



## CHAPTER VI

### SURFACTANT-ENHANCED CARBON REGENERATION IN A VAPOR- PHASE APPLICATION

#### 6.1 ABSTRACT

Coal-based granular activated carbon (GAC) is saturated with trichloroethylene (TCE) by passing air through a fixed bed adsorber. In surfactant-enhanced carbon regeneration, an aqueous solution of anionic surfactant, sodium dodecyl sulfate (SDS), is passed through the bed to induce desorption of TCE. More than 95% of the sorbed TCE was removed in the desorption operation with a 0.1 M SDS solution at a superficial flow rate of 1 cm/min. The desorption rate of TCE from pores of GAC is limited by pore diffusion and not significantly affected by either the concentration of SDS in the regenerant (when well above the critical micelle concentration) or its flow rate. From the breakthrough curve of a subsequent adsorption cycle without a flushing step following the desorption, only 7% of the virgin carbon effective adsorption capacity is observed for the regenerated carbon. With a water flushing step following the regeneration step, the effective adsorption capacity is significantly improved to about 15% of that of virgin carbon. Increased temperature of the flushing water also enhances the effective adsorption capacity of the regenerated GAC. Separate batch adsorption-desorption isotherms of SDS on GAC support the enhanced desorption of SDS at elevated temperatures. The drastic reduction in the effective adsorption capacity of regenerated GAC results from the residual SDS remaining in the pores of GAC as confirmed by thermal gravimetric analysis. Both the regeneration and water flush steps are rate limited under conditions used here.

## 6.2 INTRODUCTION

Because of its enormous adsorption area per unit weight, primarily hydrophobic surface, and convenient use form, granular activated carbon (GAC) has been widely used in removal and recovery of organic compounds from liquid and gas streams [1,2]. Spent activated carbon must either be replaced with fresh adsorbent or regenerated to restore its adsorptive capacity. Regeneration generally greatly improves the economic viability of carbon adsorption. An *in-situ* regeneration process is highly desirable economically, environmentally, and for energy conservation compared to hearth regeneration where the carbon is transferred from the fixed-bed to a furnace. Currently available regeneration methods include thermal regeneration [3-8], chemical regeneration [9-14], solvent regeneration [15-17], microwave regeneration [18-20], supercritical fluid regeneration [21], ultrasonic regeneration [22-24], and biological regeneration [25].

Surfactant-enhanced carbon regeneration (SECR) is an *in-situ* regeneration technique in which a surfactant solution at surfactant concentrations well above the critical micelle concentration (CMC) is passed through the spent GAC. The sorbate desorbs and solubilizes into surfactant aggregates called micelles formed above the CMC which can greatly increase total sorbate solubility into aqueous solutions. Following this sorbate desorption step, residual adsorbed surfactant can be flushed from the GAC using water. The SECR method for GAC consists of three main steps: passing a surfactant solution through the activated carbon bed, rinsing of excess surfactant by water, and drying the bed with warm air. The process scheme is shown in Fig. 6.1.

Previous studies of SECR by our group for both liquid-phase and vapor-phase applications have shown that organic sorbates can be desorbed by SECR; however, the effective adsorption capacity of the regenerated carbon in subsequent cycles was reduced compared to virgin carbon [26-28]. In liquid phase applications, Leng and Pinto [10] investigated the regeneration mechanism of activated carbon which adsorbed four kinds of organic compounds by using anionic surfactant, sodium dodecyl sulfate (SDS) and cationic surfactant, cetyltrimethylammonium

bromide (CTAB). Bhummasobhana *et al.* [28] used SECR to regenerate carbon saturated with phenol from waste water. In gas-phase applications, Roberts *et al.* [27] used SECR to regenerate carbon containing either toluene or amyl acetate adsorbed from polluted air. In the liquid phase application [28], a substantial (40%) reduction in adsorption capacity of the GAC was observed in a subsequent cycle. Experimentally, it is more difficult to accurately measure this capacity in a gas-phase application compared to liquid phase as is done in this paper for the first time.

The objective of this work was to investigate the regeneration of gas-phase GAC saturated with trichloroethylene (TCE) and adsorption capacities of regenerated GAC in a fixed-bed adsorber. The temperature of the water flush is a variable which has not been studied previously. The adsorption-desorption isotherms of SDS on GAC were also measured to aid in interpretation of adsorption bed results.

### 6.3 MATERIALS AND EXPERIMENTAL SECTION

#### Materials

A coal-based granular activated carbon designed for gas-phase use, BPL 4x10 provided by Calgon Carbon Corporation (Pittsburgh, USA), was used in the adsorption experiment. The reported BET surface area and appearance density of GAC is 1,100-1,200 m<sup>2</sup>/g and 0.5 g/cm<sup>3</sup>, respectively. The pore size distribution of the GAC has been reported [29]. The carbon was pretreated by boiling in deionized water for 1 h followed by rinsing with deionized water 3 times to eliminate fine particle and water soluble matter, then drying in an oven at 110°C for 24 h. Analytical grade TCE 99% from Lab-Scan Analytical Sciences (Dublin, Ireland) was chosen as the model sorbate. The surfactant used as the regenerant was an anionic surfactant, chemical grade sodium dodecyl sulfate (95%) supplied by APS Ajax Finechem (Auburn, NSW, Australia). Water for regenerant preparation and flushing was deionized water. In adsorption-desorption isotherm experiments, SDS with 99% purity from Sigma Chemical Co. was used.

## Methods

### Dynamic adsorption isotherms and regeneration

The adsorption and the regeneration experiments were performed in a 2.5 cm ID glass jacketed column packed with the GAC. Dynamic adsorption isotherms of TCE on GAC were obtained by using 9 g of GAC (corresponding to the bed height of 3.6 cm) which is sufficient to provide good breakthrough curves for both fresh and regenerated GAC. The height of the bed was fixed by an adjustable plunger to minimize the void volume in the bed. The breakthrough curve or outlet TCE concentration from an air stream on GAC at 30°C was measured in the column. The temperature of the column was maintained at 30°C by circulating water from the temperature controlled water bath to the jacket of the column. A constant feed concentration of TCE in simulated TCE contaminated air was generated by continuously bubbling the air stream into a temperature controlled TCE saturator before mixing this TCE saturated air stream with the main air stream in a mixing chamber at a pre-calculated ratio. A simplified flow scheme of the experimental setup is shown in Fig. 6.2.

