

Capture of Organic Vapors Using Adsorption and Electrothermal Regeneration

Patrick D. Sullivan¹; Mark J. Rood, M.ASCE²; Katherine D. Dombrowski³; and K. James Hay⁴

Abstract: Activated-carbon-fiber cloth (ACFC) is an alternative adsorbent to granular activated carbon (GAC) for removing and recovering organic vapors from gas streams. Electrothermal desorption (ED) of ACFC provides rapid regeneration while requiring less energy compared to traditional regeneration techniques used with GAC. This paper provides proof-of-concept results from a bench-scale ACFC adsorption system. The automated system captured 1,000 ppmv of hazardous air pollutants/volatile organic compounds (HAPs/VOCs) from air streams and demonstrated the use of ED, using ac voltage, to recover the HAP/VOC as a pure liquid. The desorbed HAP/VOC condensed onto the inner walls of the adsorber and was collected at the bottom of the vessel, without the use of ancillary cooling. Seventy percent of the HAP/VOC was collected per cycle as condensate, with the balance being retained in the regenerated adsorber or recycled to the second adsorber. ED with in-vessel condensation results in minimal N₂ consumption and short regeneration cycle times allowing the process to be cost competitive with conventional GAC-based adsorption processes. This technology extends the application of carbon adsorption systems to situations that were previously economically and physically impractical.

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Introduction

The Clean Air Act Amendments of 1990 mandate that maximum achievable control technology (MACT) be applied to control the emissions of hazardous air pollutants (HAPs) from numerous industrial sources (USEPA 1990). MACT may consist of the installation of new pollution control equipment or modification of the process such as the use of low-emission chemical substitutes. Many MACT standards were promulgated during the mid-to-late 1990s, and have led to implementation of these standards within the past 5 years.

These regulations have fostered the development of new air pollution control technologies for HAPs and other volatile organic compounds (VOCs). Some industrial users of HAPs/VOCs are not completely satisfied with chemical substitutes, because their performance may be inferior to that of the products they have replaced. The development of new technologies would allow continued use of higher-performance chemicals, re-use of those

chemicals at the source, and lower overall emissions of HAPs/VOCs to the environment. Concerns over ozone-depleting chemicals and greenhouse gas emissions have provided additional incentives to examine how new technologies can be used to capture and recover these gases. It is also beneficial for these devices to capture and recover the HAPs/VOCs instead of converting them into greenhouse gases (e.g., CO₂) or acids (e.g., HCl), as occurs during oxidative techniques.

Air pollution control for gas streams containing dilute (<10,000 ppmv) organic contaminants typically utilizes adsorption, either alone or as a concentration step prior to subsequent treatment (Ruthven 1984). Adsorption is the most energy-efficient method by which to separate dilute organic vapors from gas streams that allows recovery of the adsorbate (Ruthven 1984). Adsorbents are typically regenerated through thermal-swing or pressure-swing processes (Smisek and Cerný 1967). Development of a more efficient economical regeneration process implies some combination of using better adsorbents and improving the methods for desorption.

Activated-carbon fibers (ACFs) show great promise as a new adsorbent and catalyst support in competition with more-traditional activated carbons (Mays 1999). Carbon fibers can be woven into cloth and then activated to form activated-carbon-fiber cloth (ACFC). ACFCs are generally made from rayon, polyacrylonitrile (PAN), or phenolic (Novoloid) (Hayes 1981) resin. The fibers are woven into a fabric and then activated in steam or CO₂ at high temperatures (e.g., 800°C). In this paper we discuss ACFCs made from Novoloid. The diameter of the ACFC fibers used for this work is 12.3±1 μm (Lo 2002), which can result in two times faster adsorption dynamics than for granular activated carbon (GAC) (Tsai et al. 2000). Also, the equilibrium adsorption capacity of ACFC is as high as 250% the adsorption capacity of commercial GACs (Ramirez et al. 2001). The large adsorption capacity of ACFC is partially explained by the large percentage of pores being micropores (~93%), with large micropore volumes (e.g., 0.71 cm³/g), and narrow pore size distribution (Sun et al.

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1998; Lordgooei 1999; Hsi et al. 2000). ACFCs also have large surface areas, which range from 1,000 to 2,400 m²/g, with a typical value being 1,800 m²/g (Foster et al. 1992; Lo 2002). The adsorption of compounds onto ACFC is modeled well by Dubinin-type equations (Cal et al. 1994; Mangun et al. 1998; Ramirez et al. 2000, 2004). Multicomponent adsorption of acetone and benzene onto ACFC has also been modeled using ideal adsorbed solution theory (Cal et al. 1997). A significant advantage of ACFC is that it is produced from a synthetic feedstock. ACFC therefore does not contain impurities common to GAC that can catalyze oxidative reactions and lead to decomposition of the adsorbate and to fires within the adsorber (Zerbonia et al. 2000; Hayes and Sakai 2001).

Direct electrical resistance heating, or the Joule effect, of GAC as a thermal-swing method of adsorbent regeneration was studied more than 3 decades ago (Fabuss and Dubois 1970). This technology has not been widely implemented because of the difficulties in maintaining uniform electrical contact in the granular medium, and the difficulty in mechanically supporting the medium while electrically isolating the adsorbent in an adsorber of commercial size. Nevertheless, electrothermal desorption (ED) of GAC continues to be investigated at the bench scale for applications such as the recovery of methyl bromide (Snyder and Leesch 2001).

The development of ACFs that are woven into a fabric has provided a continuous conductive medium of adsorbent that is well suited for ED. Regeneration by ED has been shown to be more rapid and energy efficient than heating the activated carbon with steam or inert gas (Petkovska and Mitrovic 1994b). ED also allows desorption and condensation of a volatile solvent without mixing the resulting condensate with water. ED therefore eliminates the need for an adsorbent drying step, and eliminates recovered solvent/water separation processes, as required with conventional steam regeneration technology. Heating with inert gas also does not introduce water, but instead produces a much more dilute effluent than ED, and does not allow deposition of energy to the adsorbent independently of the gas flow rate.

