ADSORPTION OF HYDROGEN SULFIDE BY ACTIVATED CARBON PRODUCED BY REGENERATION OF SLUDGE FROM UPFLOW ANAEROBIC SLUDGE BLANKET

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Abstract. The objective of this study was to determine the efficiency of H_2S adsorption by activated carbon obtained from the regeneration of sludge from an Upflow Anaerobic Sludge Blanket (UASB). The H_2S adsorption system was carried out in the Chemistry laboratory of the Universidad Peruana Unión, the inflow of the fixed bed towers was 4 $L \cdot min^{-1}$ and 36 g of adsorbent media (6 different Active Carbon (AC), depending on Pyrolization temperatures (PT) and [M] KOH), AC at PT 700 ºC/1M, AC PT 500 ºC/4M, AC PT 500 ºC/1M, AC PT 700 ºC/4Mand AC PT 600 ºC/2.5 M the last with two repetitions. The adsorption of H_2S was determined by the volumetric oxidation-reduction method. The parameter of the greatest influence for the adsorption of H_2S was PT at 99 and 90% CI respectively, demonstrated by 22 and 32 factorial designs; satisfactory elimination efficiencies of H_2S were obtained and reached 94% during a retention time of 20 minutes of activated carbon at PT 700 ºC and [1M] KOH. For the response surface prediction simulation, the optimized parameters for the removal of H_2S approximately greater than 96% was at PT of 635 ºC and activating agent of [2.6 M] KOH. Therefore, the adsorption of H_2S by activated carbons for sludge is a new, viable and sustainable alternative for the environment.

Keywords: sludge, adsorption, hydrogen sulphide and carbon obtained.

Introduction

Air pollution has been of increasing concern to society, as it directly affects people's quality of life [1]. H2S is typically the most toxic and corrosive pollutant from industrial processes, and this leads to sulfur dioxide emissions, which have potentially harmful environmental effects [2]. In recent years, H2S has been found in high levels in sewage systems with links to animal slaughtering, industrial sources such as petroleum, gas processing, geothermal power plant and pulp mills, among others [3].

The sources of hydrogen sulfide emissions are the upflow reactors of the UASB type, which are currently used for the treatment of effluents from large urban centers and industries, as they are a low energy consumption alternative [4] states that the treatment of wastewater with UASB generates gases such as methane, volatile organic sulfur compounds such as $H2S$, a gas that is toxic to human health.

The elimination and removal of H2S (main odorous compound) is of vital importance for the benefit of all personnel operating within the industrial sectors that generate H2S and surrounding populations, contributing to the protection of individual and collective health of the population surrounding places, where H2S is generated in harmony with the full exercise of the fundamental right to live in a balanced and adequate environment for the development of life [5].

The multiple methodologies for the removal of $H2S$, are included in biological and physical-chemical processes. A study carried out by [6] also indicates the absorption and adsorption media used (activated carbon, iron oxides) and technologies for biological processes (biofilter, soil bed filter, biological scrubber). Chemical absorption benefits the efficiency, reaction rate and elimination operation of H2S compared to physical. It involves the formation of reversible chemical bonds between the solute and the solvent. Instead, adsorption is the mechanism by which a fluid adheres to the surface of a solid, it is a process resulting from the interaction of the Van der Waals force [7].

All of this is not new, and for decades various alternatives for the elimination of H2S have been studied [8 ; 9]. Adsorption using sodium hydroxide (NaOH) together with activated carbon was studied, and in 1982 they sought to remove H2S gas with hydrogen peroxide and nickel or iron compounds as a catalyst. [6;10] performed desulfurization by adsorption using thermally treated sewage sludge.

This study aims to evaluate the adsorption efficiency of H2S using activated carbons from sludge generated in a UASB reactor, also focused on the study of the significance of pyrolyzation temperature parameters, and molar concentration of the activating agent in obtaining the optimum adsorbent medium for H2S adsorption by applying a 22 statistical factorial design and prediction by a 32 factorial design.