Before each dynamic adsorption experiment, the fresh GAC was dried with warm air. A simulated contaminated air stream containing 1,030 ppmv TCE was then fed to the GAC bed at a flow rate of 450 mL/min. The TCE concentration in the inlet air stream and in the effluent air stream from the GAC column were analyzed using a gas chromatography (Perkin Elmer Model AutoSystem XL) equipped with a Carbowax column and a thermal conductivity detector (TCD).

The regeneration of the saturated bed consists of 3 main steps: 1) desorption of sorbed TCE by passing the surfactant solution through the GAC bed, 2) flushing the excess surfactant with water, and 3) drying the bed with warm air. The desorption of sorbed TCE from TCE-saturated GAC was performed by introducing the surfactant solution into the adsorption column as shown in Fig. 6.2. The temperature of the GAC bed was kept at 30°C. A solution of SDS at the desired concentration (0.1 - 0.2 M) was fed upward into the TCE-saturated GAC column at the desired

flow rate (5-15 mL/min). The fractions of effluent solution were periodically collected and analyzed by a Perkin Elmer gas chromatograph with a Headspace Sampler equipped with a Carbowax column and a flame ionization detector (FID). Deionized water at the desired temperature (30 and 50°C) was fed upward at the desired flow rate (5-15 mL/min) to flush out excess surfactant. The fractions of effluent water were collected and analyzed by a Shimadzu total organic carbon analyzer. After the flushing step (or after the desorption step), the GAC bed was drained and dried with heated air stream (50°C) until the moisture in the effluent air was less than 2% relative humidity.

### **Equilibrium adsorption/desorption of SDS on GAC**

In order to gain more understanding of the mechanisms governing the desorption of SDS, adsorption-desorption isotherms were generated at 30, 50 and 70°C. A series of SDS solutions at various concentrations ranging from 0.1 to 50 mM was prepared using deionized water. About 0.1 g of pretreated GAC was placed in 25 mL screw-cap vials and 10 mL SDS solution was added to the vials. The vials were shaken at the desired temperature (30-70°C) at 100 rpm for 120 h. A separate set of experiments showed that 96 h was adequate for SDS adsorption on GAC to reach equilibrium. After equilibration, the SDS solution was decanted and SDS concentration was analyzed by a TOC-5000 Shimadzu total organic carbon analyzer. In the desorption experiment, the decanted solution was diluted five times, followed by the same procedure. The vials were shaken at the desired temperature (30-70°C) at 100 rpm for 120 h. as separate set of experiments showed that 72 h was adequate for SDS desorption from GAC.

A TG 50 Metler thermal gravimetric analyzer (TGA) was used to confirm the residual SDS remaining on the GAC surface. The heating rate used in this experiment was 5°C/min.

## 6.4 RESULTS AND DISCUSSION

### Breakthrough curve for TCE from air

The adsorption experiment in the packed bed column containing 9 g of fresh GAC shows saturation at around 1,600 min or 113,207 pore volumes as shown in Fig. 6.3. A pore volume is the void space in the column containing the GAC and was measured to be 6.36 mL. The pore volume was determined by measuring the amount of water required to fill the bed packed with water-saturated GAC. We define the criterion for breakthrough as  $C/C_0$  (outlet concentration/feed concentration) of 0.01. The calculated amount of TCE adsorbed in the packed column was equal to 3.92 g or 3.31 mmole TCE/g of carbon which agrees with a literature value [30].

### Desorption of TCE

The CMC of SDS is 0.008 M [31]. At a total concentration above CMC, this is the surfactant concentration in monomer (unassociated form) while the remaining surfactant is present as micelles [31]. Organic solutes like TCE solubilize into the hydrophobic micellar interior [32]. So, total solubility of TCE in the aqueous solution increases with increasing surfactant concentration above the CMC. An aqueous solution of 0.1 M SDS (approximately 12xCMC) at a flow rate of 5 mL/min or a superficial linear flow rate of 1 cm/min was used for the desorption or regeneration step. The TCE concentration in the regenerant solution and the fraction of TCE desorbed is shown as a function of volume of regenerant solution used in Figs. 6.4 and 6.5, respectively. The sorbed TCE was rapidly desorbed with the first 300 pore volumes. The desorption of TCE then rapidly decreases until around 1,400 pore volumes and gradually decreases for the rest of the desorption step as shown in Fig. 6.4. From Fig. 6.5, it can be seen that, at 300 pore volumes of regenerant, approximately 25% of TCE easily desorbs while, at 1,400 pore volumes, another 55% desorbs with moderate difficulty, and the remaining is relatively difficult to desorb (the surfactant heel). The results suggest that the rate of removal of adsorbed

TCE from GAC is controlled by internal mass transfer of TCE which will be discussed later in this paper. Therefore, it can be expected that TCE adsorbed in mesopores would desorb easier than TCE adsorbed in micropores and submicropores, respectively. According to the results, it is speculated that the first portion (~25%) of TCE desorbed from GAC represents TCE adsorbed in mesopores of the carbon whereas the second portion (~55%) comes from TCE adsorbed in the micropores, and the last portion is from TCE adsorbed in the branched micropores (or submicropores). However, the results clearly show that more than 95% of TCE can be removed with around 2,600 pore volumes of regenerant solution as shown in Fig. 6.5.