Initial experiments that explored resistive heating of ACFC showed promise (Economy and Lin 1976). Subsequent developments include the demonstration of adsorption/ED cycling of ACFC with 1,1,1-trichloroethane (Petkovska et al. 1991), modeling of the ED process on the microscopic and macroscopic levels (Petkovska and Mitrovic 1994a,b), demonstration of a pilot-scale system (Baudu et al. 1992), demonstration of ED in an ACFC-acetone system including recovery by integrated cryogenic condensation (Lordgooei et al. 1996) and detailed modeling of adsorption dynamics (Lordgooei et al. 2001). A survey of electrothermal regeneration techniques has recently been performed (Bathen and Schmidt-Traub 1999). The drop in pressure through ACFC (Subrenat et al. 2000; Sullivan et al. 2000, 2001) and electrical properties of ACFC (Subrenat et al. 2001; Sullivan et al. 2001) have been studied to provide design information for ACFC adsorbers.

In an effort to adsorb HAPs/VOCs and then recover them as a pure liquid without using steam or heated inert gas, we developed a system that featured ACFC in hollow cylinders that also used refrigeration to condense the vapor during regeneration of the adsorbent (Sullivan et al. 2001). This adsorber was of a much simpler configuration than those used in previous studies. Simplicity was achieved by requiring fewer parts and the vessel body was constructed of a single piece of metal without an electrical insulation layer. This adsorber also resolved material compatibility issues with solvents such as methyl ethyl ketone (MEK) and

methylene chloride, demonstrated selective removal of HAPs/VOCs from humid gas streams that contain the HAP/VOC, allowed smaller drops in pressure across the adsorber, and allowed faster ED of the adsorbent. However, the system still required a refrigeration unit to condense the desorbed vapor from the gas stream and used significant amounts of N₂ during regeneration of the adsorbent, which accounted for approximately 25% of the system's operating costs (Rood et al. 1999).

This paper documents an improved method to capture and recover HAPs/VOCs wherein the compounds adsorbed onto the ACFC cartridges are regenerated electrothermally (*electrical-swing adsorption*) at a very rapid rate, causing the adsorbate to condense within the adsorption vessel itself and produce two-phase flow of the effluent during regeneration. Rapid ED with in-vessel condensation results in significant reductions in system complexity, cycle times, and N₂ consumption compared to previously demonstrated systems. This new system also operates without the use of steam, heated inert gas, vacuum, or a refrigeration system.

MEK was chosen as the primary adsorbate because it is one of the most widely used industrial solvents (USEPA 1997); it is often in dilute and difficult-to-control waste streams, such as exhaust from spray painting and paint stripping operations, and it is a HAP/VOC. Another reason for selecting MEK was to demonstrate that the system is not susceptible to the bed fires common to conventional GAC systems when capturing aldehydes and ketones (Zerbonia et al. 2000; Hayes and Sakai 2001). Additional common solvents [*n*-hexane, 2-pentanone (MPK), 4-methyl-2-pentanone (MIBK), and toluene] were subsequently examined as representative adsorbates that contain select chemical functional groups that have a wide range of relative vapor pressures to demonstrate the generality of this method. Relative pressure is defined as the ratio of the adsorbate's partial pressure to its saturation vapor pressure at 1 atm total pressure and the temperature of interest.

Experimental Apparatus and Methodology

Experimental Apparatus

Overall System Design

The bench-scale system consisted of a gas-generating system, two parallel adsorbers containing hollow cylinders of ACFC, a silicon control rectifier (SCR) power supply, a data acquisition and control system, and a total hydrocarbon analyzer with a flame ionization detector (FID; Fig. 1).

This new system is similar to one previously described (Sullivan et al. 2001), except that the cylindrical aluminum enclosures were replaced with cylinders of Pyrex glass to allow visual observation of condensation inside the vessel during ED; outlets of both ACFC adsorbers were replaced with conical bases for rapid drainage of the HAP/VOC condensate during electrothermal regeneration; the cryogenic condenser was removed from the system; and the adsorber vessels acted as passive heat exchangers, allowing the adsorbate to condense onto the vessel's inner walls during electrothermal regeneration of the adsorbent. A detailed description of the previous experimental system was reported by Sullivan et al. (2001) but will be briefly described here for clarity.

Gas-Generating System

Purified compressed air was divided into two streams that were metered by mass flow controllers. The relative humidity of the

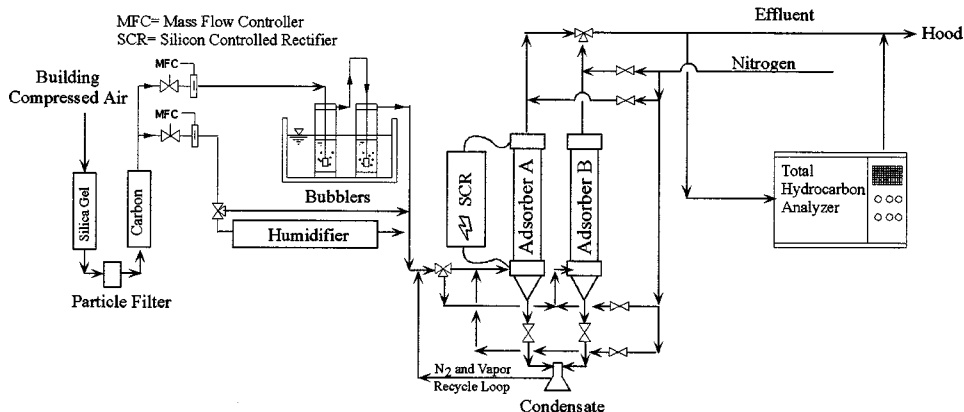


Fig. 1. Activated-carbon-fiber cloth adsorption-rapid electrothermal desorption system

gas stream was $4 \pm 1\%$. The first air stream passed through two fritted-glass bubblers containing liquid MEK, creating a saturated stream of vapor. The resulting gas stream was then blended with the second gas stream to produce a gas stream at a specified gas flow rate and MEK concentration which was fed into the adsorber. In later experiments the bubbler system was replaced with a syringe pump (KD Scientific, model KDS200) and evaporator to simplify the gas generation system.

