Modern Environmental Science and Engineering (ISSN 2333-2581)
July 2016, Volume 2, No. 7, pp. 445-453
Doi: 10.15341/mese(2333-2581)/07.02.2016/002
Academic Star Publishing Company, 2016
www.academicstar.us

Comparison of Two Water Regeneration Strategies for Activated Carbon Loaded with Hydrogen Sulphide Gas

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Abstract: This work compares two different types of water regeneration methods, continuous washing and intermittent washing, that are applied to catalytically enhanced activated carbon previously loaded with hydrogen sulfide gas. The efficiency of the two regeneration methods was evaluated using Thermo Gravimetric Analysis (TGA), Coulorimetric titration using Karl Fisher reagent and apparent density "as is". The effectiveness of both methods in terms of regenerability, apparent density, and sulphur and organic loading was investigated. Results demonstrated that regeneration of the activated carbon by intermittent washing is more effective than by a continuous washing, as the former was able to remove more sulphur and organic loading from the activated carbon material than the latter, hence retaining its adsorption capacity.

Key words: adsorption, regeneration of activated carbon, thermo gravimetric analysis (TGA), Coulorimetric titration, hydrogen sulphide, odour control

1. Introduction

Odor abatement is becoming a critical issue especially in urban districts where the presence of pollutants can affect significantly the ecosystem, and, specifically it can initiate odors into the environment. Sewage treatment plants are the main sources of the odor emissions. Some of the most common compounds found in typical sewage treatment facilities are Hydrogen Sulphide (H_2S), Volatile Organic Compounds (VOC's) and Volatile Organic Sulphur Compounds (VOSC's) [1-4]. These gases should be eliminated as they contain an immense level of toxicity, recurring exposure can cause adverse health effects, they have a foul odour and lastly, they can cause corrosion. Therefore, stringent environment policy forced engineers and scientist to search for solutions to eliminate pollutants from wastewater and air.

With that said, continuous research is required to develop more competent technologies to minimize the effect of harmful pollutants. The main process currently considered to eliminate those pollutants is adsorption [5]. Activated carbon (AC) has been widely and extensively used as an adsorbent to remove malodorous compounds such as H_2S from sewage treatment plants [6-9].

The adsorption itself occurs on the surface area of the activated carbon pores and it can be either a physical or a chemical process [3, 10]. Once the AC is fully saturated with the pollutant, it must be replaced or regenerated depending on the type of carbon used. Cost of replacement of activated carbon is not economically feasible, thus in order to reduce the operational cost, it is advisable to reuse the activated carbon after the reactivation process.

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AC can be regenerated and it can recover at least 80% of its original adsorption capacity [11]. Several reactivation or regeneration technologies have been used to treat the exhausted activated carbon such as thermal [12-14], chemical [15-17], ultrasonic methods, microwave regeneration [18-21] ion-exchanged [22, 23] and photo-oxidation [24, 25].

All the aforementioned types of regeneration have their own pros and cons and hence, in order to minimize the overall cost of regeneration an effective process has to be selected [26]. Hence, water regeneration is one of the methods considered to reactivate the exhausted adsorbent and it may be the best solution for less developed countries [27]. Water is an integral element in the process of odor control. Activated carbon surface is hydrophobic in nature hence, water adsorption in activated carbon demonstrates that oxygen groups reinforced adsorption at low relative pressure (low levels of humidity) [33]. However, it may lead to a decrease of other adsorbed substances at saturated conditions. Zhou (2009) [28] stated that regeneration using water does not only eliminate the sulphuric acid build up in the carbon but also removes sulphur in colloidal form. One of the greatest advantages of water regeneration is that the wastewater from the regeneration can be disposed of in the wastewater treatment plant directly or after pH adjustment in order to comply with the municipality requirements, hence, it is sustainable.

Additionally, amongst the different regeneration methods, the use of water is the most practical one as it offers several advantages: water is an economical universal solvent and an ideal medium to regenerate spent carbon because it does not cause any chemical oxidation, thereby preserving its physical properties; carbon is only washed with water, and as a result, it promotes longer shelf life of the carbon because it retains its adsorption capacity with minimal reduction. Nevertheless, water scarcity is prevalent and requires vital solution especially in the Middle East. To address this issue, optimization of current technologies along with development of new ones is required.

Hence, this research compares two different water strategies, continuous and intermittent spray washing, for regeneration of an activated carbon previously loaded with H_2S in order to determine the most effective water regeneration method that minimizes operational costs. A new experimental analysis that evaluates the AC in terms of sulphur loading, apparent density, regenerability and organic loading has been used.