A significant factor in this study is the use of residue as a precursor material (dry sludge from a UASB) for the activated carbons, as well as the retention time used for the adsorption of H2S. [11] In the few studies found in the literature on the use of sludge as an adsorbent medium, usually they were

treated in prolonged times longer than 1 hour, but in this study, it was 20 minutes. This time reduction is significant because, if the process is implemented on a commercial scale, it will reduce the operating costs, it will also provide viability and sustainability for the environment.

Materials and methods

Raw Material

The sludge used as a precursor material for the preparation of the adsorbent media (active carbons), was acquired from the Wastewater Treatment Research Center (Centro de Investigación de Tratamiento de Aguas Residuales, CITRAR-FIA-UNI) originated in the UASB reactor, which received a dehydration treatment by percolation, located on the campus of the National University of Engineering in Lima, Peru.

Hydrogen sulfide absorption system

The adsorption system was constituted of three parts: gas generation area (for H_2S), storage area and dilution of H_2S and an adsorption towers system for the adsorption tests of H_2S. The H_2S gas was generated chemically from iron sulfate and hydrochloric acid at 7% a total of 105.26 g of FeS and 1104.94 mL of HCl. The dimensions of the fixed bed towers were determined at a height of 20 cm, thickness of 3 mm, diameter of the holes of the cover of the fixed bed towers of 0.5 cm, free edge of 0.50 m and the volume of 1.5 liters.

The activated carbons prepared were a total of 6 at different conditions of pyrolysis temperature (*PT*) and molar concentration of potassium hydroxide ([M] KOH) shown in Table 1, 36 g of each were used for the removal of H_2S with a constant flow of hydrogen sulphide of 4 $L \cdot min^{-1}$ regulated by a flowmeter.

Preparation of activated carbon

The pretreatment procedure of the residual sludge used as an adsorbent of H_2S was carried out as follows: dehydrated sludge was collected from the drying bed of the UASB reactor of the CITRAR FIA-UNI WWTP, then it was milled and sieved with a sieve of 1 cm^2 to obtain 150 g of the sample for each type of activated carbon to be prepared. After having a homogeneous size of sludge, the activation was performed using 2 mL per gram of KOH at different molar concentrations as shown in Table 1, the mixture was left to stand for 1 hour and then filtered to remove the residual reagent from the mixture precursor and activating reagent, then it was taken to an oven at 110 ºC for a period of 12 hours. The dried sludge was pyrolyzed at the temperatures indicated in Table 2 of 500 ºC, 600 ºC and 700 ºC for 1 hour. After the thermal treatment, a washing process was carried out using 100 mL of 5% HCl solution, then it was taken to a stirrer for 4 hours, then it was filtered to eliminate the residual reagent, the washing was carried out with distilled water until obtaining approximately neutral pH of 4-5, finally the samples were subjected to the drying process at 105 ºC for 2 hours, and the active carbons were weighed. The first washing was carried out to eliminate the residual mineral matter and the products derived from the activation, while in the second one the possible remains of acids were eliminated.

Results and discussion

Active carbons

Hydrogen sulphide can be physically and chemically adsorbed due to the porous structure, the presence of metals and the alkalinity of the surface of the processed adsorbent. An important characteristic of sludge-derived materials is their basicity, mainly due to the addition of calcium or magnesium during the sludge treatment process, and the presence of components in the form of oxides.

Cotoruelo, Marqués, Díaz, Rodríguez-mirasol and Rodríguez [12] state that the activation time does not greatly influence the adsorption capacity, therefore the 6 different activated carbons prepared were subjected for 1 hour activation time, but other factors were changed in each sample, as shown in Table 1. It should be noted that the pyrolysis process is performed in the absence of oxygen to avoid combustion, in this case dry sludge and the addition of KOH are used, which allows the development of internal porosity, substances and deposited carbonaceous residues are removed, opening current pores and developing new ones.