Adsorber

Each adsorber consisted of a Pyrex glass enclosure (75-mm outer diameter, 4-mm wall thickness) containing two annular ACFC filter cartridges that were made from 14.6-cm \times 67.3-cm sheets of ACFC (American Kynol, Inc., ACC-5092-20). Each sheet formed a cylindrical roll with eight layers and an effective height of ~ 11.4 cm. The mass of ACFC in each adsorber was 24.0 g. The ACFC used in the previous adsorber [Fig. 2(a)] was transferred in the same configuration to the new adsorber [Fig. 2(b)] to perform all single-adsorber adsorption and desorption tests. Continuous electric-swing adsorption tests of the adsorber were performed with ACFC from a different production lot, which had the same dimensions but had a mass of 30 g.

The gas flows illustrated for the new adsorber reported here occur while the adsorbers are removing HAP/VOC from the gas stream. The outlet to drain the condensate is closed at that time. During electrothermal regeneration the condensate outlet is opened, and N_2 is injected into the gas inlet and gas outlet. N_2 flows from inside to outside through both ACFC cartridges and then exits through the condensate outlet. In this configuration the upper cartridge is regenerated with reverse flow compared to the adsorption cycle, and the lower cartridge is regenerated with forward flow. The valves directing the gas flows are the only moving parts used in the adsorption system.

A SCR provided an initial electrical voltage to the ACFC during regeneration. During regeneration the adsorbers were purged with 0.02–0.5 sLpm of N_2 gas (1 atm, 298 K). During regeneration, the N_2 flows from a pressurized cylinder through the regenerating adsorber, through the condensate collection vessel, through the recycle loop that merges with the influent of the on-line adsorber, through the adsorber that is online removing HAP/VOC from the system's inlet gas stream, and then to an exhaust hood.

Analytical and Ancillary Equipment

Data acquisition and control were achieved with a Keithley model 500 controller and a desktop personal computer. The total hydro-

carbon concentration in the effluent from the adsorber was monitored by a THC/FID. All voltage and current readings were measured with true root mean square (RMS) meters.

Methodology

Before each adsorption test, the THC/FID was calibrated with gas streams containing known amounts of HAP/VOC. For MEK, a compressed gas cylinder of MEK was obtained commercially and used as the standard. For gases of lower volatility, standard cylinders were unavailable. In this case, the gas and liquid flow rates

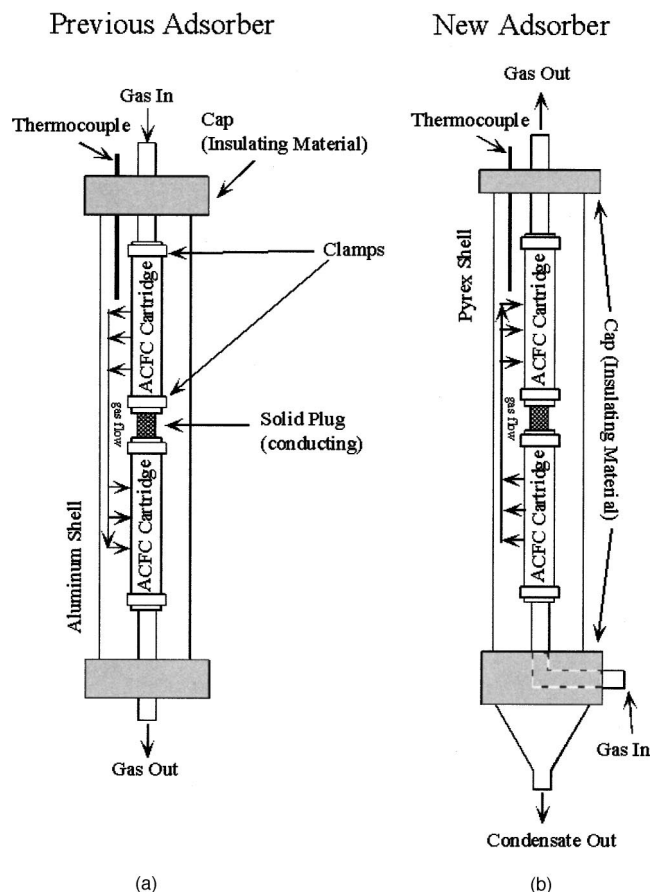


Fig. 2. (a) Previous adsorber (Sullivan et al. 2001); (b) new adsorber (present study)

from the syringe pump and mass flow controllers were calibrated with a graduated cylinder and a flowmeter (Bios International DryCal DC-2), respectively.

The gas-generating system delivered a flow of 5–60 sLpm of air that contained typically 1,000 ppmv of HAP/VOC to the adsorbers. The gas stream was measured with the THC/FID before and after each test. The gas stream then passed through the adsorber, and the adsorbate concentration was detected at the exit of the adsorber, allowing real-time detection of the temporal variability of adsorbate concentration. Flow was diverted to an alternate vessel when the exit concentration of the HAP/VOC reached a specified setpoint of either 5 or 50% of the inlet concentration. While gas flow was diverted to the alternate adsorber, the adsorber that contained the HAP/VOC was purged with N₂ to reduce the concentration of O₂ in the adsorber and was then electrothermally regenerated.