2. Methods and Materials

2.1 Case Study

A wet well from the coast of the Persian Gulf in the United Arab Emirates, specifically from the Abu Dhabi Gulf Resort (coordinates: 24.415120, 54.528677), became the source of a foul nuisance smell because of the high H_2S emission, and subsequently, an odor control system using activated carbon was put in place. The foul smell problem was resolved, however, the activated carbon reached its saturation capacity and thereby regeneration had to be conducted. Since activated carbon is water regenerable, sufficient amount of water has to be used in order to wash out the elemental sulphur build up. The current water method for regenerating the carbon involves its continuous spray washing, which consumes large amounts of water. In this work, a new water regeneration strategy, intermittent washing, is investigated as a new potential regeneration method that could yield a more efficient as well as a more environmentally friendly operation.

2.2 Materials

A commercial activated carbon, Norit RST, was selected as the adsorbent material for the separation process as it is used in odor control systems. Norit RST is an extruded, steam-activated coal-based carbon, which was not impregnated with any chemicals. It shows a maximum H_2S adsorption capacity of 36 g/100g carbon (Norit Technical Bulletion 0164) and it can tolerate a relative humidity of 40-100%. As a physical adsorbent, it is capable of removing VOC's,

siloxanes, low concentration of mercaptans and ammonia (Norit Technical Bulletin 0164). Furthermore, RST carbon has low density and low pressure drop and water regeneration can restore much of its adsorption capacity. Characteristics of the Norit RST activated carbon are presented in Table 1.

2.3 Process Parameters and Regeneration Strategies

Considering the normal operation of the wet well based on process parameters from Table 2, the activated carbon bed needs to be regenerated after four months of continuous use. Samples were taken from the top and bottom of the carbon bed (Fig. 1). The air gas stream to be treated enters the adsorption bed through the bottom and the air and adsorbent concentrations change with time and bed position, depending on where the mass-transfer is taking place.

Table 1 Overview properties of Norit RST (Norit Technical Bulletion 0164).

Properties of Norit RST	Units	Range
Impregnated		No
Oxygen necessary -Minimum concentration O_2: H_2S	 Molar basis	Yes 4:1
Relative humidity necessary -Working range -Optimum	 % %	Yes 40-100 60-70
H_2S adsorption capacity	g/100 g	36
Adsorbed H_2S water regenerable -Regenerability -Preferable H_2S range for optimal regeneration -Applicable H_2S range for regeneration	 % ppm ppm	Yes 60-80 < 10 10-100
Empty bed contact time: the volume available for the gas flow divided by the air flow rate through the bed.	s	3-6
Superficial velocity -RST 4	 cm/s	 5-40
BTEX: Benzene, Toluene, Ethyl benzene and Xylene at 10 ppm each, 20 °C.	g/100g	22
D5 Siloxane	g/100g	38
Mercaptans	g/100g	2
Apparent density	Kg/m^3	350

Table 2 Summary of data selected for this research

Process Data	
Flow medium	Air containing H_2S from wet well
Flow temperature	Up to 60°C
Flow humidity	Up to 100%
Equipment location	Outdoor
Ambient temperature	Up to 60°C
Ambient relative winter humidity	50%
Ambient relative summer humidity	100%
Performance Data	
Capacity	450 m^3/hr
Inlet H_2S (Average)	50 ppm
Inlet H_2S (Peak)	250 ppm
Air superficial velocity	0.15 m/sec
General Data	
Type of Carbon	Extruded activated carbon
Model of Packing media	RST4
Manufacturer	Norit