Table 1

Characteristics of active carbon parameters

A.C. Number	*TS*, ºC	*TS*, h	KOH [M]	*TC* AA, h	*PT*, ºC	pH
PT 500 ºC/ [1M] KOH	110	12	1 M	1.0	500	4.5
PT 500 ºC/ [4M] KOH	110	12	4 M	1.0	500	4.2
PT 700 ºC/ [1M] KOH	110	12	1 M	1.0	700	5.0
PT 700 ºC/ [4 M] KOH	110	12	4 M	1.0	700	4.0
PT 600 ºC/ [2.5M] KOH	110	12	2.5 M	1.0	600	4.8
PT 600 ºC/ [2.5M] KOH	110	12	2.5 M	1.0	600	4.7

The pyrolyzed carbons were washed with 5% HCl at room temperature in a magnetic stirrer; all the activated carbons produced obtained a pH of 4-5 (Table 1). [13] Basic pH favors the formation of elemental and polymeric sulfur as final oxidation products; there is a pH threshold below which this process becomes unfeasible. However, it probably does not apply to our sludge-derived materials, since even at acidic pH, the adsorption capacity is still significant, due to the porosity of the activated carbons allowing the adhesion of H_2S on their surfaces.

Efficiency of H_2S absorption

Table 2 shows the results of adsorption efficiency by applying the 2^2 factorial design of experiments with central points. It is observed that the highest adsorption values (%) of H_2S are in the central treatments.

Carbon is a solid material which is an excellent adsorbent, as well as being versatile and easy to handle, these properties give it the ability to absorb a multitude of different molecules on its surface, removing them from the medium with which it is in contact, a characteristic demonstrated by the results shown in Figure 4. A lower efficiency of H_2S absorption is observed with the activated carbon obtained at *PT* of 500 °C and [1 M] of KOH, reaching the efficiency of 66.2% of H_2S removal. The average concentration of hydrogen sulfide in the effluent was 43.15 ppm, a potentially harmful level, which can cause headaches and irritation of the respiratory tract, therefore it is concluded that the efficiency of this adsorption medium is deficient. Research conducted by [14] found that adsorbents prepared at low temperatures have lower adsorption capacity, probably due to insufficient development in the formation of the porous structure, which is indeed corroborated in the results obtained.

An adsorption medium prepared at *PT* and activation of 500 °C and [4 M] KOH has an efficiency in an average range of 70-80%, 27.53 ppm is the average concentration of H_2S after treatment with this type of adsorbent medium. Taking into account the same *PT* of 500 ºC, higher adsorption efficiency is found for the activated carbon with higher concentration [4 M] KOH. [15] state that when the activating reagent is applied to it and cleans the precursor material, this influences the quality of the adsorbent obtained, which is likely the explanation for the results found in this case.

The activated carbon at *PT* 600 ºC and [2.5 M] KOH has a similar efficiency to the activated carbon at *PT* 700 °C and [1M] KOH, however, the results show some tests in which the results are less than 90% (see Table 2), whereas the activated carbon at *PT* 700 ºC and [1M] KOH remains constant, therefore, the one with the highest efficiency is the latter. [14] state that at an activation temperature higher than 700 °C the formation of the pores of the activated carbon will be greater, which implies a greater adhesion of H_2S in the activated carbon obtained, which is in line with our findings in this study. The addition of the alkali metal KOH was meant to provide a catalytic surface to the adsorbent medium, to achieve the oxidation of H_2S leading to its more complete adsorption.

Table 2

Replicated results of H_2S adsorption efficiency in 2^2 factorial design with central points

Treatment	Parameters		Replicas of H_2S absorption, %		
	***PT*, ºC**	**KOH [M]**			
1	500	1.0	68.75 68.57 68.57 63.16	68.57 67.74 65.83 60.27	61.43 63.09 62.47 65.31 ± 3.28*
2	700	1.0	92.85 94.78 94.78 92.28	95.40 92.28 92.28 96.90	94.95 94.13 93.38 94.01 ± 1.52*
3	500	4.0	84.85 87.50 87.50 87.50	91.43 79.98 72.64 88.18	85.84 79.84 72.01 83.38 ± 6.44*
4	700	4.0	91.51 84.98 84.98 80.56	81.67 88.90 95.08 95.87	94.95 82.68 84.80 87.81 ± 5.71*
5	600	2.5	93.79 88.67 88.67 93.15	94.13 93.15 96.90 97.52	95.40 99.08 96.90 94.30 ± 3.38*
6	600	2.5	90.58 93.16 93.16 94.29	94.80 94.93 93.45 94.43	91.96 94.63 94.60 93.63 ± 1.36*