### **Effect of water flushing**

After the desorption of TCE, the GAC bed was flushed with water at 30°C with a flow rate of 5 mL/min. This process was conducted until the SDS concentration in the effluent water was below the detectable limit to ensure that all readily desorbable SDS was removed. The calculated SDS removed by this flush step was 8 % of the adsorbed SDS. The breakthrough curve for TCE from air with the regenerated carbon when flushing is done shows a higher adsorptive capacity by a factor of two than the regenerated carbon when the flush step is omitted as shown in Fig. 6.3. The improvement of its adsorptive capacity could be due to the removal of SDS adsorbed on the GAC surface by the flushing, thus, providing more adsorption sites on the GAC for TCE to adsorb. However, the adsorption capacity of the regenerated carbon with the flushing is relatively low (about 15% of its original capacity). The residual surfactant on the GAC is responsible for the reduced adsorption capacity of the regenerated carbon and the greater amount of residual SDS, the worse the effect. This is the same behavior observed in previous work for SECR of liquid phase GAC [28]. It was optimistically hypothesized that the residual adsorbed SDS might transition to a phase (e.g. precipitate) upon drying in a vapor-phase application which is not as detrimental to subsequent adsorption capacity, but this was not observed here.

### **Effect of regenerant flow rate**

Experiments using TCE saturated GAC were carried out with 0.1 M SDS at flow rates of 5, 10, and 15 mL/min (corresponding to the superficial velocities of 1, 2, 3 cm/min, respectively) to desorb TCE from the carbon surface. The concentration profiles of TCE in the regenerant solution are shown in Fig. 6.6. All concentration profiles of TCE show the same trend; initially, concentration of TCE in effluent solution increases with an increase in the SDS solution volume passing through the bed and reaches a maximum before gradually decreasing and finally remains almost constant. This heel of residual adsorbed TCE which is difficult to remove during desorption might be due to the coadsorption with the strongly adsorbed surfactant which has been shown to be difficult to remove during the water flush step. From Fig. 6.6, the maximum TCE concentration in the effluent from different regenerant flow rates were observed at the same time. Moreover, the fractions of TCE desorbed were almost independent of flow rate when plotted as a function of time as seen in Fig. 6.7. In other words, at 0.1 M SDS, the rate of TCE removal does not depend on the flow rate of the SDS solution but rather on the contact time with the regenerant solution. The results suggest that the desorption of TCE could be conducted at low flow rate which would reduce the material cost. In addition, it can be concluded that the rate of TCE removal is controlled by the internal mass transfer of TCE from the micropores of GAC to the bulk solution. The rate of desorption is mass transfer limited, not equilibrium limited at the superficial velocities and surfactant concentrations used here, both of which are reasonable for fixed bed adsorption columns.

### **Effect of surfactant concentration in regenerant**

Two sets of experiments using TCE saturated GAC were conducted with 0.1 and 0.2 M SDS at the flow rate of 5 mL/min to desorb TCE from the carbon surface. The concentration profiles of TCE in SDS solution are shown in Fig. 6.8. At the beginning, TCE concentration in the regenerant solution with 0.2 M SDS is higher



than that with 0.1 M. This might be caused by TCE which is adsorbed in the mesopores. The adsorbed TCE in the mesopores is more easily desorbed than that in the micropores. However, in general, the amount of TCE desorbed is almost the same with the increase in pore volume (or time of desorption) as seen in Fig. 6.9. This indicates that the rate of TCE removal does not depend on the concentration of SDS in the regenerant solution but the TCE removal rate is controlled by the internal mass transfer of TCE from the micropores to bulk solution, consistent with regenerant flow rate effects. The regeneration step being rate-controlled has also been concluded from prior studies of SECR [27]. The negligible effect of SDS concentration on TCE desorption observed here suggests that the desorption of TCE may sufficiently be conducted at low surfactant concentration which would favorably result in the reduction of the surfactant cost.

#### **Effect of water flow rate during flush step**

Three sets of TCE desorbed GAC beds were flushed with deionized water to remove residual SDS (excess SDS in the bed void and the SDS adsorbed on the GAC surface). The flow rates of flushing water were 5, 10, and 15 mL/min (corresponding to the superficial velocities of 1, 2, 3 cm/min, respectively) and results are shown in Fig.6.10. There is no effect of flow rate on fractional removal of SDS when plotted as a function of time. This implies that the removal of SDS is rate limited under these conditions as was the regeneration step. This is not in agreement with previous studies [27, 28], in which the flushing step was concluded to be equilibrium limited which might be due to the use of different type of GAC.

#### **Equilibrium adsorption/desorption of SDS on GAC**

The adsorption-desorption isotherms of SDS on the GAC surface at 30°C shown in Fig. 6.11 (a) were constructed in order to substantiate the hypothesis that strongly or irreversibly adsorbed SDS residue is present on the regenerated GAC surface. From the figure, hysteresis of adsorption and desorption isotherms is clearly

observed. The adsorbed SDS during desorption is greater than that during adsorption at the same equilibrium concentration, indicating some irreversible adsorption, supporting the existence of a difficult to desorb surfactant heel observed during column studies.

In addition, the adsorption-desorption isotherms at higher temperature, 50 and 70°C, were studied to assess conditions, at which residual SDS can be efficiently removed. The adsorption-desorption isotherms are shown in Figs. 6.11 (b) and 6.11 (c). The hysteresis between the adsorption-desorption isotherms are still observed, however, the hysteresis loops at these two temperatures are narrower than the one at 30°C. These data indicate that a smaller surfactant heel may be present when flushing with water at elevated temperature.

### **Effect of flushing water temperature**

TCE desorbed GAC beds were flushed with deionized water at 30°C and 50°C at a flow rate of 5 mL/min. The results shown in Fig. 6.12 indicate that the SDS removal approximately doubles when the flushing water temperature is increased from 30°C to 50°C. The results of the effect of water flushing temperature are consistent with the adsorption-desorption from Figs. 6.11 (a) and 6.11 (b) isotherms at 30 and 50°C.

From the thermograms in Fig. 6.13, one can observe the rate of weight loss of regenerated GAC due to the decomposition of adsorbed SDS during heating in TGA. The higher the rate of weight loss of regenerated GAC, the more adsorbed SDS decomposes from GAC surface. The thermograms indicate that SDS remains on the GAC in all cases. The amount of SDS remaining on the regenerated GAC without water flushing is greater than that with the water flushing at 30°C and 50°C, respectively. So, elevated flushing solution temperatures are beneficial in SECR in reducing the deleterious residual surfactant heel, consistent with the column flushing results as well as the hysteresis in the adsorption/desorption isotherms.