Results that describe temporal variation of the MEK concentration at the outlet of the adsorber were used to produce breakthrough curves. These curves plot the dimensionless adsorber outlet concentration versus the dimensionless breakthrough time. The dimensionless outlet concentration is defined as the adsorber's effluent gas-phase HAP/VOC concentration (C_{out}) divided by its inlet gas-phase HAP/VOC concentration (C_{in}). The dimensionless breakthrough time is equal to $t/t_{50\%}$, where t and $t_{50\%}$ are the duration the adsorber is on line and the duration for the effluent concentration to attain 50% of the influent concentration, respectively. Analysis of the breakthrough curves provides the total mass of MEK adsorbed (M_{sat}), the adsorption capacity of the ACFC, the throughput ratio (TPR), and the length of unused bed (LUB). The total mass of MEK adsorbed by the ACFC was determined to be

$$(M_{sat}) = \frac{P(MW)Q_{air}}{RT} \int_0^{t_{sat}} \left(\frac{C_{in}}{1-C_{in}} - \frac{C_{out}}{1-C_{out}} \right) dt \quad (1)$$

where P =total pressure of inlet gas stream; MW =molecular weight of adsorbate; Q_{air} =flow rate of carrier gas; R =ideal gas constant; T =absolute temperature of inlet carrier gas; C_{in} =mole fraction of adsorbate in inlet gas stream; C_{out} =mole fraction of adsorbate in exhaust gas stream; and t_{sat} =time at which C_{out} has reached its final, steady-state value because the adsorbent is saturated with respect to the adsorbate.

The adsorption capacity of MEK achievable by the ACFC in the adsorber is the ratio of M_{sat} to the mass of ACFC in the adsorber. TPR is defined as

$$TPR = \left(\frac{t_{5\%}}{t_{50\%}} \right) (100) \quad (2)$$

where $t_{5\%}$ =duration required for the effluent concentration to attain 5% of the influent concentration. As TPR approaches 100%, the time required to develop the mass transfer zone in the adsorber becomes insignificant compared to the time required to saturate the adsorbent.

The LUB describes the percent of adsorption capacity that is not utilized due to the length of the adsorption zone within the bed:

$$LUB = \left(1 - \frac{M_{5\%}}{M_{sat}} \right) (100) \quad (3)$$

Here $M_{5\%}$ =mass of adsorbate in the adsorber at 5% breakthrough time and M_{sat} =mass of adsorbate in the adsorber when the adsorbent is saturated. The TPR and LUB are used to quantify utilization of the activated carbon's adsorption capacity when used

under realistic operating conditions. Calculations for GAC adsorber design typically use a design factor of about 50% carbon utilization, which includes the LUB plus a safety factor (USEPA and Mussatti 2002).

The mass of desorbed MEK vapor (M_{desorb}) is described with the use of a material balance,

$$(M_{desorb}) = \frac{P(MW)Q_{N_2}}{RT} \int_0^{t_{fin}} \left(\frac{C_{out}}{1-C_{out}} \right) dt \quad (4)$$

where Q_{N_2} =carrier gas flow rate and t_{fin} =duration of desorption cycle. The total mass of desorbed material is the sum of that material in the gas and liquid phases that are released from the adsorber during the desorption cycle.

It was necessary to quantify the saturation vapor pressure of the organic vapor to calculate its mass that exits the adsorber during regeneration. The Wagner equation was used to determine the saturation vapor pressure for each compound (Reid et al. 1987):

$$\ln \left(\frac{P_{i,s}}{P_c} \right) = \left(\frac{VP_A x + VP_B x^{1.5} + VP_C x^3 + VP_D x^6}{1-x} \right) \quad (5)$$

where $P_{i,s}$ =saturation vapor pressure in bar; P_c =critical pressure (42.1 bar for MEK); $x=1-(T/T_c)$; T_c =critical temperature (536.8 K for MEK); and VP_A , VP_B , VP_C , and VP_D =Wagner constants (-7.71476, 1.71061, -3.6877, and -0.75169, respectively, for MEK). Wagner constants were not available for MPK, so the Antoine equation (Reid et al. 1987) was used to determine the value of $P_{i,s}$ in bar:

$$\ln P_{i,s} = A - B/(T+C) \quad (6)$$

where A , B , and C =Antoine constants (9.3829, 2,934.87, and -62.25, respectively, for MPK) and T is in units of K. Equilibrium adsorption capacities for the organic vapor and ACFC were needed to calculate the mass of adsorbate retained in the fabric after regeneration. The Dubinin-Radushkevich (DR) equation has been shown to accurately represent the adsorption capacity of ACFC for temperatures of 20–50°C and concentrations from 0.1 to 90% of the saturation vapor pressure for several relevant organic vapors:

$$W_i = W_{i,o} \exp \left[- \left(\frac{RT \ln(P_{i,s}/P_i)}{E_i} \right)^2 \right] \quad (7)$$

where W_i =volume of adsorbate per mass of adsorbent; $W_{i,o}$ =micropore volume per unit mass of adsorbent in cm³/g; and E_i =adsorption energy of adsorbate i in kJ/mole. The values of $W_{i,o}$ used for MEK and MPK were 0.724 and 0.749 cm³/g, respectively. The values of E_i used for MEK and MPK were 14.43 and 15.25 kJ/mole, respectively (Ramirez et al. 2000).