* Mean and Standard Deviation

In [13] the average pore diameter is evaluated and found that the lowest value obtained was at an activation temperature of 600 ºC, indicating the development of microporosity. In this case the efficiency was not low, since KOH of considerable molar concentrations was applied, increasing the adsorption capacity. They also demonstrated that the activation carried out at 600 ºC is superior to 400 ºC and the influence of time is not significant, remaining practically constant, which is corroborated by the results obtained in our study. The adsorption of H_2S after passing through an activated carbon at the conditions indicated in Table 1 reaches a significant removal of 94%, the average H_2S concentration of the effluent is 9.3 ppm, a value that according to the National Institute for Occupational Safety and Health [16] is within the established level of up to 10 ppm, however, not sufficient for the optimal adsorption of H_2S, since an even higher adsorption of H_2S would contribute to the reduction of the effects caused by this agent because it is highly toxic.

Statistical analysis

Experimental factorial design. In table 3 it can be observed that the model is statistically significant, with a confidence level of 95%. The graphs generated for fit to the first order model reached a coefficient of determination of 87.3%, which implies that the model is a good fit for the experimental data. According to [17] for a factorial design, a maximum of 4 repetitions of each test level are necessary. However, the purpose of this research was not only to test the statistical design, but also to determine the efficiency of the purification of the toxic gas, thus the results from additional tests provide consistency to the statements made in this research, especially considering that the behavior of H_2S is

complex. Therefore, the simulation and prediction by the response surface for a second order model was performed, extrapolating the data and achieving a representativeness of 96%.

Table 3

Analysis of variance for H_2S adsorption

Factor	Sum of squares	Gl	Mean square	*F*-Ratio	*p*-value
Curvatr.	1514.974	1	1514.974	91.7291	0.000000
(1)*PT*, ºC	3015.742	1	3015.742	182.5980	0.000000
(2)KOH [M]	388.773	1	388.773	23.5396	0.000009
1 by 2	1618.385	1	1618.385	97.9904	0.000000
Error	1007.460	61	16.516	-	-
R^2 = 87.3					

La and Gandarillas [18] corroborate that for the adsorption of H_2S by activated carbon, it must be impregnated with a chemical compound, but while the usual method is with sodium hydroxide, in this case it was performed with KOH, which allows for the union of adsorption forces from chemical affinity. Thus, activated carbon provides the surface area and retention is performed by chemical adsorption.

Figure 1 shows the contour plot and response surface, the carbon obtained at higher pyrolysis temperature and a lower molar concentration had a higher efficiency in the removal of the toxic gas (H_2S), with a peak value at *PT* 700ºC and with an activating agent of [1M] KOH corroborating in the research conducted by Carvalho and Castro [19], who state that with the increase of pyrolysis temperature, the adsorption capacity of activated carbons regenerated from sludge increases significantly and thus the efficiency in the removal of the toxic gas H_2S.

a) b)