## 6.5 CONCLUSIONS

In this work, the regeneration of TCE saturated GAC in packed-bed gas-phase adsorbers by using SDS was studied in order to investigate the parameters affecting the regeneration. The results showed that more than 95% of TCE can be removed by using a 0.1 M SDS concentration in the regenerant. The water flushing step improved the adsorption capacity of the regenerated carbon by the factor of two compared with the regenerated carbon without the water flushing (15% and 7%, respectively). The adsorption capacity for virgin carbon of the regenerated carbon was reduced to about 15% of its original capacity which is similar to previous work using cationic surfactants and liquid phase application. TCE desorption by this method is internal mass transfer limited as the regenerant flow rate and SDS concentration in the regenerant do not significantly affect the removal rate. The irreversibly adsorbed surfactant left as a heel following regeneration is very difficult to desorb. However, when flushing temperature is increased from 30°C to 50°C, the SDS removal doubled. The adsorption-desorption isotherms indicate that the surfactant heel is related to a fraction of the SDS which is irreversibly adsorbed. This heel is the cause of reduced adsorption capacities at breakthrough for TCE from air for the regenerated versus virgin carbon.

## 6.6 ACKNOWLEDGEMENTS

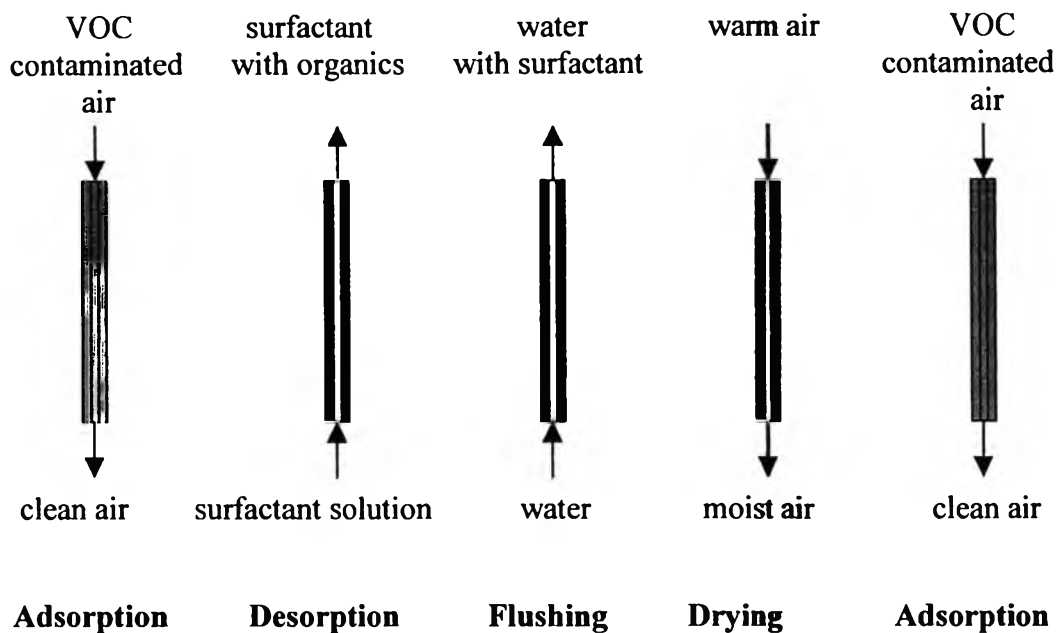
The authors would like to thank Ramkhamhaeng University for providing scholarships. The Petroleum and Petrochemical College, and Ratchadapisake Somphot Endowment Fund, Chulalongkorn University provided financial support. The author also would like to thank Calgon Carbon Corporation for providing GAC. Financial support was received from the sponsors of the Institute for Applied Surfactant Research at the University of Oklahoma, including Akzo Nobel, Clorox, Conoco/Phillips, Church and Dwight, Ecolab, Halliburton, Huntsman, Oxiteno, Procter & Gamble, Sasol, Shell, and Unilever.

