

## Article

# Thermal Regeneration of Activated Carbon Used as an Adsorbent for Hydrogen Sulfide (H<sub>2</sub>S)

Brendan Bunker<sup>1</sup>, Bruce Dvorak<sup>1</sup>  and Ashraf Aly Hassan<sup>2,\*</sup> <sup>1</sup> Department of Civil and Environmental Engineering, University of Nebraska-Lincoln, Lincoln, NE 68583, USA<sup>2</sup> Civil and Environmental Engineering Department and National Water and Energy Center, United Arab Emirates University, Al-Ain P.O. Box 15551, Abu Dhabi, United Arab Emirates

\* Correspondence: alyhassan@uaeu.ac.ae; Tel.: +971-03-7135334

**Abstract:** The treatment of gaseous contaminants, such as hydrogen sulfide (H<sub>2</sub>S), is often carried out with adsorbent materials that are disposed of after saturation. The reuse of such materials promotes sustainability and the reduction in unnecessary waste. Granular activated carbon (GAC) is a well-known adsorbent used to capture gaseous H<sub>2</sub>S which can be reused. It is hypothesized that it can also concentrate contaminants for future treatment, thereby reducing secondary treatment costs. Cyclic adsorption/desorption experiments were completed with samples of GAC to investigate the feasibility of implementing the concept of repeated H<sub>2</sub>S adsorption/desorption in the construction of a pilot odor control device. A column filled with GAC was exposed to a stream of H<sub>2</sub>S gas and then heated to 500 °C to regenerate the carbon. The concentration of H<sub>2</sub>S at the inlet and outlet of the column was measured at regular intervals. Three samples of GAC had an average adsorption efficiency of 82% over the course of three cycles and were regenerated to 70% of initial adsorptive capacity after one cycle, and 60% after two cycles. These results indicate that after being saturated with H<sub>2</sub>S, GAC can be regenerated at high temperatures, evidence that H<sub>2</sub>S may become concentrated during the process. Additional characterization experiments confirmed that the sulfur content of the carbon increased after adsorption and decreased after thermal regeneration. The procedures demonstrated in this experiment were further utilized with a pilot device designed to provide a low-cost method for reducing odors in landfill gas.



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**Keywords:** adsorption; thermal regeneration; granular activated carbon (GAC); hydrogen sulfide (H<sub>2</sub>S)

## 1. Introduction

Granular activated carbon (GAC) is a widely used adsorbent for the removal of hydrogen sulfide (H<sub>2</sub>S). Its high porosity and extensive surface area make it an extremely effective adsorbent [1]. It is one of the best alternatives based on cost and simplicity [2]. GAC is produced from a variety of carbon sources including coal [3], wood [4], peanut shell [5], and coconut shell [6], each with varying pore structures and carbon content. GAC is a particularly attractive alternative because of its ability to remove H<sub>2</sub>S at low concentrations such as those at wastewater treatment plants, concentrated feeding animal operations, or sanitary landfill sites [7].

The process of adsorption can be influenced by the physicochemical and structural characteristics of GACs. For example, GAC can be impregnated with cations distributed on the pores of the carbon surface [8]. Sodium hydroxide (NaOH), sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), potassium hydroxide (KOH), potassium iodide (KI), potassium carbonate (K<sub>2</sub>CO<sub>3</sub>), copper (II) nitrate (Cu(NO<sub>3</sub>)<sub>2</sub>), and iron (III) chloride (FeCl<sub>3</sub>) are examples of compounds used in impregnated GACs (IACs) [9–11]. The presence of cations in IACs increases the effects of chemisorption but reduces the effect of physisorption because of the increased number of occupied adsorption sites on the carbon surface [12,13]. Adsorption of H<sub>2</sub>S can also be influenced by other surface properties of the carbon including pH, pore size distribution, moisture content, and specific surface area [14,15].