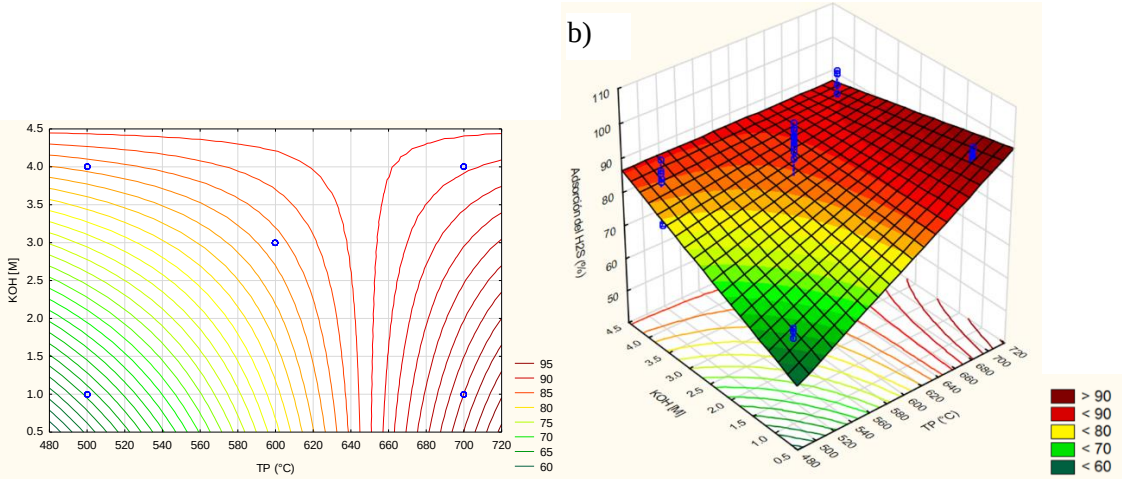

Fig. 1. **Design of factorial experiment of H_2S adsorption by contour plot (a) and response surface (b)**

$$\text{Adsorption of } H_2S\ (\%) = -32.645 + 0.183T + 26.2407C - 0.0404TC, \quad (1)$$

where T – pyrolysis temperature (*PT*) (ºC);
C – molarity concentration [M] of potassium hydroxide KOH.

The first order model corroborates that the T parameter (*PT*) had higher significance in the adsorption of H_2S, with a coefficient of 0.183T and concentration of [M] with the coefficient of 26.27. The contour plot, response surface and the model predict that at medium concentration level [M] could be generated a second order curvature, where the adsorption of H_2S is greater than 90%. In order to predict and optimize the adsorption greater than 96% we simulated by the response surface using the 3^2 experimental factorial design.

Simulation by response surface: Table 4 shows the prediction simulation of the significant variables for the adsorption of H_2S, the temperature (*PT*) in the highest proportion followed also by the molar concentration of the activating agent, which presents a significant correlation with a representation greater than 90.00%.

Table 4

Coded and uncoded variables for H_2S adsorption using the response surface

Treatment	Coded		Uncoded		H_2S Absorption, %
	X_1	X_2	*PT*, ºC	KOH [M]	
1	-1.0	-1.0	500.0	1.0	63.88 ± 2.28
2	-1.0	0.0	500.0	2.5	73.02 ± 1.49
3	-1.0	+ 1.0	500.0	4.0	82.16 ± 0.71
4	0.0	-1.0	600.0	1.0	78.78 ± 0.79
5	0.0	0.0	600.0	2.5	93.73 ± 3.82
6	0.0	+ 1.0	600.0	4.0	84.97 ± 0.48
7	+ 1.0	-1.0	700.0	1.0	93.68 ± 1.01
8	+ 1.0	0.0	700.0	2.5	90.73 ± 1.29
9	+ 1.0	+ 1.0	700.0	4.0	87.79 ± 1.59
10	0.0	0.0	600.0	2.5	93.66 ± 0.85

To evaluate the fit of the model obtained, an analysis of variance was performed, from which it was determined that the model applied was significant ($p < 0.05$). Figure 1 (a, b) shows that the model is statistically significant for all parameters except the interaction of linear *PT* and quadratic KOH. The parameter that has the largest significant effect is linear *PT* and linear KOH, followed by quadratic *PT* and quadratic KOH.

Table 5

Analysis of variance by response surface for H_2S adsorption

Factor	*SS*	*dF*	*MS*	*F*	*p*
(1)Temperature, ºC (L)	1411.620	1	1411.620	113.5870	0.000000
Temperature, ºC (Q)	179.669	1	179.669	14.4572	0.000867
(2)KOH [M] (L)	172.412	1	172.412	13.8733	0.001053
KOH [M] (Q)	179.669	1	179.669	14.4572	0.000867
1L by 2L	437.993	1	437.993	35.2434	0.000004
Error	298.264	24	12.428	-	-
Total SS	2751.495	29	-	-	-
$R^2 = 89.16$					

In Table 5 it can be observed that the model is statistically significant, with a confidence level of 95%. The graphs generated for fit to the second order model reached a coefficient of determination of 89.16%, which implies that the model is a good fit for the predicted experimental data.