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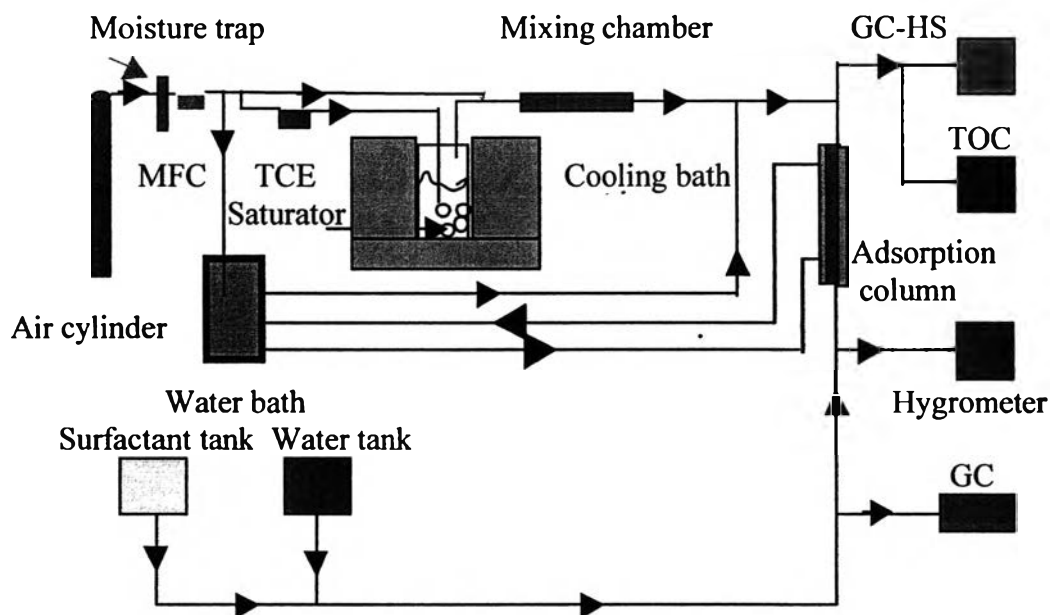
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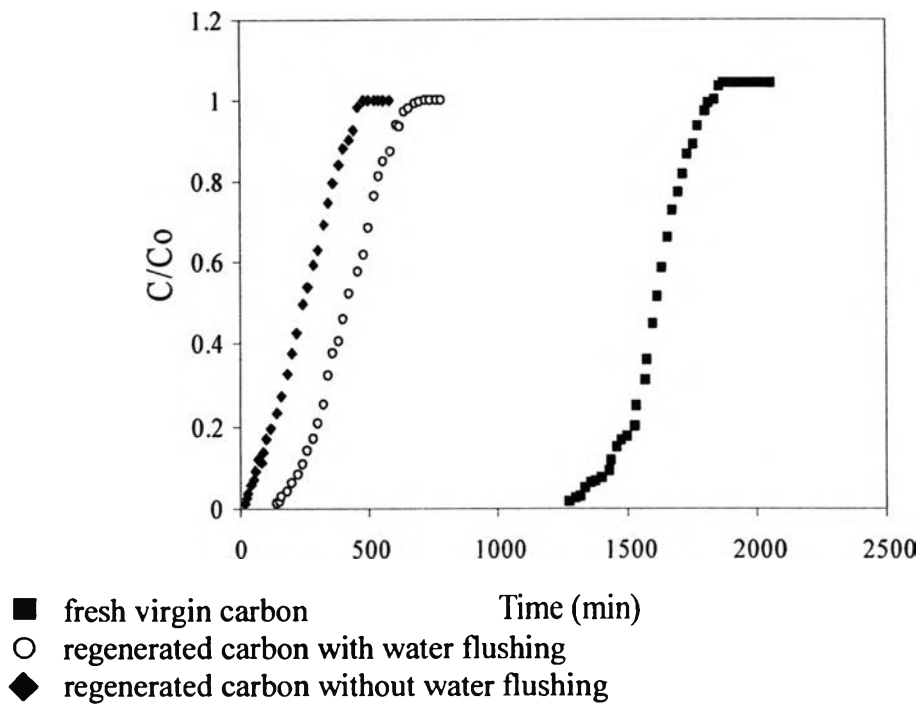


**Figure 6.1** Surfactant-enhanced carbon regeneration for a vapor-phase GAC application.



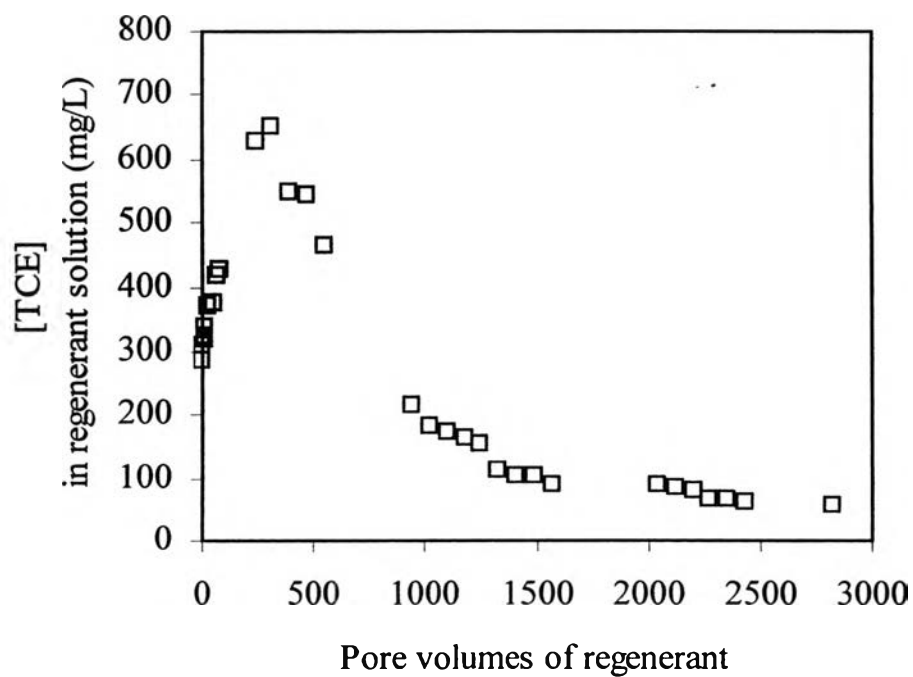
GC = Gas chromatograph (gas analysis chromatography)      GC-HS = Head space gas chromatography  
MFC = Mass flow controller      TOC = Total organic carbon analyzer

**Figure 6.2** Schematic diagram of the experimental set up.



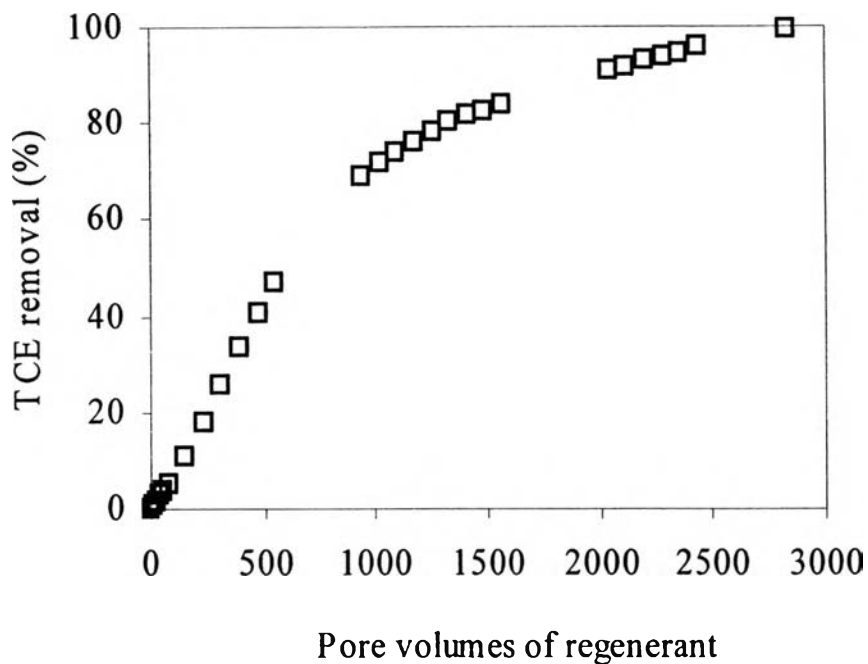
( $C_0$ ,  $C$  = concentration of TCE in influent and effluent air stream, respectively)