Exhausted GAC can be regenerated as an alternative to discarding or incineration. Regeneration helps protect the environment and conserve resources by reducing the creation of waste materials [16]. Many methods for regeneration are being investigated, including microwave regeneration, solvent extraction, extraction with supercritical fluids, electrochemical regeneration, and catalytic oxidation [17]. Furthermore, the most well-developed method for the regeneration of H<sub>2</sub>S-saturated GAC is conventional thermal regeneration. This process has been performed with streams of air and water [18]. Regeneration of H<sub>2</sub>S-saturated carbon using water is marginally successful because of the presence of bulky sulfur polymers that are formed on the carbon surface during adsorption [19]. Large sulfur-containing compounds such as sulfuric acid are the result of chemisorption that creates a low-pH environment that is difficult to regenerate with water. However, another study successfully removed phenols, textile dyes, and pesticides using liquid water at subcritical conditions [20]. Water heated to a temperature of 300 degrees Celsius (°C) and pressurized to 120 atmospheres (atm) fully regenerated samples of exhausted carbon. After five repetitions of adsorption followed by regeneration, or adsorption–regeneration (A-R) cycles, the adsorption of capacity was fully maintained. Thus, higher temperatures and pressures seem to be necessary for successful liquid water desorption of H<sub>2</sub>S [21].

The use of inert gases for H<sub>2</sub>S desorption has also been investigated. Passing heated inert gases over exhausted carbon resulted in significant removal of sulfur species from the carbon surface [22]. However, the use of nitrogen gas was determined to be much less efficient than the use of air. The amount of sulfur removed from exhausted samples of carbon was six times greater in samples that were treated with air as opposed to nitrogen. It was postulated that the oxidation of elemental sulfur as well as the reduction in sulfuric acid occurred more readily in an oxygenated air stream. Similar A-R experiments have been completed with phenol-based compounds using microwave heating technologies [23]. Experiments conducted at 1123-Kelvin (K) showed that thermal regeneration was favorable under a CO<sub>2</sub> atmosphere compared to an inert, nitrogen-rich atmosphere. This further confirms that an oxidizing atmosphere is essential for effective regeneration [24].

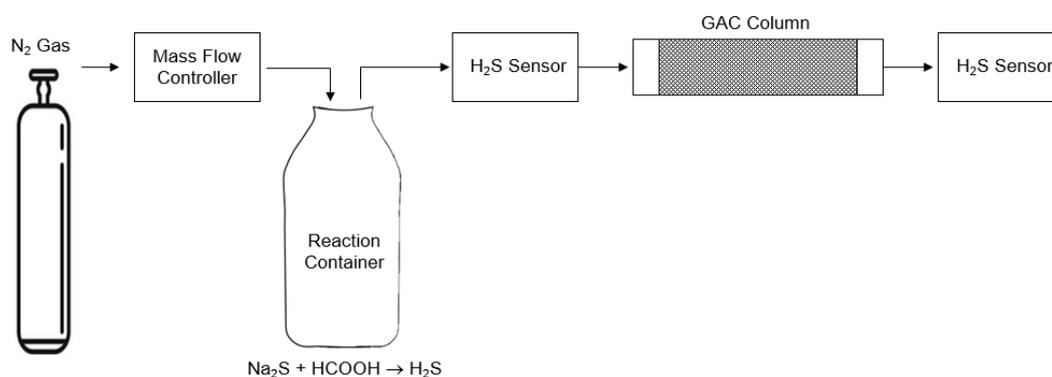
In this work, an explanation of a series of performed A-R experiments is provided. The data obtained from these experiments is presented, and the adsorptive and regenerative capabilities of a GAC developed commercially for the removal of H<sub>2</sub>S from air streams are considered. It is hypothesized that this GAC can be sustainably regenerated with some expected capacity loss.

## 2. Materials and Methods

Samples of a commercial granular GAC, GC Sulforsorb Plus (General Carbon, Paterson, NJ, USA) were obtained for this study. This GAC was non-impregnated, bituminous coal-based, and developed commercially for the removal of H<sub>2</sub>S from air streams. It has an approximate particle diameter of 3.4 millimeters (mm).

The carbon was packed, as received, into a stainless steel column (1 inch inner diameter, 12 inch length). A layer of glass wool and pea gravel was used to secure the carbon inside the column during experimentation.

H<sub>2</sub>S was generated by purging nitrogen (N<sub>2</sub>) gas through a container containing sodium sulfide with acetic acid. The flow rate of N<sub>2</sub> gas entering the column was maintained at 2.5 liters per minute (L/min). The concentration of H<sub>2</sub>S was measured using H<sub>2</sub>S gas loggers at the inlet and outlet. The gas loggers, obtained from Acrulog™ (Clontarf, Australia), were standardized to ±2 parts per million (ppm) using a gas cylinder. When active, the sensors recorded data every 10 seconds. The experimental setup is depicted in Figure 1.