Figure 2 shows graphically the simulation for the optimization of the parameters of temperature and [KOH M] that affect the response variable (adsorption of H_2S), where it can be observed that at an interval of [2.25-2.75 M] of KOH and at an interval of 618 to 655 ºC of *PT* the adsorption of H_2S is higher than 96%, which means that the optimization point is at the temperature of 635 ºC of *PT* and at [2.6 M] of KOH the removal of H_2S is expected to be 99.00% approximately.

The data generated to a second order polynomial model is presented in the following mathematical model, where:

$$\text{Adsorption of } H_2S\ (\%) = -224.845 + 0.797T - 0.001T^2 + 37.488C - 2.25C^2 - 0.63TC - 0.04TC, \quad (2)$$

where T – pyrolysis temperature (*PT*) (ºC);
C – molarity concentration [M] of potassium hydroxide KOH.

The second order model corroborates that the T parameter (*PT*) had higher significance in the adsorption of H_2S, with a coefficient of $0.001T^2$ and concentration of [M] with the coefficient of $-2.25C^2$. The contour plot, response surface and the model predict that at medium concentration level [M] could generate a second order curvature where the adsorption of H_2S is greater than 96%.

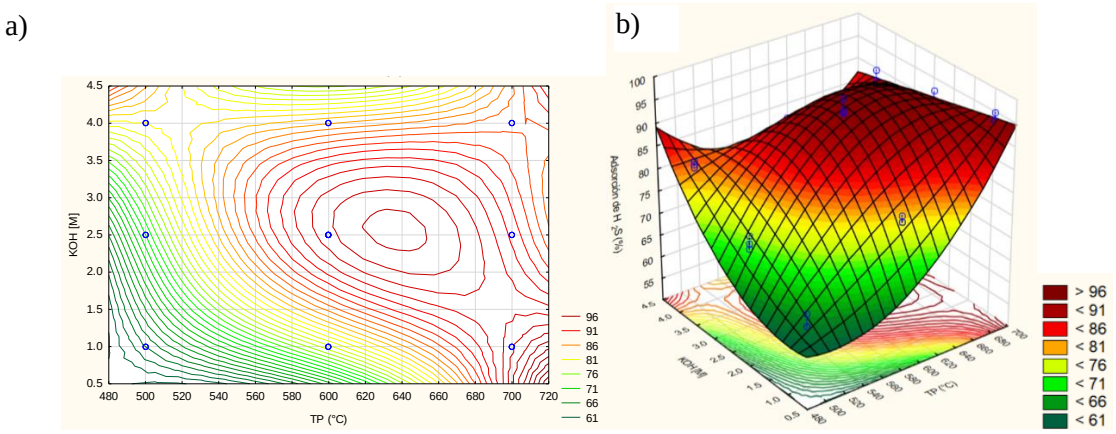

Fig. 2. **Simulation of H_2S adsorption optimization by contour plot (a) and response surface (b)**

Conclusions

The efficiency of hydrogen sulfide adsorption by activated carbon obtained from the regeneration of sludge from an Upflow Anaerobic Sludge Blanket (UASB) was optimal at 94%. The production of adsorbents from sewage sludge generated in a UASB reactor is feasible. According to the factorial design analysis, the factor with the greatest influence on the adsorption capacity of the adsorbent media produced for H_2S absorption was the pyrolysis temperature followed by the molar concentration.

Activated carbon produced at higher *PT* and lower molar concentration of KOH was the most efficient in the 2^2 factorial experiment design at *PT* 700 ºC and the activating agent of [1 M] KOH.

For the response surface prediction simulation, the optimized parameters for the removal of H_2S approximately greater than 96% were at the PT of 635 ºC and the activating agent of [2.6 M] KOH.

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Nomenclature

TC – Contact time
A.A – Activating agent
UASB – Upflow Anaerobic Sludge Blanket
g – Grams
T – Temperature ºC
PT – Pyrolization Temperature
[M] – Molar Concentration
L – Liter
min – Minutes
NaOH – Sodium hydroxide
ml – Milliliters
h – Hour
cm – Centimeters
m – Meters
AC – Active Carbon
TS – Dryng time