**Figure 6.3** Breakthrough curves of fresh and regenerated carbon.

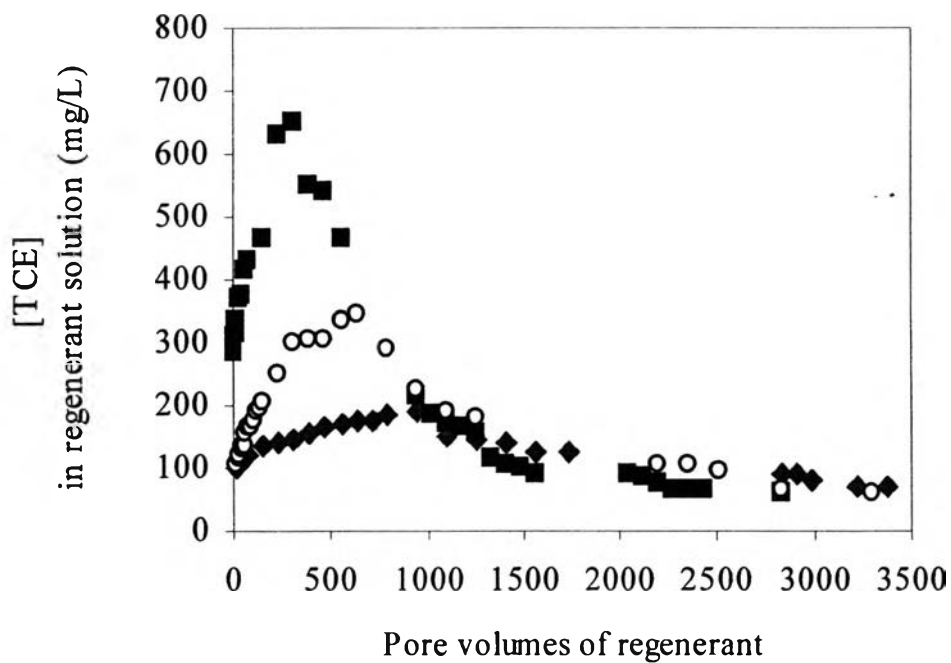


**Figure 6.4** TCE concentration in effluent regenerant solution with [SDS] of 0.1 M and a flow rate of 5 mL/min.

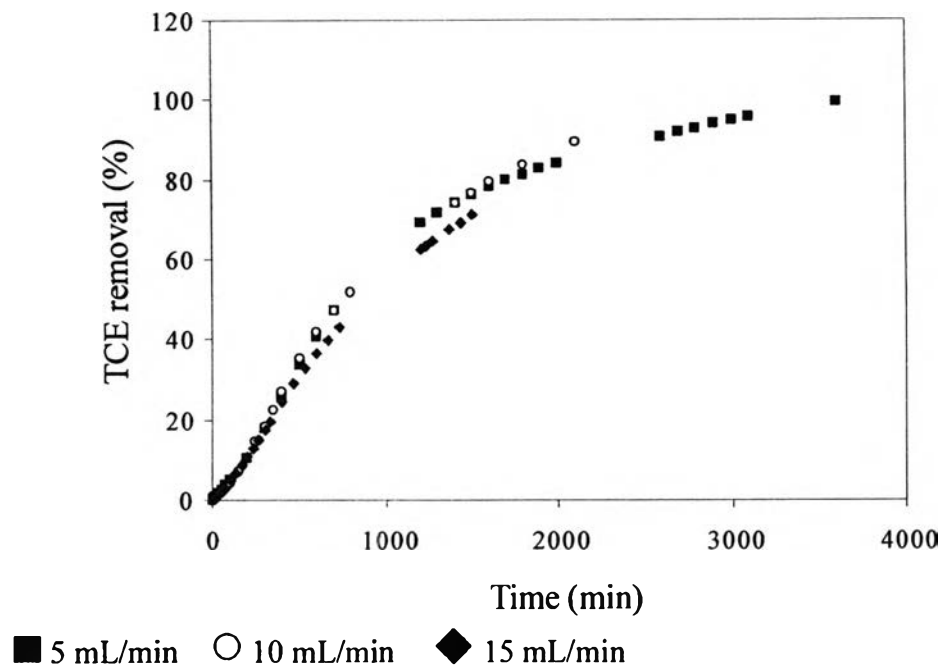




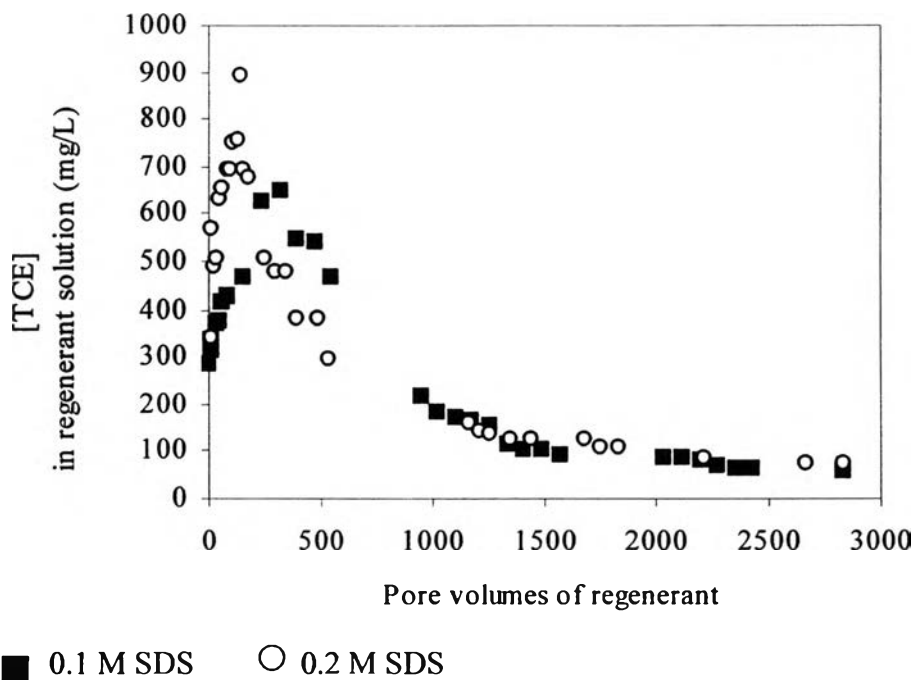
**Figure 6.5** Fractional TCE removal during regeneration with [SDS] of 0.1 M and a flow rate of 5 mL/min.



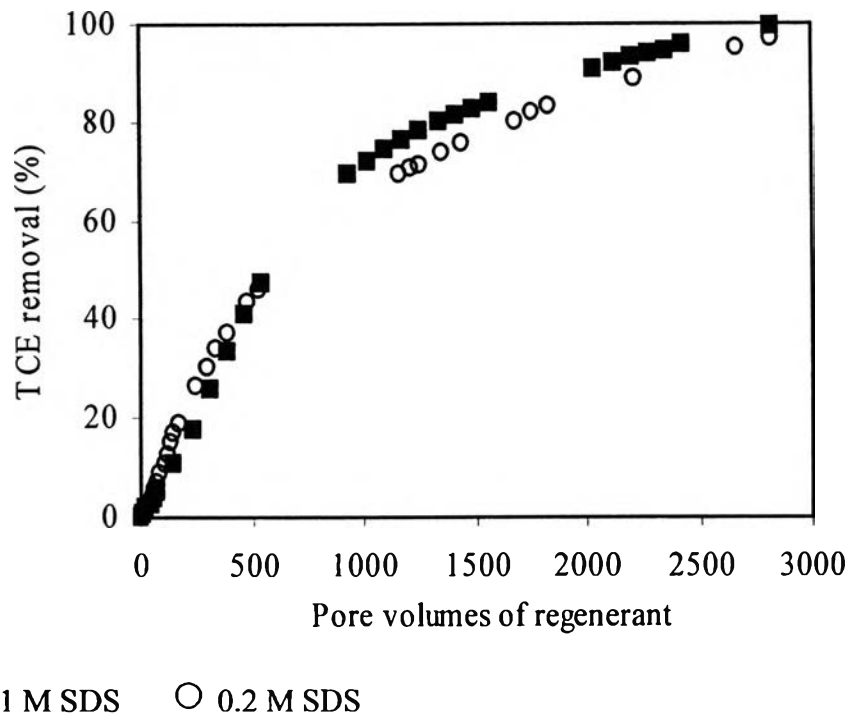