**Figure 1.** Experimental Setup.

### 2.1. Adsorption and Regeneration Experiments

A series of three consecutive A-R experiments were conducted on three separate samples of the commercially obtained GAC, a total of nine A-R experiments. Each A-R experiment, or cycle, consisted of the GAC undergoing H<sub>2</sub>S adsorption followed by thermal regeneration. During adsorption, the GAC was subjected to a stream of gas with a constant flow rate containing a measured, but constantly changing concentration of H<sub>2</sub>S. A variable concentration of H<sub>2</sub>S was used to simulate a typical range of landfill gas emissions [25]. Data were recorded continuously at 10 s intervals with the use of H<sub>2</sub>S sensors placed at the inlet and outlet of each column. The test ended when the influent concentration reached zero following the beginning of the H<sub>2</sub>S-producing reaction. Each adsorption experiment was carried out at room temperature (approximately 25 °C) [26]. The adsorption capacity and efficiency were calculated using the weight of the carbon, flow rate, and the H<sub>2</sub>S concentration in the inlet gas and outlet gas. The adsorption efficiency is the ratio of the mass of H<sub>2</sub>S that entered the column during each test to the mass of H<sub>2</sub>S that exited during the same test. During thermal regeneration, the column containing the GAC sample from the adsorption experiment was heated using heat tape to the designated temperature of 500 °C. During heating, the ends of the column were sealed to prevent any H<sub>2</sub>S from escaping the column volume. Upon reaching the temperature set point, a stream of nitrogen gas was passed through the saturated column at a flow rate of 2.5 L/min. Concentration measurements were obtained from the outlet of the column with the previously mentioned gas data loggers until the concentration of H<sub>2</sub>S leaving the column was negligible. The regeneration efficiency for each sample of carbon was calculated for each successive adsorption run following regeneration. It was obtained by computing the ratio of the mass of H<sub>2</sub>S that was captured by the GAC during adsorption in the first cycle and the mass of H<sub>2</sub>S that was captured during subsequent cycles. The procedures of adsorption and thermal regeneration were repeated three times for each sample.

### 2.2. Material Characterization

Various physical characteristics of the GAC were explored to better understand the results of the experiments. Several tests were conducted to quantify these characteristics, which include elemental composition, specific surface area, micropore volume, and pore size distribution. Three samples were investigated: a fresh (virgin) carbon sample (Carbon I), a carbon sample that had reached breakthrough (Carbon II), and a carbon sample that had been thermally regenerated (Carbon III). The surface morphology and the elemental content of these samples were measured with a scanning electron microscope (SEM) and energy dispersive X-ray analysis (EDX). SEM and EDX measurements were performed using an FEI Nova NanoSEM 450 (University of Nebraska–Lincoln). The carbon was also characterized with nitrogen sorption isotherms at a temperature of 77 K using a computer-controlled Micromeritics ASAP 2460 apparatus (Micromeritics Instrument Co., Norcross,

GA, USA). These isotherms were used to determine the specific surface area by applying the Brunauer–Emmett–Teller (BET) method. The calculation of the mesopore volume (pores 2–50 nm) was accomplished with the Barrett–Joyner–Halenda (BJH) method using nitrogen adsorption data, and the calculation of the micropore volume (pores < 2 nm) was accomplished using the t-plot method.