Results and Discussion

Breakthrough Curve Results

Dimensionless breakthrough curves for MEK from the previous adsorber [Figs. 3(a–c)] are compared with those from the new adsorber [Figs. 3(d–f)]. Breakthrough curves for flows between 5 and 60 sLpm and concentrations between 980 and 4,360 ppmv have a consistent breakthrough time and slope. The dimensionless outlet concentration does not achieve unity at the end of the tests because of the slow rate of adsorption to the ACFC that was contained behind the clamps used to assemble the cartridges, and possible experimental error. The total gas flow rates of 5 and 60

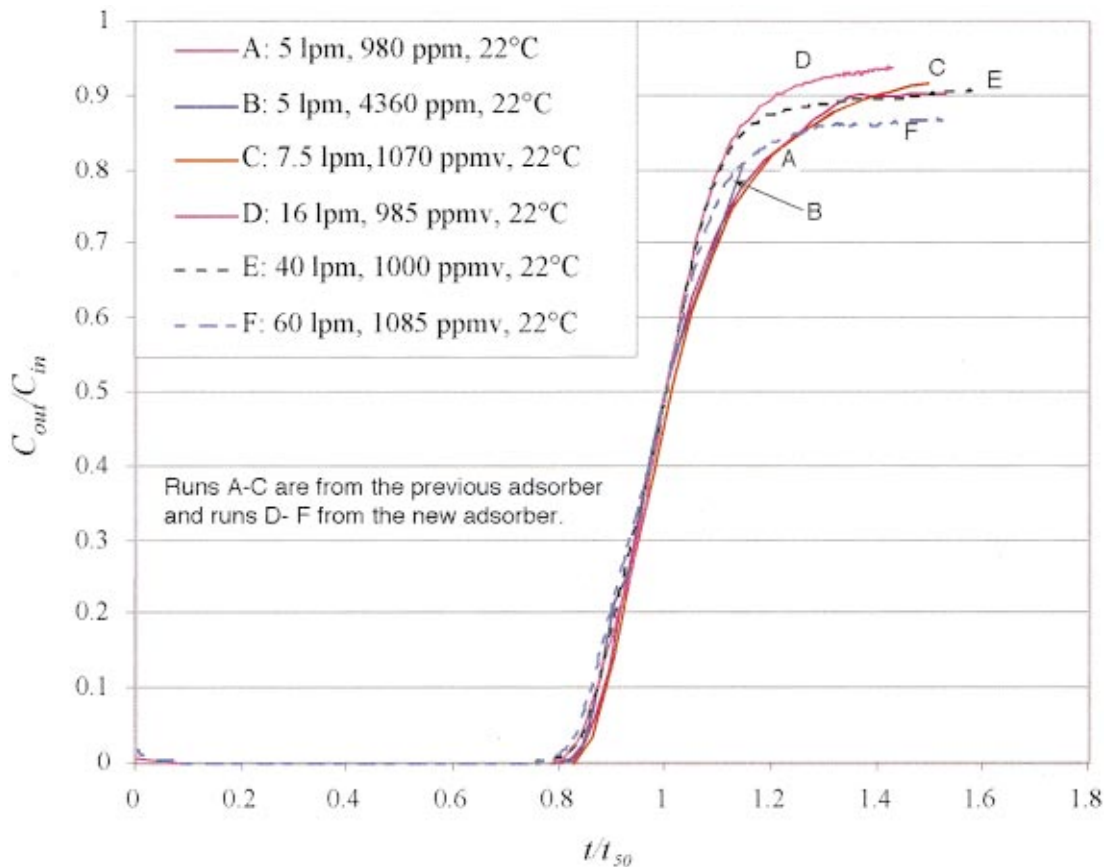


Fig. 3. (Color) Dimensionless breakthrough curves for methyl ethyl ketone and activated-carbon-fiber cloth system

sLpm equate to face velocities of 1.4 and 16.6 cm/s, respectively. The permeability of the fabric was measured at $13.87 \text{ m}^3/\text{min m}^2$ when operating at 125 Pa of pressure drop by an independent commercial laboratory (Grubb 2001). Independent testing for pressure drop of the assembled adsorber was also performed at flows up to $3.67 \text{ m}^3/\text{min m}^2$ ($r^2=0.998$, $n=11$) at the University of Illinois. Extrapolation of permeability data obtained with the adsorber that contained cartridges to the same 125 Pa of pressure drop yields a permeability of $18.29 \text{ m}^3/\text{min m}^2$, a difference of 24%.

ACFC was regenerated between tests by purging the vessel with N_2 and electrothermally heating to a target temperature of 150°C . The flow rate of 60 sLpm of air-containing MEK equates to a pressure drop of 1.52 kPa, which is lower than the pressure drop of most GAC adsorbers (Cooper and Alley 1994; Noll 1999). At a flow rate of 60 sLpm and a concentration of 1,000 ppmv MEK (2.94 g/m^3 at 1 atm, 298 K) the breakthrough time is 30 min. This is approximately equal to the minimum regeneration cycle time, and thus is the maximum flow rate that could be used in a continuously operated system for the mass of ACFC used, without reducing the concentration of the inlet vapor or using a three-vessel configuration. The breakthrough curves indicate that the TPR is constant over the range of projected operating flow rates of the system.

Desorption Test Results

Once the adsorber was saturated by 1,000 ppmv of MEK vapor, it was purged with N_2 and regenerated. The liquid MEK collection rate was measured volumetrically (Fig. 4).

The amount of time that power was applied to the adsorber during ED was reduced by more than a factor of 10 compared to the previous adsorber (i.e., from 45 min in the previous system to 4 min). The desorbed MEK rapidly condensed on the inside walls of the adsorber and drained through the outlet of the adsorber with the MEK-saturated N_2 gas stream. Subsequent desorption tests yielded similar results at 0.2 sLpm N_2 flow.

