Operation of Biofilter with Mixed Agricultural Residue as Filter Material: Effects of Humidification and Inlet Hydrogen Sulfide Volume Fraction on the Performance

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Mixed agricultural residue, inoculated with aerobic sulfide oxidizing microbial consortium, was used as biofilter media to study the removal efficiency ($\eta_R$) of hydrogen sulfide ($H_2S$). The effect of humidification and inlet $H_2S$ volume fraction on the performance of biofilter was also investigated. A 3.9 l bench scale biofilter was continuously operated to treat air containing $H_2S$ gas in the range of $\varphi = 275$ to $2833 \cdot 10^{-6}$ for 150 days. $\eta_R$ of 99 % was obtained at $\varphi = 2020 \cdot 10^{-6}$ of inlet $H_2S$ volume fraction during continuous operation with humidification. However, $\eta_R$ dropped to 51 % when the inlet $H_2S$ fraction increased beyond $\varphi = 2020 \cdot 10^{-6}$. A maximum elimination productivity of 91 g m$^{-3}$ H$_2$S of filter bed h$^{-1}$ and inlet mass loading rate of 91 g m$^{-3}$ H$_2$S of filter bed h$^{-1}$ was achieved when the $t_{EBRT}$ was in the range of 15 – 155 s. The filter was operated without the humidifier at the inlet $H_2S$ fraction of $\varphi = 2020 \cdot 10^{-6}$ and observed that $\eta_R$ dropped to 51 % due to the drop in moisture of the filter material. However, $\eta_R$ could be recovered up to 83 % upon reintroduction of humidifier into the circuit of biofiltration process indicating, that humidification of the waste gas was essential to achieve the highest possible $\eta_R$ at particular inlet $H_2S$ fraction. The filter recorded $\eta_R$ above 99 % even in the acidic phase at $\varphi = 2020 \cdot 10^{-6}$ of inlet $H_2S$ fraction without intermittent washing. The gas dispersion characteristics were comparable to the previously tested bed materials, offering low-pressure drop across the biofilter in the range of 21 – 74 mm of H$_2$O per meter height of packing material.

Key words: Biofiltration, mixed agriculture waste, H$_2$S, moisture, humidifier, pressure drop, sulfur oxidizing bacteria

Introduction

Increasing regulations made it necessary to apply air pollution control measures for the treatment of $H_2S$ gas from different sources, especially for the industries, which are located near prime residential areas.$^{1,2}$ Traditionally, various physical and chemical methods like adsorption, absorption, condensation, oxidation etc. have been utilized for the removal of $H_2S$ from industrial waste gases$^{3-6}$ but, these conventional processes are expensive and energy intensive. In the past few decades biological control of air pollution is gaining interest because of its obvious advantages besides being competitive.$^{7-11}$ Also, biological methods are environmentally benign, because in physico-chemical methods the contaminant is simply transferred from one phase to another, whereas in biological method contaminant is degradable into innocuous products. At this juncture biofiltration process is emerging as economically cheap, ease in operation, and environmentally suitable for waste air streams containing biodegradable pollutant.$^{12-16}$ Biofilters are studied at great length for the removal of $H_2S$ and there are full-scale biofilters based on the pilot scale studies at industrial level.$^{17}$

Various natural materials and industrial biological residues such as peat, coconut coir pith, sugar cane bagasse, compost and wood bark are frequently used as filter material in biofilters.$^{3,18-20}$ A number of biofiltration studies for $H_2S$ removal have been carried out using some specific packing materials as carriers, including fibrous peat, compost, activated carbon, and recently, immobilized micro-organism.$^{21-25}$ Such materials offer many advantages including their availability at low prices, a rich variety of indigenous microbial species content, and a propitious biological medium for microbial growth and activity, especially, for their nutrient supply.$^{26}$ Besides this, biofiltration would be cheaper, if locally available natural material could be used as packing material.

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The natural materials that were used as a bed material previously as stated above were tested for various parameters like compaction characteristics, moisture retention capacity, etc. during the biofiltration of pollutants. Therefore, locally available natural material that is intended for use as bed material has to be investigated for the aforesaid parameters. Objective of the present work was to assess the performance of mixed agriculture residue as a bed material for the biofiltration of air contaminated with H₂S and to study the effect of humidification and inlet H₂S fraction.