### 3. Results and Discussion

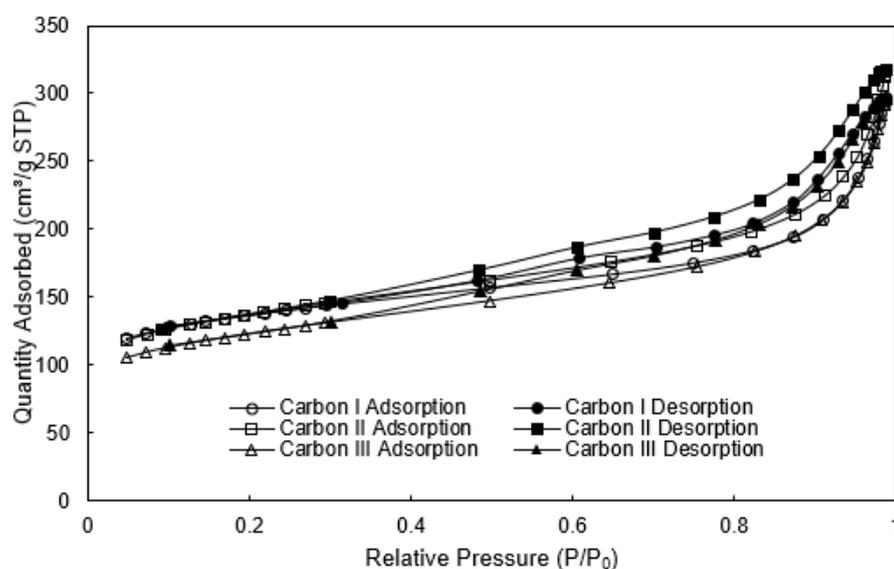
#### 3.1. GAC Characterization

The specific surface area of the samples is a primary indicator of their ability to adsorb contaminants. A summary of the characteristics of the samples is presented in Table 1. The BET surface area of the carbon samples ranged from 372 square meters per gram ( $\text{m}^2/\text{g}$ ) to 433  $\text{m}^2/\text{g}$ . This is lower than typical values for granular GACs, but not unlike some carbons developed from coconut shell, which can have BET surface areas around 380  $\text{m}^2/\text{g}$  [27]. Each value was calculated from its respective nitrogen sorption isotherm. Carbon I demonstrated the highest value of specific surface area. This indicates that it may have the highest physical adsorptive capacity of the four samples. In contrast, Carbon II showed the lowest surface area and total pore volume, both of which sharply declined compared to Carbon I. These are both expected effects of  $\text{H}_2\text{S}$  adsorption. The surface area and average pore diameter of Carbon III are higher than those of Carbon II, an indication that thermal regeneration may have produced vacancies on the surface of the GAC.

**Table 1.** BET Surface Area, pore volume, and pore diameter data for Carbons I–III.

Sample	BET Surface Area ( $\text{m}^2/\text{g}$ )	Micropore Volume ( $\text{cm}^3/\text{g}$ )	Mesopore Volume ( $\text{cm}^3/\text{g}$ )	Total Pore Volume ( $\text{cm}^3/\text{g}$ )	Average Pore Diameter (nm)
Carbon I	433	0.13	0.18	0.31	3.34
Carbon II	372	0.10	0.16	0.27	3.34
Carbon III	397	0.10	0.22	0.34	3.62

The isotherms of Carbon I–III are shown in Figure 2. All the carbon samples displayed International Union of Pure and Applied Chemistry (IUPAC) type-I isotherms. Each of the carbons demonstrates a significant increase in nitrogen adsorption at higher relative pressures, evident by the “ski-jump” shape in each isotherm. The hysteresis loops in each isotherm are well-pronounced, which suggests that each of the carbon samples have a well-developed microporosity and mesoporosity.



**Figure 2.** Nitrogen adsorption/desorption isotherms to accompany the data in Table 1.

The three samples of GAC were analyzed using EDX and the results of the elemental analysis appear in Table 2. According to the analysis, the sulfur content of the samples increased after each adsorption cycle, as expected. The weight fraction of sulfur doubled during the first run but increased by only approximately 30% during the second cycle. The amount of sulfur also notably decreased after thermal regeneration from 2.6% to 2.2%. This is an indication that some of the adsorbed sulfur was effectively removed through thermal regeneration, but not all of it. This finding agrees with the results obtained from the cyclic experiments. Furthermore, it is known that thermal regeneration with the use of nitrogen does not remove sulfur-containing compounds from GAC as well as oxygenated air [22]. This is because elemental sulfur cannot be oxidized in the presence of nitrogen gas.

