

Regeneration of adsorbent spent with Volatile Organic Compounds(VOCs)

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Abstract. This study investigates the adsorption of volatile organic compounds (VOCs), acetone and methyl Ethyl Ketone (MEK) on an activated carbon and its thermal regeneration. The adsorbent used in this study is an industrial grade granular activated carbon. A gravimetric method was used to determine the adsorption capacities of MEK and acetone. The adsorbent was regenerated at different temperatures and at varying moisture contents. The influence of these parameters on the regeneration efficiency was analyzed.

Keywords: VOC, Regeneration efficiency, activated carbon.

1. Introduction

Process of adsorption has been widely used in many industries at large scales for purification, separation and compliance purposes. The main categories of adsorbents used to adsorb long range of adsorbates with varying concentrations are, activated carbons (ACs), polymeric adsorbents and zeolites. The fact that adsorption can be used with liquid and gas process streams with high efficiency, low cost and friendly operating conditions has made it a usual process in various industries. ACs are used for a large range of applications. World demand for virgin activated carbon is forecasted to expand 5.2 percent per year through 2012 to 1.15 million metric tons. Consumption of activated carbons for industrial use has now become an indicator of development and environmental management efficiency. The per capita consumption of ACs (Figure, 1) per year is 0.5 kg in Japan, 0.4 kg in the U.S., 0.2 kg in Europe, and 0.03 kg in the rest of the world [1].

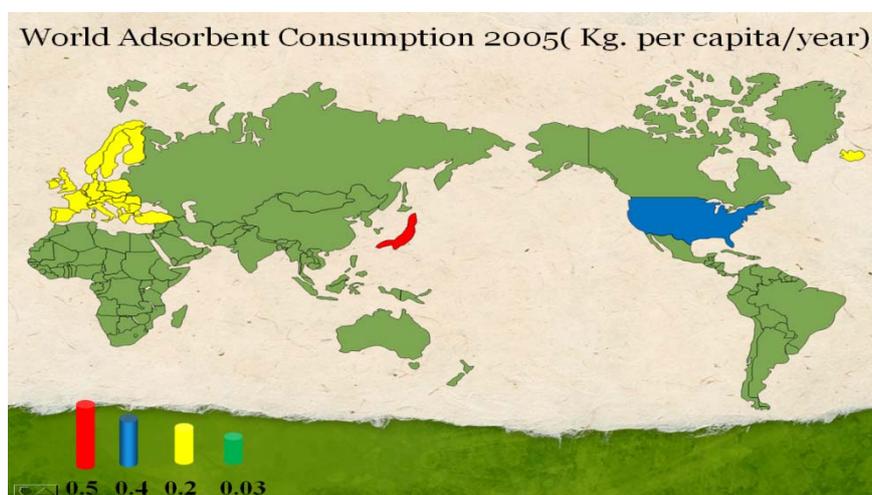


Figure 1 World adsorbent consumption (2005)

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After the adsorbents are exhausted, they are either to be disposed off or regenerated for use. This depends upon the demand, economics involved, and the kind of pollutant that was adsorbed. In many cases, spent adsorbents are to be treated as hazardous waste and need to be incinerated [2]. Adsorbents exposed to a gas or liquid stream will adsorb various compounds from the process stream. Trace compounds can be concentrated on the adsorbent during its use. They may be toxic and flammable [3] or explosive [4]. Exposure of spent adsorbents to ambient air may result in accumulation of heat due to adsorption of moisture and desorption of toxic adsorbates, creating hazardous conditions. Dumping spent adsorbents may also cause odour resulting in nuisance. The other option that industry can use is regeneration. Regeneration costs may equal to stabilization costs or just more than that, but if consumption of virgin adsorbent is reduced then multiple economic, industrial and environmental benefits can be gained (Figure 2). Extensive research has already been conducted regarding adsorption of pollutants onto ACs, but investigations on regeneration remain scarce [5]. In many cases, the adsorbates may be a resource and need to be recovered or concentrated to earn recovery credits [6]. Considering all above arguments it is evident that spent adsorbent need to be stabilized after being discarded. Because of high costs of production, stabilizing or proper disposal seem unwilling operations. Regeneration of adsorbents could prove double rewarding by stabilising adsorbents and recovering valuable adsorbates, thereby minimizing demand for virgin adsorbents.