Materials and Methods

Biofilter setup

Biofilter: Glass column having 0.08 m internal diameter and 1.2 m height was used as biofilter column. The height of the filter media was 0.77 m. Four equidistant sampling ports were provided along the length of the filter media (Port 1 at bottom, Port 4 at the top and Port 2 and Port 3 in between.

Humidifier: Glass column having internal diameter of 0.06 m and height of 1 m. Water was sprayed at the top through fine nozzles with 1/16 HP self-priming centrifugal pump and the air was supplied counter currently form the bottom of the humidifier. The water collected at the bottom of the humidifier was continuously recycled to the top of the humidifier. Water loss due to evaporation was made up as per requirement.

H₂S generation unit: H₂S gas was generated by the addition of sodium sulfide and hydrochloric acid solutions in a glass column. The solutions (w = 1 % Na₂S and 0.1 mol l⁻¹ HCl) were taken separately in two glass reservoirs with stoppers for flow control and trickled at desired flow rate to a glass jar. The H₂S gas formed in the glass jar was collected by passing air from the cylinder to the bottom of the jar. The airflow was regulated from the humidifier. The airflow rate was monitored with the help of a calibrated rotameter in the range of 0.09 m³ h⁻¹ to 0.927 m³ h⁻¹.

Inoculum

Aerobic mixed culture was collected from an activated sludge process reactor treating distillery spent wash and passed through a mesh to remove foreign material. The sludge was having a TSS (Total Suspended Solids) of 48.0 g l⁻¹ and VSS (Volatile Suspended Solids) of 25.0 g l⁻¹. This sludge was mixed with Thiobacillus sp. media and kept in an aerobic batch reactor with continuous sterile air supply from an air cylinder. The aeration was continued for a period of t = 15 d by replacing the Thiobacillus sp. media every alternate day. The enriched Thiobacillus sp. culture developed was used as inoculum for mixing with the filter material.

Microbial count: Cell numbers were measured at the start and end of the experiment. About 5 g of packing material was used as sample and mixed thoroughly in sterile distilled water kept on shaker at n = 150 min⁻¹ for 8 h. Serially diluted suspension was spread onto the thiosulfate mineral media for sulfur oxidizing bacteria. Then the flasks were incubated at 30±2 °C for a period of 7 d. The cell numbers were expressed as colony forming units (cfu g⁻¹), per mass of sample.

Filter material

Mixed agriculture residue, which was used as bed material, was composed of rice husk, sawdust, bagasse, and coconut coir pith in equal proportion. The filter material was mixed with inoculum (1 g per every 10 g of filter material) and CaCO₃ (500 mg per every 10 mg of filter material). The above material was packed in the filter in layers of 0.25 m height and on each layer coconut coir was distributed evenly to avoid the compaction of the bed.

Analytical methods

The inlet and outlet gas from the biofilter was analyzed for H₂S by the Tutweiler’s apparatus. Filter media was characterized for various parameters (Tab 1) and filter media samples were collected periodically from the sampling ports and analyzed for moisture content as per standard methods. The pH of the media was also analyzed by soaking one part of filter material in ten parts of doubly distilled water for 30 min.

Pressure drop measurement: The pressure drop was measured by connecting a U-tube manometer across the biofilter.

Experimentation

Phase I: In the first phase, experiments were carried out as shown in Fig 1 with the humidifier in the circuit. H₂S volume fraction was varied from φ = 275 · 10⁻⁶ to 2833 · 10⁻⁶ in a stepwise manner and biofilter was operated for 90 d. The desired fraction of H₂S gas in the air mixture was obtained by manipulating the air flow and flow rate of H₂S generating chemical solutions (w = 1 % Na₂S and 1 mol l⁻¹ HCl)

Phase II: In the second phase, experiments were carried out without the humidifier in the process. H₂S fraction was kept constant at φ = 2020 · 10⁻⁶ with the air flow rate of 0.128 m³ h⁻¹ and biofilter was operated for 35 d.
**Phase III:** In the third phase, experiments were carried out by again introducing the humidifier in the circuit as shown in Fig. 1. However, in this phase H$_2$S fraction was kept constant at fraction of $\varphi = 2020 \cdot 10^{-6}$ and biofilter was operated for 25 d.