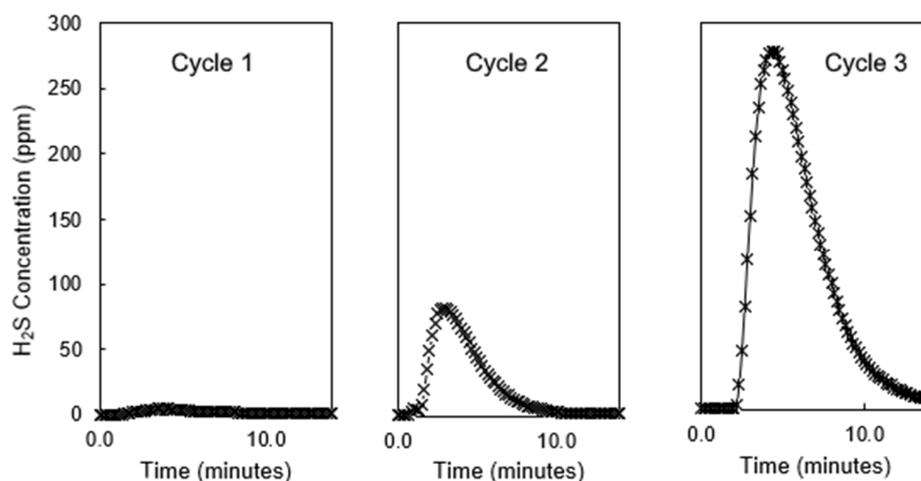
**Table 2.** Results from EDX analysis. Values shown are %wt.

Element	Carbon I	Carbon II	Carbon III
C	65.1	73.3	64.5
O	11.4	10.1	14.4
Na	0.4	0.0	0.0
Mg	1.3	0.7	1.5
Al	2.0	1.1	1.4
Si	2.3	1.7	1.6
S	1.3	2.6	2.2
Ca	12.9	8.1	10.2
Fe	3.5	2.3	4.3

The amount of oxygen fluctuates for each sample but stays relatively constant. During the adsorption experiments described previously, a nitrogen stream was used to transport H<sub>2</sub>S to the carbon. It may be possible that some of this nitrogen stream became adsorbed to the surface of the carbon and this mass of nitrogen replaced the weight of oxygen, thus resulting in a loss of oxygen during each period of adsorption. In many instances, unless there is an unusually high amount of nitrogen on the surface of the carbon, the carbon peak conceals the nitrogen peak. Therefore, nitrogen does not appear in Table 2.

### 3.2. Adsorption

In many of the experiments, the GAC samples experienced breakthrough shortly after the beginning of the H<sub>2</sub>S-producing reaction. The measured concentration of H<sub>2</sub>S during experienced by the GAC during adsorption over the course of the experiments peaked at 200 ppm and varied as the H<sub>2</sub>S-producing reactions progressed. A plot of consecutive breakthrough curves for one of the GAC samples is shown below in Figure 3. The sample was thermally regenerated after each period of adsorption, as previously described.



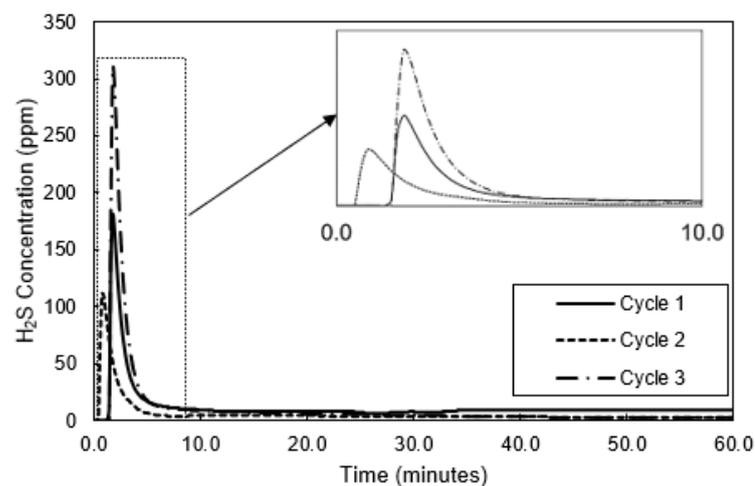
**Figure 3.** Data obtained from the outlet of the lab column during adsorption for three subsequent A-R cycles experienced by GAC Sample #3 (as designated in Table 1).

The first cycle showed an excellent capture of  $\text{H}_2\text{S}$ , with minimal breakthrough. The amount of  $\text{H}_2\text{S}$  captured by the GAC decreased in subsequent cycles, indicating that the capacity of the GAC to adsorb  $\text{H}_2\text{S}$  decreased after the first regeneration. For the sake of simplicity and to reduce the effect of experimental error, the adsorption capacity of the GAC used in these experiments was estimated based on its performance in a virgin cycle where breakthrough was negligible. The amount of  $\text{H}_2\text{S}$  adsorbed in the first cycle shown below is approximately 2.0 milligrams (mg). This value, which corresponds to an adsorption capacity of 0.4 milligrams per gram (mg/g), was used as a baseline to later calculate the efficacy of thermal regeneration. This adsorption capacity closely resembles values obtained in other studies involving  $\text{H}_2\text{S}$  adsorption by GACs. One study reported an  $\text{H}_2\text{S}$  adsorption capacity of 0.5 mg/g [28].