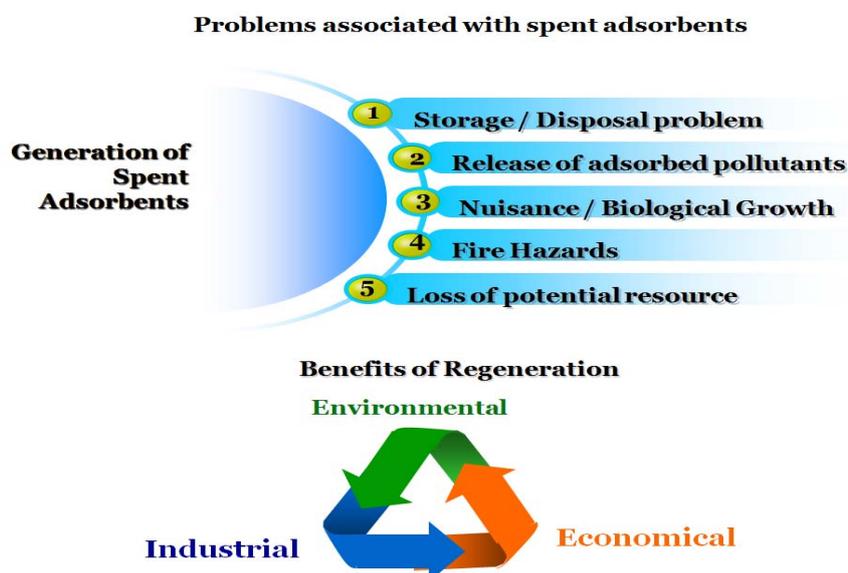


Figure 2 Problems and issues of spent adsorbent generation and benefits of regeneration

2. Materials and Methods

Adsorption and desorption thermodynamic data were measured onto a commercial activated carbon adsorbent, hereafter referred as BX-3, which was spent by Acetone and Methyl Ethyl Ketone (MEK). Some of the properties of BX-3 are given in Table 1. Properties of acetone and MEK are given in Table 2. A thermogravimetric analyzer coupled to a differential scanning calorimetry was used to measure the adsorption and desorption capacities at the equilibrium. The cell containing the adsorbent sample was placed under gas flowing. The VOC concentration, gas flowrate and humidity content were continuously monitored, as well as the temperature of the sample. A scheme of the experimental setup is shown in Figure 3.

Table 1 Physical properties of BX-3

BET surface(m ² /g)	Micro porosity(cm ³ /g)		Mesoporosity(cm ³ /g)	Bulk Density(g/ml)
1468	0.35-0.5mm	0.5-1.6mm	0.67	0.18
	0.07	0.56		

Table 2 Physical properties of Acetone and MEK

PROPERTY	ACETONE	MEK
Molecular Weight(g)	58.079	72.11
Boiling Point(⁰ C)	56.05	79.60
Density (g/m ³ at 20 ⁰ C)	0.79	0.80
Water solubility	miscible	miscible