**Results and Discussions**

**Packing Material**

The agricultural residue that was used as filter material for the present studies consists of rice husk, sugar cane bagasse, coconut coir pith, and saw dust. These are the waste products of native agro processing industries. Equal proportions of aforesaid filter materials were mixed and analyzed for its physical parameters like pH, moisture, bulk density and particle diameter before the experiment, and tabulated in Tab. 1. Tab 1 shows that the mixed filter material was having 0.112 g cm$^{-3}$ of bulk density, 28% moisture and pH of 7.2 at atmospheric temperature (30 ± 2 °C). The performance data of the biofilter at stable conditions for all the inlet H$_2$S fraction studies were tabulated in Tab 2. The filter material could withstand the low pH of 4.1 and perform to the extent of 83% $\eta_R$ at inlet H$_2$S fraction of $\varphi = 2020 \cdot 10^{-6}$. The filter material regained its moisture when the humidifier was reintroduced after suspension to the extent of 50%, which helped in improving the $\eta_R$ of the filter to 83% from 52%. Mixed agricultural waste was having less compaction characteristics compared with other filter materials cited previously as it offered low-pressure drop in the range of 21–74 mm H$_2$O.

![Biofiltration process flow diagram with humidifier](image-url)
per meter of the packing height throughout the filter operation allowing gas to disperse efficiently for maximizing the removal efficiency. Preliminary properties of the mixed agricultural waste were comparable to the properties of other bed materials that were used previously by earlier researchers and it could be one of the alternate biofilter bed materials like other natural fibrous material as it exhibited comparable performance (Tab. 2) for H₂S removal.

### Pressure drop

Airflow rates were changed in order to change the H₂S inlet fraction to the filter during the course of operation of the biofilter. Superficial gas velocity at each airflow rate was determined and the pressure drop at each superficial gas velocity was measured. Variation of pressure drop across the biofilter at each superficial gas velocity was shown in Fig 2. Fig. 2 shows that the pressure drop was in the range of 21 to 74 mm H₂O per m of packing material. Elias et al. used pig manure and saw dust as a packing material and reported pressure drop values in the range of 15 to 460 Pa m⁻¹. Yang and Allen reported pressure drop values in the range of 500 to 1000 Pa m⁻¹ for compost and they also suggested replacement of filter material when the pressure drop exceeds 2.5 kPa per unit length of packing. The results obtained in our study was in the range of 21 (206 Pa m⁻¹) to 74 (726 Pa m⁻¹) mm H₂O per m of packing material below the critical values prescribed by earlier authors and comparable to the values obtained for saw dust and pig manure and compost. Previously, porous ceramics, calcinated cristobalite, calcinated & formed obsidian, granulated and calcinated soil native material were also used as packing in the biofiltration studies and pres-

### Table 2 – Performance data of biofilter

<table>
<thead>
<tr>
<th>Sr. No</th>
<th>$Q$ / m³/hr⁻¹</th>
<th>$\rho_i$ / 10⁻⁶</th>
<th>$\rho_o$ / 10⁻⁶</th>
<th>$t_{EBRT}$ / s</th>
<th>Operational period, days</th>
<th>Efficiency, $\eta_R$ / %</th>
<th>moisture, $u$ / %</th>
<th>pH</th>
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<td>10*</td>
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<td>49</td>
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* With humidifier, † Without humidifier.

![Figure 2](image-url) — Variation of pressure drop across the biofilter with superficial velocity.
sure drops of 6.1, 21.7, 15.5 and 31.1 mm H2O per m of packing material, respectively, were reported. The data obtained for mixed agriculture residue used in the present study was comparable to the above values also. This indicates that agriculture residue used in the biofilter, as inert support material, was good in terms of maintaining the less compaction characteristics.