The average adsorption efficiency was approximately 82%, with an average effectiveness of 94%, 74%, and 79% for the first, second, and third cycles, respectively. These data suggests that the carbon used in these experiments performs well in its virgin state but loses effectiveness after its first regeneration. These findings agree with previous studies that reported regeneration of the adsorptive capacity of GAC to at least 30% of its initial value [22] after treating gaseous  $\text{H}_2\text{S}$ .

### 3.3. Thermal Regeneration

After each sample of GAC was subjected to a stream of  $\text{H}_2\text{S}$ , the carbon was regenerated by heating to a temperature of 500 °C. Figure 4 shows the concentration of  $\text{H}_2\text{S}$  during successive regenerations for the same sample as in Figure 2, Sample #3. The height of each peak in Figure 4 varies based on the magnitude of  $\text{H}_2\text{S}$  that was introduced to the carbon during adsorption. The theoretical amount of  $\text{H}_2\text{S}$  that entered the column, based on experimental data, was 2.0 mg, 2.2 mg, and 3.5 mg for the first, second, and third cycles, respectively. It is believed that Cycle 3 produced the largest concentration of  $\text{H}_2\text{S}$  during thermal regeneration both because the carbon had been saturated three times and because the input during adsorption was the largest of the three cycles. The total amount of  $\text{H}_2\text{S}$  desorbed during the previously described cycles was 1.5 mg, 0.8 mg, and 1.3 mg, respectively.



**Figure 4.** Desorption data from subsequent cycles for one sample of GAC.

Following one A-R cycle, the adsorptive capacity was reduced to approximately 70% of its initial value, on average. After two cycles, the capacity dropped to 60%, noting that the third sample produced dissimilar results to the other two samples. The reason for this variance could be the presence of residual  $\text{H}_2\text{S}$  that was not removed during thermal regeneration. It was also determined that the commercially obtained GAC produced gases that were detected as  $\text{H}_2\text{S}$ . It is well known that cross-sensitivities exist for  $\text{H}_2\text{S}$  sensors at elevated temperatures [29]. Therefore, the results for Samples #1 and #2 may be artificially

high. The adsorptive capacity for each sample following one and two A-R cycles is shown below in Table 3.

**Table 3.** The fraction of the initial adsorptive capacity that remained for each sample after one and two A-R cycles. The initial adsorptive capacity for each sample was approximately 0.4 mg/g, as previously noted.

Sample	1 A-R Cycle	2 A-R Cycles
Sample 1	85%	75%
Sample 2	82%	45%
Sample 3	43%	61%
Average ( $\pm\sigma$ )	70 $\pm$ 23%	60 $\pm$ 15%

#### 4. Conclusions

The primary objective of the experiments described in this work was to investigate the concept of thermal regeneration of a non-impregnated, bituminous coal-based GAC that was developed commercially for the removal of H<sub>2</sub>S from air streams. Though the regeneration efficiencies are not as high as other studies have reported, successful regeneration of saturated carbon was observed.

Results from the experiments indicated that after being saturated with H<sub>2</sub>S, GAC can be effectively regenerated at high temperatures. The samples used in these experiments were regenerated at an efficiency of approximately 70% after one adsorption cycle with lower efficiencies in later cycles. A temperature of 500 °C is suggested to remove the sulfur-containing compounds from the GAC.

#### 5. Implications and Future Work

The results of these experiments provided further evidence to the already existing body of proof that thermal regeneration can be a successful approach to reduce treatment costs for processes that utilize frequently replaced adsorbents. This approach may also allow for the concentration of H<sub>2</sub>S as a method for reducing the cost of subsequent treatment. Further applications were investigated in a pilot study that utilized a packed GAC column for adsorption of H<sub>2</sub>S at much lower concentrations than used in this study, which was regenerated to provide a concentrated stream of H<sub>2</sub>S [30]. This procedure could hypothetically reduce the cost of a subsequent biological process by reducing the air volume treated.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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