A material balance was performed to determine partitioning of the MEK upon completion of a desorption cycle. The total purge gas volume is approximately 2 L during heating of the ACFC. The mass of MEK in the purge gas stream accounts for 4% of the

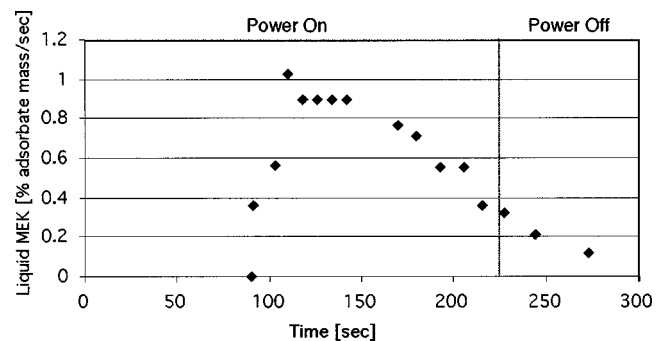


Fig. 4. Liquid methyl ethyl ketone collection rate during electrothermal desorption as percent of total adsorbed mass versus time; N_2 flow rate=0.5 sLpm, electrical power applied at $28 \text{ V}_{\text{RMS}}$ ac and 6–11 A; activated-carbon-fiber cloth was at equilibrium at 1,000 ppmv prior to desorption

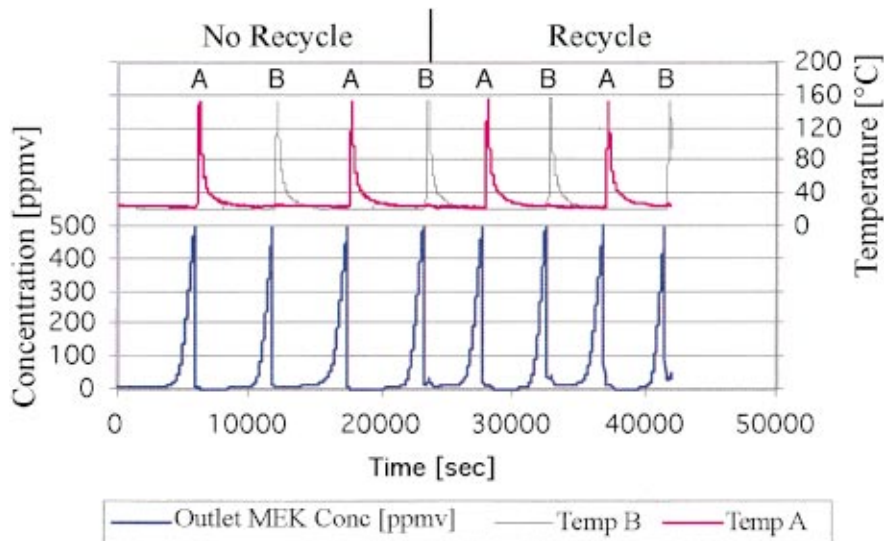


Fig. 5. (Color) Adsorber temperature and effluent methyl ethyl ketone (MEK) concentration versus time during continuous operation of the system with and without recycling of the effluent gas stream during electrothermal regeneration

total adsorbed mass, assuming the purge gas is saturated with MEK at 20°C (average temperature of the gas exiting the adsorber) during ED. The mass of MEK remaining in the ACFC after ED is 25% of the mass captured in the adsorption cycle determined by the DR equation. The remaining 71% of the MEK mass should be collected as liquid exiting the adsorber. Observed MEK condensate that was collected ranges from 67 to 74% of the MEK mass that was adsorbed from the air stream. A scaled-up version of this design with a larger adsorbent-to-surface area ratio is planned to allow mass and energy balances to be performed more effectively.

Continuous Adsorption–Desorption Cycling

The system was operated with continuous adsorption–desorption cycling with an inlet MEK concentration of 1,000 ppmv and total gas flow rate of 25 sLpm. These tests were used to demonstrate the functionality of the process in an uninterrupted controlled scenario. The concentration of vapor emitted from the system and the adsorbent temperatures were monitored in automated mode (Fig. 5). The first and second adsorbents in the sequence are designated adsorbent “A” and adsorbent “B,” respectively. The breakthrough concentration for the vessel adsorbing MEK was set at 50% of the system’s inlet concentration to observe and confirm the periodicity of the adsorption cycles for both adsorbents. When the effluent from the online adsorbent reached the breakthrough concentration, the gas flow was diverted to the alternate vessel and regeneration of the adsorbent was initiated. The desorbing vessel was purged with 0.5 sLpm of N₂ (2% of the total flow rate during adsorption). After 2 min had elapsed, 28 V_{ACRMS} was applied, causing the temperature of the adsorbent to increase from 22 to 160°C within 3.5 min. The voltage applied was reduced as necessary to maintain current below 11 A_{RMS}. The ACFC then experienced an exponential decay in temperature for 25 min after termination of the electrical power.

The system was operated with recirculation (shown in Fig. 1) and without recirculation of the MEK-saturated N₂ purge gas stream. During operation without recirculation, the recycle loop was vented to the exhaust hood instead of being merged with the influent of the online vessel. These two scenarios define the upper

and lower bounds of condensing the MEK from the recirculating N₂ purge gas stream. There was no recirculation of the purge gas stream during the first 25,000 s of the test, after which recirculation occurred for the remainder of the test. Breakthrough times (the horizontal distance between the peaks on the left-hand side of Fig. 5) for the first 25,000 s simulate a system that condenses all of the MEK vapor before the gas stream is recirculated. The breakthrough times for tests >25,000 s represent a system that does not condense the MEK beyond its saturation vapor pressure before the gas stream is recirculated. Recirculation of the effluent during ED without removing all of the MEK from the gas stream resulted in breakthrough times of ~1.3 h, a 20.2% reduction in t_{50} compared to the case with no recirculation of the effluent. The TPR changed from 87 to 80% and the LUB increased from 21 to 30% with a corresponding decrease in t_{50} . The calculated mass of MEK in the recirculated gas stream accounts for 60% (0.12 of 0.20) of the reduction in t_{50} . These results are very encouraging because they demonstrate that ancillary treatment (e.g., cooling) of the recirculated gas stream can be eliminated from the system with only a 20% reduction in breakthrough time for MEK. Construction and operation of a system without ancillary treatment is favorable to make the system less complicated, more compact, and less expensive.