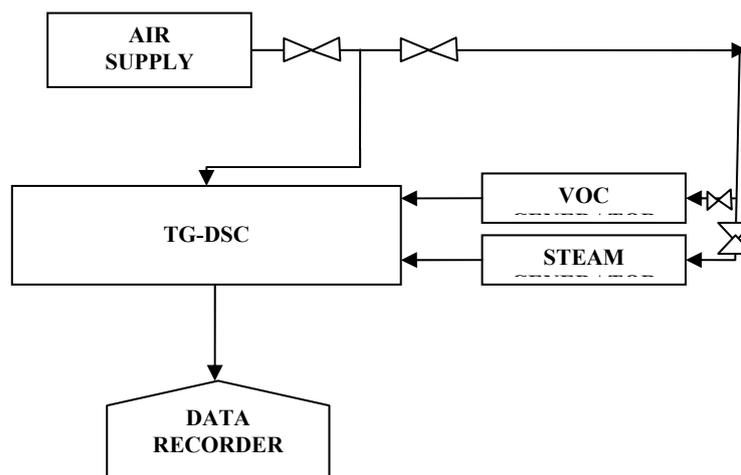


Figure 3 Schematics of experimental setup

Brooks's smart mass flow controller with digital control was employed to control flow into TG-DSC (Setaram 111). Flow rate for VOC adsorption as well as thermal regeneration was fixed as 5L/hr. Adsorption of VOCs was performed at 20⁰C and regeneration was conducted between 80⁰C and 160⁰C. A steam generator was used to produce air streams with varied moisture contents. The VOC concentration was controlled by using a tight glass syringe fitted in an infusion pump, which injected the liquid solvent at constant and predetermined speed into the inlet air stream. The VOC concentration was thus adjusted to 50g/m³ of Acetone or MEK. Adsorption was conducted till complete saturation of the adsorbent. The maximum time of regeneration was fixed to 2 hours. The following parameters were taken into account to assess the regeneration performances of spent adsorbents:

- **Adsorption capacity (Q)**

The adsorption capacity (Q) is defined as the amount of VOC adsorbed per unit of mass of adsorbent, once equilibrium has been reached.

- **Regeneration Efficiency(R.E)**

The regeneration efficiency is computed as:

$$R.E = Q_i / Q_o \times 100$$

Where Q_i is the adsorption capacity of the regenerated adsorbent and Q_o is the adsorption capacity of the fresh adsorbent.

- **Single-step stripping Efficiency(S.S.E)**

S.S.E is defined as the ratio of the adsorption capacity of the adsorbent to the adsorption capacity measured in the previous regeneration cycle.

$$S.S.E = Q_i / Q_{i-1} \times 100$$

3. Results and Discussion

Temperature has a great influence on the regeneration process of spent adsorbents. During regeneration of Acetone, a reasonable regeneration efficiency was achieved at 80°C, while regeneration of MEK was not sufficient at 80°C (Fig. 4{a}). This result is attributed to the lower boiling point of Acetone as compared to MEK (Table 2). In the case of MEK, the regeneration efficiency increased with the temperature, while it doesn't change significantly in the case of Acetone. The MEK has also a higher adsorption capacity than Acetone (Fig. 4{b}). The regeneration efficiency of adsorbent spent with MEK increased from 82.9% at 80°C to 97.5% at 160°C (Fig. 6). At 80°C, the MEK adsorption capacity declined during first and second regeneration cycles, after which it becomes steady. At 160°C, no change in the adsorption capacity is observed from a cycle to another. One can conclude that 80°C is not a sufficient temperature for effective regeneration of the adsorbent spent with MEK. This argument is also supported by the reported values of the S.S.E (Fig. 5). At 80°C, the SSE is minimal during the first regeneration cycle. After the second regeneration cycle, it is increased and remains fairly constant for the rest of the regeneration cycles. This implies that a constant amount of heel is left over the adsorbent. At 160°C, the S.S.E is fairly high since the first cycle, and does not vary significantly in the following cycles.

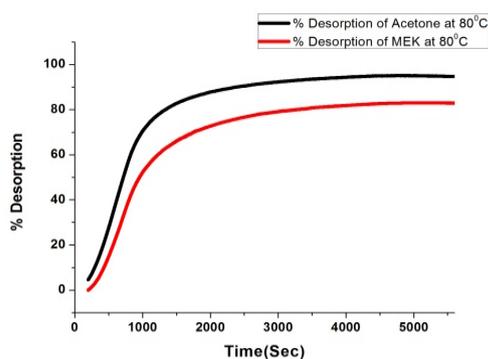


Figure 4(a)

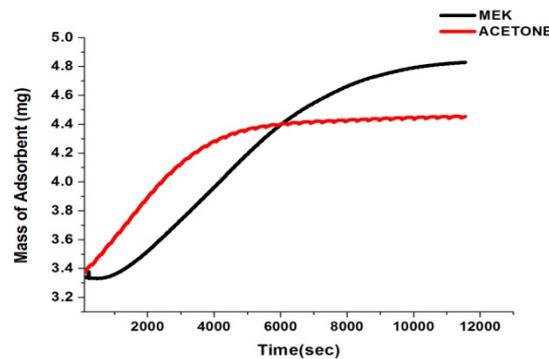


Figure 4(b)

Figure 4(a) Adsorption of Acetone and MEK on Adsorbent (BX-3), Figure 4(b) Effect of temperature on desorption of MEK