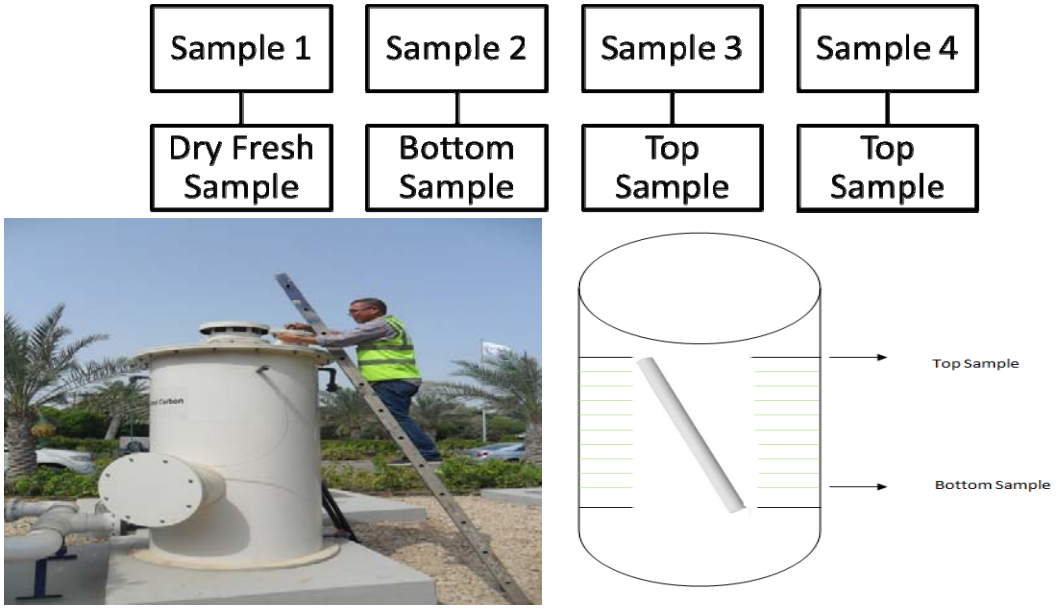

Fig. 1 Samples collected in experimental protocol.

Two regeneration methods were followed in order to restore the AC adsorption capacity; the first one was the conventional method of continuously washing the activated carbon and, the second one was the intermittent washing of the carbon. Four samples were then obtained for analysis (Fig. 1): dry fresh reference sample (Sample 1); bottom sample loaded (Sample 2); top sample after a continuous washing regeneration strategy using a fixed volume of water (Sample 3); and top sample, after intermittent regeneration with the same volume of water as in the continuous regeneration strategy (Sample 4). For samples 3 and 4, their weight was determined and the AC density (360 kg/m^3) was used to calculate the amount of water needed for washing the carbon. In the conventional method, that amount of water is equal to five times the amount of carbon volume "Norit Tech Bulletin 0164 RST". Once the total amount of water needed was calculated, the equivalent to one carbon bed volume of water was sprayed continuously onto the sample and allowed to drain completely. The procedure was then repeated four times and sample (Sample 3) was set aside for measurement. For Sample 4, intermittent washing of the carbon bed was conducted. Same total amount of water was sprayed onto the sample every five minutes, and the sample was allowed to drain completely.

The assumptions made in the experiment were: (1) air flow rate is constant, (2) H_2S concentration from the odor source is not more than 50 ppm during normal operation condition and, (3) temperature and pressure of the carbon bed are constant.

2.4 Analytical Methods

Thermo gravimetric Analysis (TGA) was conducted for all samples in order to determine the effectiveness of the two different washing methods in restoring the AC adsorption capacity. In each TGA test, 60 mg of sample were heated up to 600 °C and temperature was held constant for 30 minutes under a helium (inert) flow rate of 75 ml/min in order to remove water from the samples. Additionally, VOC's are also eliminated by the inert flushing. After that, samples are heated at a heating rate of 10 °C/min up to 1000 °C under a helium flow rate of 75 ml/min and the weight loss of the sample is recorded.

Total sulphur and sulphate content in the samples were measured by combusting the samples in pure oxygen. The combustion products are absorbed in a medium containing hydrogen peroxide. After evaporation, the residual concentrate is analyzed for its sulfate content.

Water content on the samples was determined using the Karl-Fisher titration method. Samples were milled and then titrated coulometrically by a heating method that uses temperatures between 200 and 270 °C. A dried sample of carbon is subjected to volumetric titration and back coulometric titration using Karl fisher reagent. A titer is known by the quantity of electricity used in back coulometric titration.

Apparent density "as is" was also measured for all samples by placing the sample in a 100 ml graded cylinder and determining the carbon weight according to the ASTM method (NSTM 4.2). Activated carbon samples used for gas purification contain VOC's hence; the apparent density is determined "as is". Drying of the sample will remove both water and VOC loading.

3. Results and Discussion

Activated Carbon life is affected by the actual concentration of hydrogen sulphide and other organic compounds in the gas stream. At higher loading of H_2S, elemental sulphur is formed and hence; it will not be washed during regeneration. As a result, carbon will be saturated easily and thereby carbon life is shortened. On the other hand, at lower H_2S loading carbon can maximize its shelf life. A general trend observed for all exhausted carbons is that a more acidic environment promotes the formation of sulfur oxides and sulfuric acid despite rendering small H_2S removal capacities. On the other hand, a basic environment favours the formation of elemental sulfur (sulfur radicals), rendering high capacities.