**Microbial count**

The biofilter media showed the existence of generic class of *Thiobacillus sp.* Initially the microbial count of the composite sample of the bed material was $1.1 \times 10^3$ cfu g$^{-1}$ of sample. This might be due to the fact that inoculum was obtained from the existing effluent treatment plant, treating high sulfate containing distillery spent wash and was also cultured for almost 15 d. At the end of the experiment the *Thiobacillus sp.* count of the composite sample of bed material increased to $13 \times 10^5$ cfu g$^{-1}$ and the *Thiobacillus sp.* found to be gram negative. It was evident from the table 2 that $\eta_R$ was in the range 90–95 % even though the filter was operated under acidic pH. Earlier researchers established that this class of bacteria could survive under acidic pH$^{20,32}$ and similar removal efficiencies were also reported but with frequent washing of filter bed with water.$^{32}$ Frequent washing of filter bed results in compaction of bed and growth of anaerobic bacteria.$^{26}$ Washing of the filter bed was avoided in the present study to observe the performance in terms of pH and $\eta_R$ of the biofilter. In the present studies pH fell from 8.9 to 4.1 with net increase in colony count of $1299 \times 10^3$ cfu g$^{-1}$ of sample, and $\eta_R$ in the range of 90–95 % was obtained without washing the filter bed.

**Effect of pH on removal efficiency**

The variation of pH (pH of the port 1 filter material sample) and H$_2$S removal efficiency, during the course of operation of the biofilter, was plotted and shown in Fig. 3. Fig. 3 and Tab. 2 shows that pH fell from 8.9 to 4.9 during initial 90 d of operation of biofilter when inlet H$_2$S volume fraction was varied in the range of $\varphi = 275-2833 \times 10^{-6}$ and $\eta_R$ was in the range of 99.5–10 % during this phase of operation. Subsequently when the biofilter was operated without humidifier for 35 d at inlet fraction of $\varphi = 2020 \times 10^{-6}$, the pH fell to 4.3 from 4.9 and $\eta_R$ in this phase was in the range of 50.7–99.5 %. In the last phase of operation of the filter for 25 d, with humidifier in the circuit at $\varphi = 2020 \times 10^{-6}$ of inlet H$_2$S fraction, pH fell to 4.1 from 4.3 and $\eta_R$ increased to 83 % from 51.1 %. The above results indicated that $\eta_R$ was independent of pH and the $\eta_R$ was in the range of ~99.5 % even in the acidic phase. Similar performance for other bed materials were reported previously$^{20,24,25,32}$ but with intermittent washing of the biofilter. Our above finding was for mixed agricultural for which there are no previous reports. The stable performance of the filter without washing obtained in the present study was useful as the neutralizing chemical, that were being used during operation of the biofilter, could be avoided to maintain the pH$^{38}$ and bed compaction, that was observed in previous studies due to intermittent washing could also be avoided.

After the termination of the experiments (i.e after 150 d), the pH of filter material in all the ports was analyzed. Fig. 4 shows that pH was lower in lower ports (4.1) of the bed in comparison with the upper ports (6.2) of the bed. As the air containing H$_2$S moves along the length of the reactor, lower part of the bed of the reactor was getting exposed to the high fraction of the H$_2$S and due to the availability of higher driving force (in terms of net H$_2$S fraction) in the lower part of the bed most of the reaction might be taking place in the bottom part of the reactor. The phenomenon of the lower pH in bottom port and higher pH in the upper could be attributed to this.

![Fig. 3](image1) Variation in pH at bottom port of the biofilter and removal yield of biofilter during operation period

![Fig. 4](image2) pH at different ports along the biofilter at the end of 150 days of biofilter operation. Port 1 is the bottom port, Port 4 is at the top, Port 2 and Port 3 is in between along the length of the filter.
Effect of inlet H$_2$S volume fraction and humidification on removal efficiency

In order, to evaluate the effect of inlet H$_2$S fraction on filter $\eta_R$, the filter was operated at different inlet H$_2$S fraction in the range of $\varphi = 275-2833 \cdot 10^{-6}$. The variation of inlet and outlet fraction of H$_2$S and removal $\eta_R$ during the course of operation of filter for 150 d was shown in Fig. 5. Initially the filter was started with $\varphi = 275 \cdot 10^{-6}$ of H$_2$S. At this fraction the filter was operated for 15 d. During this period $\eta_R$ was in the range of 50–100 %. The $\eta_R$ was 50 % initially and increased steadily as operational period increased. In the first 6 d of filter operation the $\eta_R$ was in the range of 50–91 %. The efficiency increased to 100 % after 12 d of filter operation. The initial 12 d period could be considered as a start up period for initial growth of biofilm and acclimatization of the microbial culture as reported previously. Further, the filter was operated continuously by progressively enhancing the inlet fraction. At each fraction the filter was operated to get stable $\eta_R$. As the inlet concentration increased, the time taken by the filter to reach steady state $\eta_R$ was also increased (Tab. 2).

