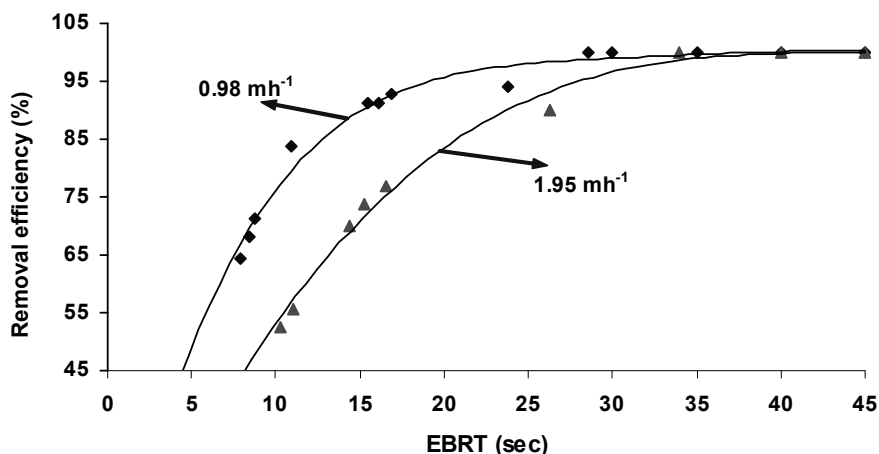


Figure 8. Effect of EBRT on the H_2S removal efficiency at constant H_2S inlet concentration of 55 ppm at two different superficial liquid velocities

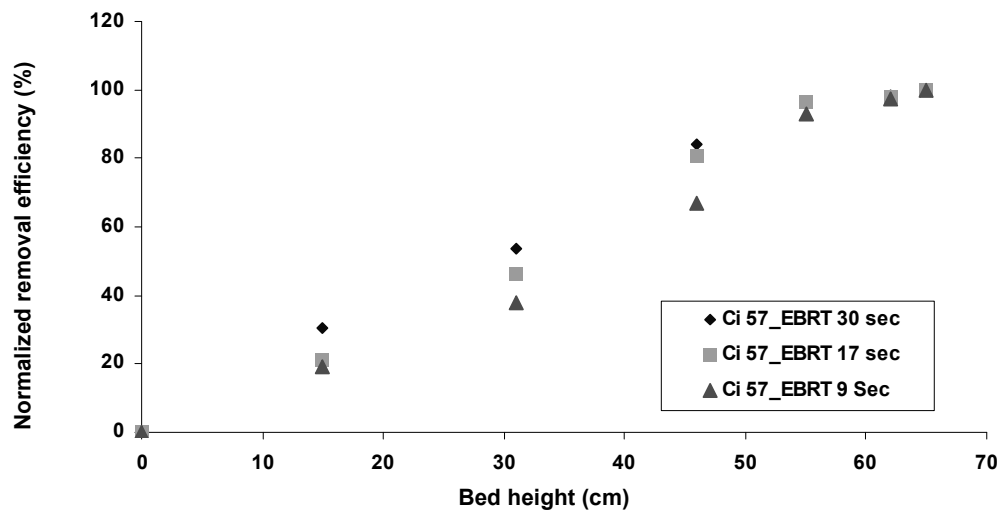


Figure 9. Normalized removal efficiency versus effective bed height of BTF at constant C_{in} of 57 ppm and different EBRTs

Martin et al. observed that with an EBRT of 6.5 sec, 97% of elimination occurs in the first 46 cm of the bed [21]. Shojaosadati and Elyasi, while working with a compost biofilter at an EBRT of 92 sec and inlet H_2S concentration of 120 ppm, saw that more than 50% of elimination occurs in the first 25% of bed height [28]. Elias et al. divided a compost biofilter into three equal parts and observed that at EBRTs in the range 13.5 to 27 sec, and H_2S inlet concentrations in the range 45 to 248 ppm, most of the elimination occurs in the first 75% of biofilter height [4]. Jin et al. [29] observed that increasing concentration of pollutant leads to a more homogenous elimination in the height of BTF. Jones et al. investigated the elimination of H_2S and NH_3 at different heights of BTF in two BTFs with different supports, one compost and the other synthetic and found a marked difference in the elimination at different heights of biofilters for the two beds [30].

3.5 - System response after upsets

System upsets like flooding, transient and shock loadings, non-use periods, bed drying and decrease in pH of the system are some of

the inevitable unsteady conditions that occur in any BTF and challenge the robustness of it [31].

In the present study, because of the blockage of the outlet of recirculating liquid, a flooding occurred for a period of 1 hour. Removal efficiency of BTF decreased from 93% to 82% and did not recover, even after one week from the incident. During the same period, a decrease in environmental temperature of the order of 10°C was observed. A heater was installed in the vessel to keep the temperature of recirculating liquid at around 28°C. Upon its setup the performance improved but did not fully recover even after a week. Even re-inoculating the bed did not rectify the situation completely. After the inoculation, thiosulfate was consumed in the BTF, but to a lesser extent, and the pH started to increase instead of decreasing. Consumption of thiosulfate using *Thiobacillus* species would lead to a decrease in pH rather than an increase. The increase in pH was a sign of contamination in BTF. The contamination of the system was confirmed by plate count techniques on nutrient agar and DSMZ-486 from the recirculating liquid and biofilm and

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was considered to be the reason for the decrease in performance, so the system was dismantled. It should be pointed out that periodic observations of contamination during the course of operation of the BTF did indicate contamination of the system with mainly heterotrophic bacteria, but this contamination became dominant only after the abovementioned system upsets.

Kanagawa and Mikami, while using *T. thioparus* for the elimination of H₂S under non-sterile conditions, encountered a widespread heterotrophic contamination in the system [32]. The contamination did not affect the performance of the biofilter and system upsets did not occur to challenge the robustness of the system as happened in the present work. No report on the contamination of systems using *A. thiooxidans* has been reported so far. Neutral pH used in this study and in the work of Kanagawa and Mikami allows for the presence of such contaminations in the system. An advantage of using extremophiles such as *A. thiooxidans* is the absence of such widespread contamination in the system.

4- Conclusions

A BTF using lava rock as the support was immobilized with *T. thioparus* and had an acceptable level of performance during steady conditions. A short immobilization and acclimatization process was achieved in comparison with other works in this field. The negative effect of SLV on the performance of BTF in the range of the inlet concentration and EBRT studied was demonstrated. Decreasing EBRT led to a slight increase in the homogeneity of the H₂S elimination across the bed height and had a more pronounced effect than increasing C_{Gi} in the range of EBRT and C_{Gi} studied. BTF did not recover from an upset in the system and widespread contamination was observed.

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Nomenclature

C _{in}	Inlet gas concentration (ppm)
C _{out}	outlet gas concentration (ppm)
EBRT	Empty bed residence time (s)
E.C	Elimination capacity (gm ⁻³ h ⁻¹)
L	Loading rate (g m ⁻³ h ⁻¹)
R.E.	Removal efficiency (%)
R.E. _n	Normalized R.E. (%)
SLV	Liquid superficial velocity (mh ⁻¹)

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