Sulphur loading is then an essential measurement for the condition of the carbon. It will indicate the amount of sulphur that was not removed during regeneration. As a result, the higher the sulphur loading is, the lesser the adsorption capacity of the carbon is and vice versa. RST carbon needs a minimum temperature of 10 °C to catalytically convert the H_2S into a different compound. When H_2S reaches a maximum concentration of 100 ppm it will become H_2SO_4 provided that sufficient oxygen is available. In this case, H_2SO_4 can be washed out by water. Analysis of data (Table 3) indicates that Sample 4 is the one with the smallest amount of total sulphur, 0.57 mass-% compared to 0.64 mass-% for Sample 3. For Samples 1 and 2, sulphur loading was 0.76 and 0.65 mass-%, respectively, which is not a significant difference. As for measured total sulphate, a value of 0.461 mass-% for Sample 4 is reported versus a value of 0.628 mass-% for Sample 3. Hence, lower values of total sulphur and sulphate loading, which can be observed for Sample 4, are indicative of a better regeneration procedure.

Table 3 Analysis of samples 1 to 4.

Sample no.		Sample 1	Sample 2	Sample 3	Sample 4
Sample point		Dry fresh (Reference)	Bottom sample loaded	Continuous Regeneration	Intermittent Regeneration
Apparent density (as is)	kg/m^3	627.14	760	781.11	839.53
Apparent density dry without loading	kg/m^3	365	361	364	361
Water loading	mass-%	40.2	52.8	51.8	53.1
Loading 25 < T < 500°C	g/100g	2.7	0.6	3.4	9.1
TGA 20 < T < 500 °C	mass-%	41.8	52.5	53.4	57.0
Water loading 25 < T < 500°C	g/100g	69.1	111.2	111.2	123.5
TGA 500 < T < 850 °C	mass-%	2.4	0.9	1.6	1.1
Loading - water 20 < T < 500 °C	mass-%	1.6	0.3	1.6	3.9
Total sulphur	mass-%	0.76	0.65	0.64	0.57
Sulphur loading	g/100g	0.8	0.6	0.7	0.6
Sulphate (SO_4)	mass-%	0.97	0.63	0.62	0.46
Total Organic Volatile Loading (OVL)	g/100g	71.8	110.5	132.6	114.6
Organic loading (OL)	g/100g	2.0	1.3	8.4	2.8
Regenerability	mass-%	41.8	31.6	31.8	26.3

Similarly, the total and organic loading values are as important as the sulphur loading. The higher the organic loading is, the sooner the carbon has to be replaced because the pores of the carbon are blocked by the organic loading. Moreover, higher organic loading has a negative effect on the adsorption capacity which point out that the carbon is oxidized. As it can be seen in Table 3, the organic loading of Sample 4 (2.8 g/100g) is significantly lower than that of Sample 3 (8.4 g/100g) which clearly demonstrates that intermittent washing can eliminate the organics more effectively than the continuous washing. Usually a 10 g/100g limit is kept, above which a negative effect on the adsorption capacity is likely to occur.

The apparent density gives information about the degree of oxidation of the carbon. When comparing Sample 4 versus Sample 3, the values of the apparent densities are 781.11 kg/m^3 and 839.53 kg/m^3 respectively, while the recalculated values of apparent densities for both samples are 364 kg/m^3 and 361 kg/m^3, respectively. A recalculated apparent density which is much lower than the original apparent density often indicates oxidation of the carbon. This indicates that carbon in sample 4 was the most heavily used because it has adsorbed more H_2S gas compared to sample 2 and 3.

It is also worth pointing out that high water content in the sample may inhibit the adsorption of H_2S and organic vapours; hence, it can yield early breakthrough curves, as previously mentioned by Rivin and Kendrick (1997). Samples 3 and 4 show water contents of 111.2 g/100g and 123.5 g/100g, respectively, indicating the carbon was not dried completely (water contents of greater than 50 g/100g have been measured). Also, even though regeneration of Sample 4 was achieved to a lower extent (26%) than those for Samples 2 and 3 (approximately 31 %), Sample 4 yet still showed the least sulphur content among the samples.

Fig. 2 shows the results of the TGA analyses on Samples 1 to 4. TGA is used to examine chemical reactions related to change of weight of a substance. Weight reduction occurred at 300 °C to 500 °C and it became constant at 800 °C. Activated carbon develops an endothermic reaction and as a result, it accumulates heat and the carbon decomposes rapidly leading to a negative peak. With regards to the melting effect, sample 1 and 2 at 60-70 °C started to evaporate and became dry at 600 °C. Similar condition was observed on sample 3 and 4 at 65-75 °C but became completely dry at a higher temperature due to more moisture present in the samples.