![Fig. 5 - Change in removal efficiency with change in inlet H$_2$S volume fraction during the entire 150 days of biofilter operation](image)

Fig. 5 and the Tab. 2 shows that till the inlet fraction was $\varphi = 477 \cdot 10^{-6}$ (27 days of filter operation), the $\eta_R$ of H$_2$S was 100 %. The $\eta_R$ dropped in the range of 99.4 to 99.7 %, when the inlet fraction of H$_2$S was increased in the range of $\varphi = 570-2020 \cdot 10^{-6}$ from 28th to 65th day of filter operation, and exit fraction of H$_2$S was varied in the range of 2 to 13 \cdot 10^{-6}. In the subsequent phase of 15 d, at inlet fraction of $\varphi = 2833 \cdot 10^{-6}$, $\eta_R$ further dropped to 95 % and outlet fraction rose to $\rho = 140 \cdot 10^{-6}$. The outlet fraction of $\rho_o = 13 \cdot 10^{-6}$ which was obtained at inlet fraction of $\rho_i = 2020 \cdot 10^{-6}$ which was in the acceptable range of H$_2$S discharge standards as per Central Pollution Control Board of India compared to the $\rho_o = 140 \cdot 10^{-6}$, which was obtained at $\rho_i = 2833 \cdot 10^{-6}$ inlet fraction. Therefore, the inlet fraction was brought back to the previous value of $\varphi = 2020 \cdot 10^{-6}$ and operated the filter at that value for 10 d (81th to 90th d). In this period of operation, $\eta_R$ of 99.5 % and exit H$_2$S fraction of $\varphi = 10 \cdot 10^{-6}$ was obtained reproducing the previous performance at $\varphi = 2020 \cdot 10^{-6}$ of inlet H$_2$S fraction. The performance indicated that $\eta_R$ was strongly dependent on the H$_2$S inlet fraction and for the present case of biofilter configuration, a maximum inlet H$_2$S fraction of $\varphi = 2020 \cdot 10^{-6}$ was only possible for steady state effective performance of the filter with in the acceptable range of $\varphi = 40 \cdot 10^{-6}$ of outlet H$_2$S fraction.

It was evident from the figure 5 and table 2 that $\eta_R$ dropped to 50 % in the second phase of operation when the biofilter was operated without humidifier at $\varphi = 2020 \cdot 10^{-6}$ of inlet H$_2$S fraction. Fig. 5 and Tab. 2 also shows that in the last phase of operation of the filter for 25 d with humidification, $\eta_R$ increased to 83% at $\varphi = 2020 \cdot 10^{-6}$ of inlet H$_2$S fraction. The above results revealed that humidification was essential to achieve the maximum $\eta_R$ at particular fraction and beyond $\varphi = 2020 \cdot 10^{-6}$ of inlet H$_2$S fraction even with humidification, obtaining exit fraction of H$_2$S in the acceptable limits of $\varphi = 40 \cdot 10^{-6}$ was not possible for the present configuration of biofilter.

Effect of humidification and empty bed residence time ($t_{EBRT}$) on removal efficiency

As stated earlier, filter could be operated stably at $\varphi = 2020 \cdot 10^{-6}$ of inlet H$_2$S fraction with 99.5 % efficiency and $\varphi = 10 \cdot 10^{-6}$ of exit H$_2$S fraction with humidification. This is equal to the loading of 91 g m$^{-3}$ h$^{-1}$ H$_2$S and of 33 m$^3$ m$^{-3}$ h$^{-1}$ of filter. At this junction in order to establish the effects of humidification and moisture ($u$), the filter was operated bypassing the humidifier in the circuit. Trends of $t_{EBRT}$, $u$ and $\eta_R$ during the course of operation of filter for 150 days was drawn and shown in Fig. 6. Fig. 6 shows that until 90th day of operation with the humidifier in the circuit, $\eta_R$ was more than 99 % and moisture was above $u = 65 \, \%$. Even with the humidification, the $\eta_R$ dropped to 83 % when the inlet H$_2$S fraction was increased to $\varphi = 2833 \cdot 10^{-6}$, even though moisture was around $u = 65 \, \%$, due to the effect of high inlet concentration as explained earlier.