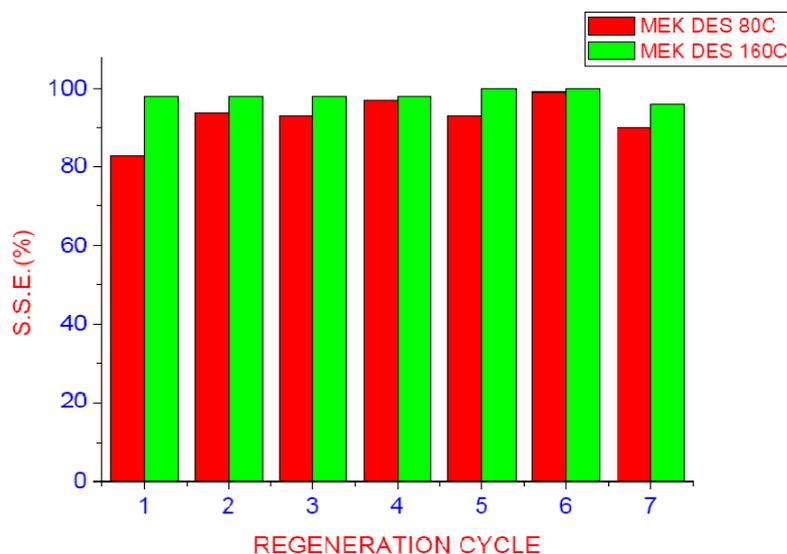


Figure 5 SSE of MEK spent adsorbent regenerated at different temperatures

After thermal regeneration tests with dry air, attempts were made to regenerate adsorbents with humid and hot air streams. The adsorbent was regenerated at 120°C with an air stream having 40% of relative humidity (RH). Regeneration with the wet air stream was achieved for 10 minutes; afterwards regeneration was pursued for one hour and fifty minutes under a dry air stream. A short period of wet air regeneration was selected in order to avoid long drying period and re-adsorption of water molecules on the adsorbent. The

regeneration performances obtained under these conditions were close to those obtained under dry air at 160 °C (Fig 6).

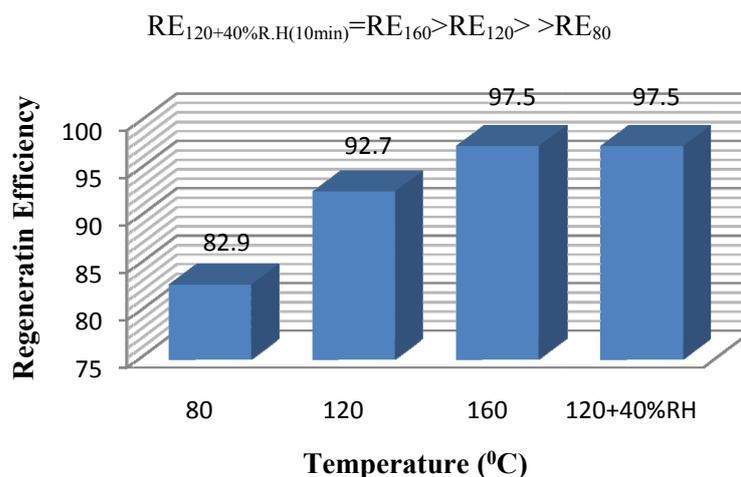


Figure 6 Regeneration Efficiency of MEK spent activated carbon at different temperatures and conditions

4. Conclusion

Thermal regeneration of Acetone and MEK from spent AC was conducted at temperatures between 80°C and 160°C. Regeneration efficiencies greater than 90% were found at temperatures above 120 °C. The adsorbent was regenerated with an almost equal regeneration efficiency at 160°C under dry air flowing and at 120°C by using during a short period, humid air stream. Complete regeneration under humid air stream was not so successful and yielded low regeneration efficiencies compared to dry air stream regeneration at the same temperature. This is explained by re-adsorption of water molecules during the cooling phase. To avoid this effect, the introduction of a short drying phase at high temperature or a longer drying phase at low temperature was required. However in both cases, the regeneration time increases which is not acceptable. Instead a short term steam injection (SSI) protocol was applied, during which air stream with 40% RH was passed for a short period, followed with a longer drying period. Such protocol was proved to be successful by meeting out both the objectives of reduced drying times and high regeneration efficiencies at a moderate temperature.

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6. References

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