Continuous recovery tests were subsequently performed with an array of solvents at a total gas flow rate of 40 sLpm to demonstrate the generality of this method. Single-component organic vapor tests were performed with MIBK, toluene, MPK, MEK, and *n*-hexane at 1,000 ppmv in dry air. The breakthrough setpoint of 5% of the system’s inlet HAP/VOC concentration was used, which resulted in overall removal efficiencies of 99.9%, measured by the system’s effluent concentration.

Breakthrough time ($t_{5\%}$) for vessel A initially decreased over time over the course of the continuous experiment for MPK and then stabilized (Fig. 6). The 5% breakthrough time for cycle No. 1 was longer than the subsequent breakthrough times because the ACFC had its largest initial adsorption capacity at the beginning of the cycle, when it was completely free of adsorbate. Subsequent breakthrough times were shorter because the adsorbent was not completely desorbed from the previous cycle and because the

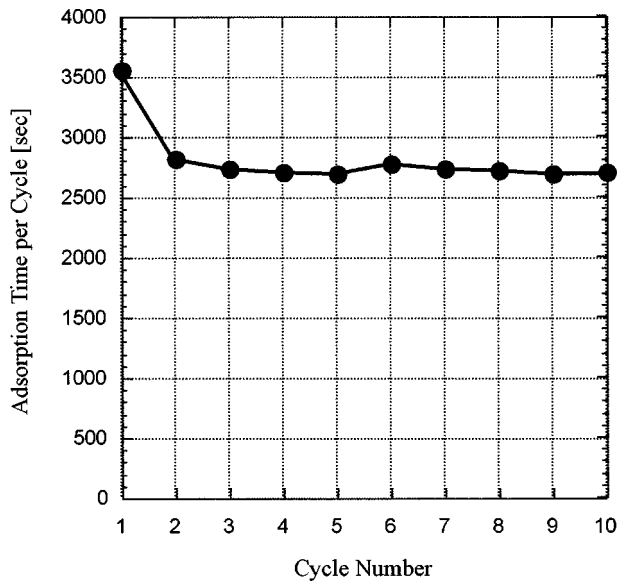


Fig. 6. Adsorption times for vessel A for continuous recovery of methyl *n*-propyl ketone

inlet gas stream contained recirculated MPK from the vessel that experienced ED. The cycle times achieved steady-state conditions after the initial cycle. This behavior was observed for all of the compounds tested except *n*-hexane.

The reduction in breakthrough time from the initial cycle to the subsequent cycles for MPK is 22%. This value can be compared with the amount of MPK predicted to remain in the adsorbent assuming the ACFC is in equilibrium with the purge gas stream at the end of each cycle. The equilibrium adsorbed mass of MPK at 1,000 ppmv and 20°C is 0.54 cm³/g. The equilibrium amount of adsorbed MPK at 36,000 ppmv (the concentration of the purge gas) and 200°C is 0.095 cm³/g. The residual MPK in the ACFC accounts for a 17.6% reduction in the breakthrough time after the initial cycle. The MPK mass in the recycled purge gas accounts for an additional 2.4% reduction in the breakthrough time. Therefore, the MPK that is residually adsorbed in the ACFC and retained in the recycled gas stream accounts for a 20% reduction in the breakthrough time, in comparison to the 22% reduction in breakthrough time that was measured during automated cycling of the system (Fig. 6).

Breakthrough curves for *n*-hexane revealed quite different behavior from that of the other organic vapors. Steady-state operation of the dual-vessel system was not achieved, as evidenced by the decrease in breakthrough times (Fig. 7). One reason why *n*-hexane was not captured on a continuous basis is revealed upon reviewing the desorption cycles. A desorption cycle time of at least 2,000 s is needed to ensure adequate desorption of the MEK and cooling of the ACFC for the adsorbent to regain its adsorption capacity before it experiences the next adsorption cycle (Fig. 8). Breakthrough times of less than 2,000 s will result in reduced adsorption capacities, premature breakthrough, and unstable operating conditions.

Of the compounds and conditions tested here *n*-hexane was the only alkane: it has the highest relative pressure and the lowest adsorption capacity for the ACFC. The high relative pressure also causes *n*-hexane to have the highest concentration in the recirculated purge gas stream. *n*-Hexane also exhibited film condensation on the inside walls of the adsorber, while all other compounds exhibited drop-wise condensation. Drop-wise