In the second phase of operation without humidifier in the circuit, air having approximately $\varphi = 2020 \cdot 10^{-6}$ of H$_2$S, was passed through the biofilter for 35 d. Fig. 6 shows that efficiency of 90 to 99 % was achieved during the first 21 days of biofilter operation and moisture was in the range of 47 to 65 %. Subsequently, for the next 14 days of operation the $\mu$ gradually decreased to 35 % and $\eta_R$ of the biofilter dropped to 51 % from a maximum $\eta_R$.
of 90%. In the present study, during 35 d of biofilter operation without humidifier, the moisture of the filter material had come down to 35% from 99%. Therefore, it was evident from the above results that a minimum moisture of 50-65% was very much essential for efficient operation of biofilter, so that sufficient aqueous layer on the inert material to the extent of H₂S gas phase transformation and microbial activity was available in the biofilter. Our observations support the results of the earlier research work\textsuperscript{15-36} that moisture in the range of 55 to 65% was optimum for the removal of pollutants from waste air streams.

Fig. 6 and Tab. 2 shows that the $t_{EBRT}$ varied in the range of $t = 15-155$ s during the operation of the filter with inlet H₂S fraction in the range of $\varphi = 275-2833 \times 10^{-6}$ at constant loading. The results from the above figure and table revealed that at $\varphi = 2020 \times 10^{-6}$ of inlet H₂S fraction, when the filter was operated with humidifier, $\eta_R$ and $\mu$ were 99.5% and 65%, respectively, whereas the $\eta_R$ and $\mu$ dropped to 50.7% and 35%, respectively, when the filter was operated without humidifier even though same $t_{EBRT}$ of 109 s was maintained. This shows that moisture was having pronounced effect than $t_{EBRT}$ on the filter performance.

**Conclusion**

The filter was operated for 90 days with gas humidification and it was observed that $\eta_R$ of 99.5% and exit volume fraction of $\varphi = 10 \cdot 10^{-6}$ could be obtained at $\varphi = 2020 \cdot 10^{-6}$ of inlet H₂S fraction under stable conditions. Studies were conducted with and without humidification and observed that humidification of the pollutant gas was necessary to maintain the moisture in the optimum range of $u = 55-65\%$. The studies also revealed, that inlet H₂S fraction was having pronounced effect on the filter performance and beyond $\varphi = 2020 \cdot 10^{-6}$ of inlet H₂S fraction an exit H₂S fraction of $140 \cdot 10^{-6}$ was obtained, which was not in the limits of standards prescribed by Indian regulatory agencies. Filter exhibited stable performance in terms of $\eta_R$ even in acidic phase without intermittent washing. The study showed that agriculture residue mixed with sulfur oxidizing bacteria could be one of the possible alternatives of bed material in biofilter.

**ACKNOWLEDGEMENTS**

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**Nomenclature**

- $Q$ – gas flow rate, m³ h⁻¹
- $t_{EBRT}$ – empty Bed Residence Time, s
- $\eta_R$ – removal efficiency, %
- $q_G$ – mass fraction granulometric analysis, %
- $n$ – shaker speed, min⁻¹
- $\rho_i$ – inlet volume fraction, $10^{-6}$
- $\rho_o$ – outlet volume fraction, $10^{-6}$
- $\rho$ – elimination copacity, g m⁻³ h⁻¹
- $v$ – surface loading rate, m h⁻¹
- $\Gamma$ – mass loading rate, g m⁻³ h⁻¹
- $u$ – moisture, %
- $V$ – volume of the biofilter, m³
- $w$ – mass fraction, %

**References**