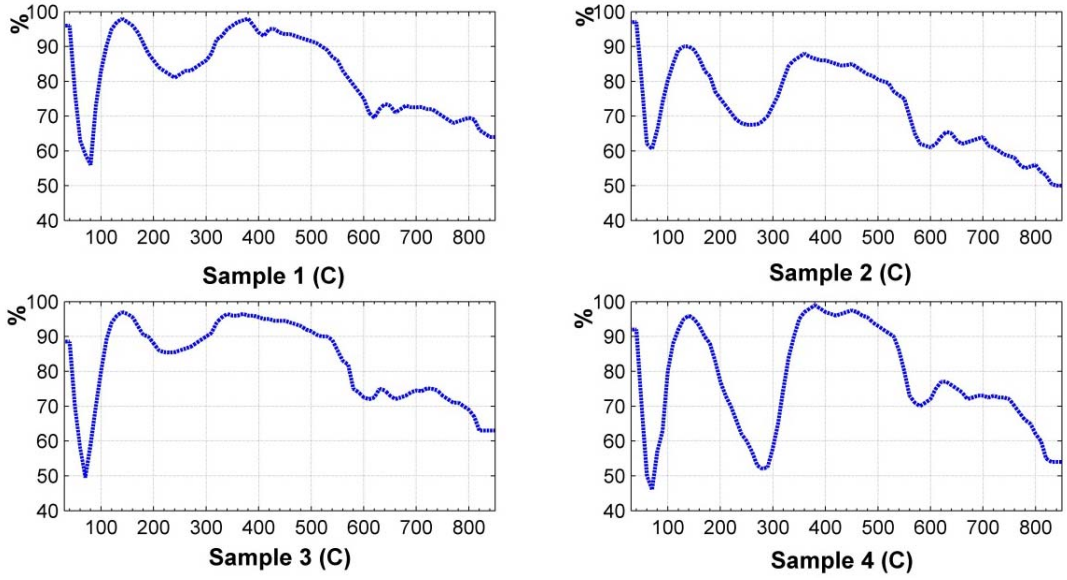

Fig. 2 TGA results for 4 samples.

In addition, depicted TGA curves for Sample 1 to 4 show weight losses due to: (1) Chemical reactions, which in this case are related to decomposition of the samples and, (2) physical transition from the wet to the dried samples.

4. Conclusions

A commercial activated carbon (Norit RST4) was used effectively to eliminate H_2S from gas streams coming from a municipal wet well in the Abu Dhabi Gulf resort. Even though activated carbon is an effective way to eradicate foul smell, it has a limited adsorption capacity and hence, when it becomes saturated, regeneration is required. Thus, sufficient amount of water will be used to wash the carbon and eliminate the sulphur build up. Regeneration of RST4carbon using water was evaluated. Saturated RST4carbon was subjected to two different regeneration methods: continuous and intermittent water regeneration.

It was demonstrated from the experiments conducted here that intermittent regeneration yields less total sulphur and organic loading in the carbon compared to the continuous regeneration procedure. Furthermore, the apparent density of the intermittently regenerated sample is slightly lower when compared to the continuously regenerated one, which indicates that the sample that was regenerated intermittently oxidized less than the sample that was regenerated continuously.

It has been proofed that the efficiency of an intermittent water regeneration process is higher than the continuous water regeneration of the sample. In addition, RST4 intermittent regeneration appeared to have minimal reduction of the adsorption capacity which is favourable to the end-user because it will promote longer life of the carbon, thereby minimizing the operational cost. Then, based on results presented in this study, it can be stated that intermittent water regeneration is the most effective method of regeneration for Norit RST4 carbon.

It is worth noting that this research has considered constant water properties such as pH, hardness and temperature due to the fact that water used for regeneration is normally the water coming directly from the municipality line. Additionally, VOC's were not measured because the purpose of this research was the removal of the most predominant gas which is H_2S. However, the presence of VOC's in significant amounts may contribute to a reduction in adsorption capacity of the carbon and, subsequently, the selection of the regeneration method. Also, adsorbed VOC's in substantial amounts cannot be washed out using water alone. In addition, VOC's can produce sulphonated hydrocarbons which are less soluble in water than SO_2 [36]. All the aforementioned limitations should also be considered when targeting a more effective water regeneration method.

Therefore, detailed research could be conducted to account for the effect of a wider range of water temperatures and pressures on optimal regeneration criteria. Further studies could be considered to determine the removal of minor contaminants since most sewage treatment plants would contain different mixtures of organic compounds. Lastly, additional research could be also conducted to assess the possibility of using oxidants like caustic to deal with the organic compounds adsorbed in the carbon media.

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