**Figure 6.6** TCE concentration in effluent regenerant solution with [SDS] of 0.1 M at several flow rates.



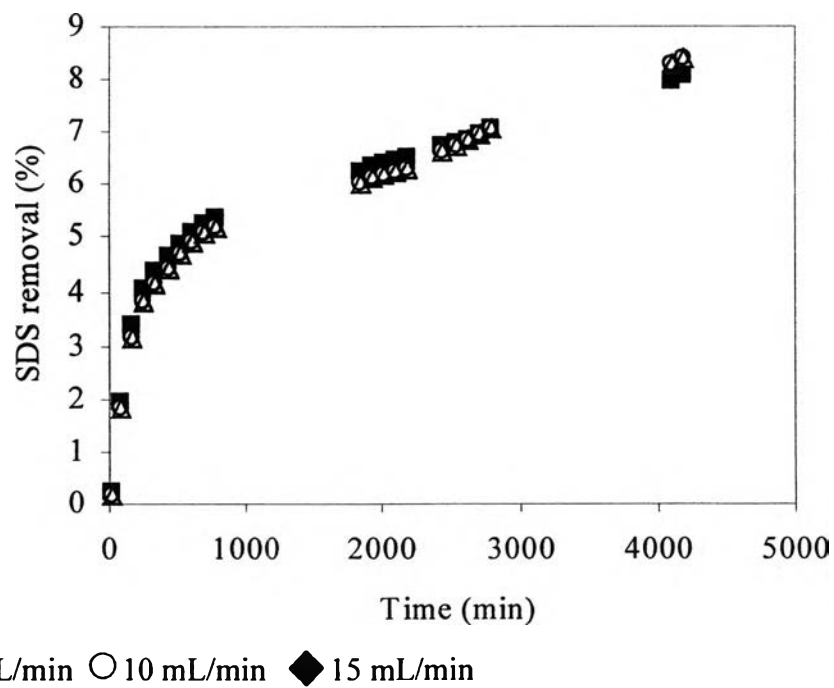
**Figure 6.7** Fractional TCE removal during regeneration with [SDS] of 0.1 M at several flow rates.



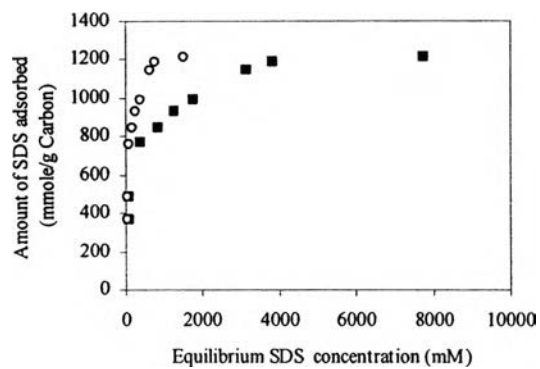
**Figure 6.8** TCE concentration in effluent regenerant solution with [SDS] of 0.1 and 0.2 M at a flow rate of 5 mL/min.



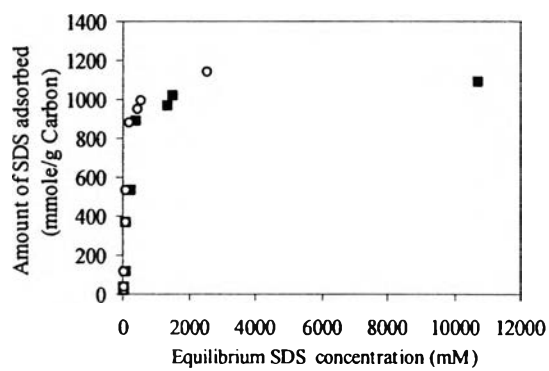
**Figure 6.9** Fractional TCE removal during regeneration with [SDS] of 0.1 and 0.2 M at a flow rate of 5 mL/min.



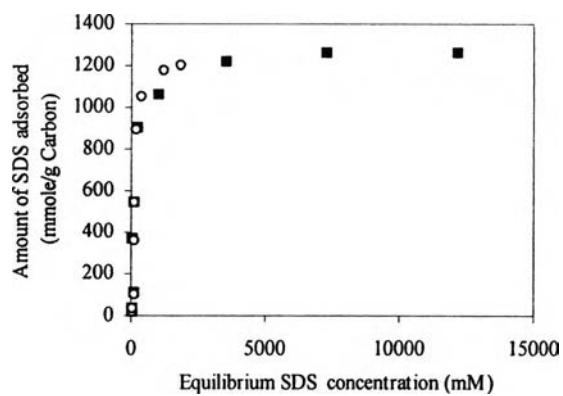
**Figure 6.10** Fractional SDS removal during flush with water at several flow rates.



(a)



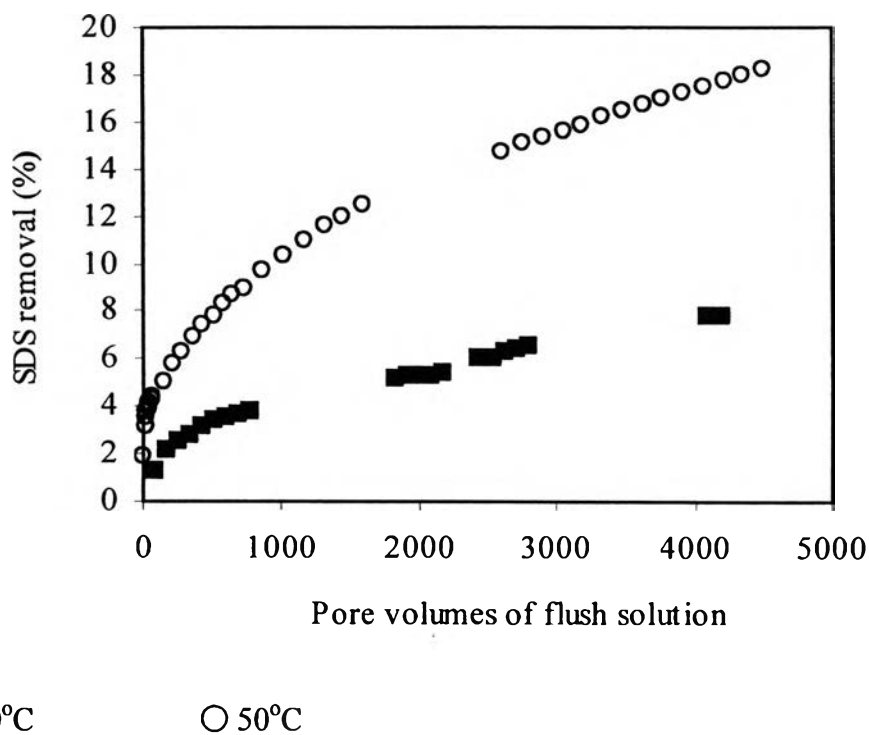
(b)



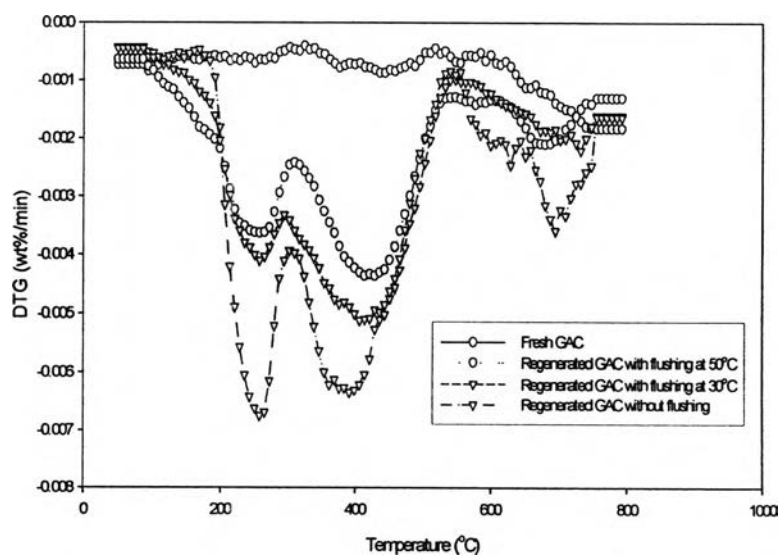
(c)

■ adsorption      ○ desorption

**Figure 6.11** Adsorption-desorption isotherms of SDS on/from GAC  
(a) 30°C (b) 50°C (c) 70°C



**Figure 6.12** Fractional SDS removal during flush with water at 30°C and 50°C.



**Figure 6.13** Thermograms from thermal gravimetric analysis of GAC (at a heating rate of 5 °C/min).