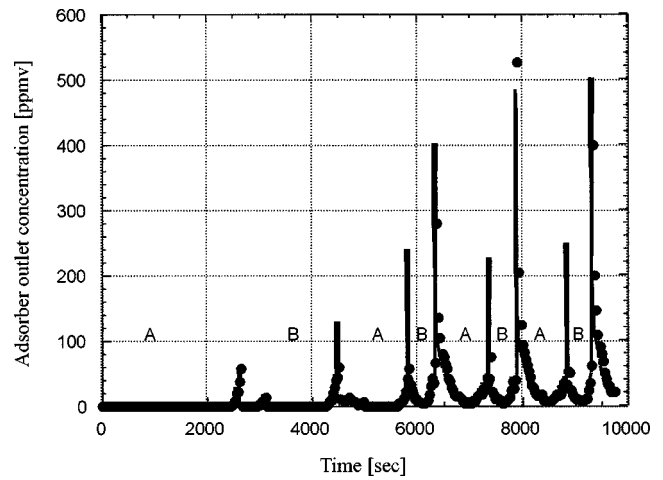


Fig. 7. Adsorption cycles for 1,000 ppmv *n*-hexane in air

condensation allows more rapid condensation of the vapor compared to film condensation (Incropera and Dewitt 1981). If condensation to the walls of the adsorber is the rate-limiting step in transferring the adsorbate from the gas to liquid phase, then the *n*-hexane concentration in the recycle stream could be greater than its saturation vapor pressure at 20°C. For *n*-hexane, the initial adsorption run time was less than that required for a complete desorption and cooling cycle. The elevated temperature at the end of the incomplete ED cycle resulted in higher concentrations in the recycled purge gas stream. Film condensation may also have contributed to higher concentrations in the recycled purge gas stream. The increased concentrations in the recycle stream further shortened the adsorption run time and resulted in the inability of the system to reach steady-state adsorption-desorption cycling. Increasing the amount of adsorbent in the vessels and treating the recycle stream (e.g., cooling) between the adsorbers will allow recovery of *n*-hexane on a continuous basis with this system.

A continuous-recovery test for a mixture of 500 ppmv MIBK and 500 ppmv toluene in dry air was performed to evaluate the effectiveness of the system to remove multicomponent vapors relevant to painting operations. The average time to reach 5% breakthrough for vessel A was 39.9 min, which is within 8% of the

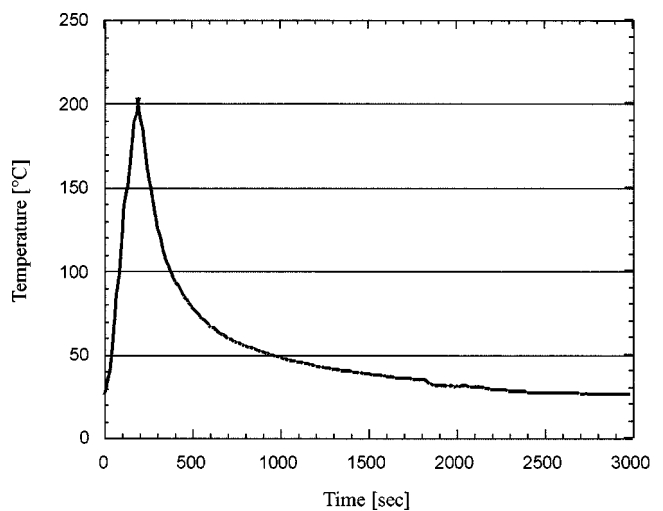


Fig. 8. Temperature profile of heating/cooling cycle measured near activated-carbon-fiber cloth surface

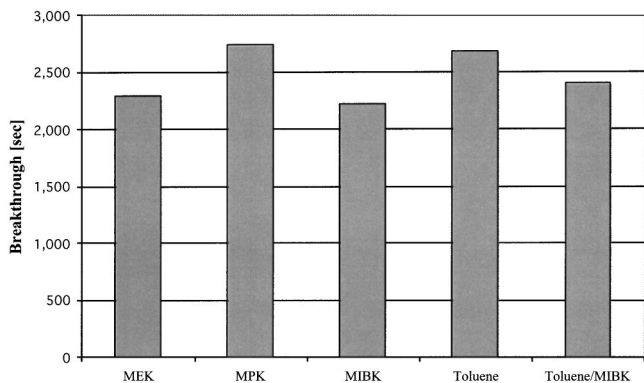


Fig. 9. Average breakthrough times for continuous adsorption-desorption tests

results for the single-component runs of MIBK at 1,000 ppmv. Breakthrough was measured as the total THC/FID response from MIBK and toluene in the gas stream, since the THC/FID does not differentiate between these organic compounds. The results for the mixture are comparable because the adsorption capacities of the two compounds are similar, and they are miscible within each other. These results illustrate that continuous recovery of multi-component vapors is possible with the system.

Breakthrough times for the tests described above are summarized in Fig. 9. Continuous capture and recovery was demonstrated for all compounds with a relative pressure between 9.5×10^{-2} and 1.97×10^{-2} . Effective capture and recovery of compounds with relative pressures as high as 0.47 (methylene chloride) will require additional treatment (i.e., cooling) of the recycle stream. Compounds with relative pressures lower than 1.97×10^{-2} have not been investigated, but they may be difficult to desorb, which would require higher regeneration temperatures, and could result in thermal degradation of the adsorbate or adsorbent.

Cost Estimates

A cost estimate for an ACFC/ED system of commercial size was completed to gain insight into the commercial viability of the technology. A system that could treat $68 \text{ m}^3/\text{min}$ of total gas flow rate was selected to be of sufficient size to be within the range of available cost correlation data that are compiled for conventional adsorption-based vapor recovery systems (Table 1). The cost analysis was performed following the general methods originally developed by Vatauvuk (1990) and the EPA Air Pollution Control Cost Manual (USEPA and Mussatti 2002). The following assumptions were made: the capital recovery cost (CRC) was amortized over 15 years at 7% annual interest rate, operator and mechanic hourly labor rates were set to 15 and \$17, respectively, electricity cost = \$0.06/kWh, and natural gas cost = \$0.55/therm. A "mobilization cost" was added to the GAC replacement labor. Also, an energy efficiency conversion factor of 0.85 for generating steam with natural gas was included in the analysis. For comparison purposes, a cost estimate for an equivalent-size GAC/steam-regenerated system was also prepared using the USEPA's cost-estimating software for air pollution control devices: *Air Compliance Advisor* (ACA), version 7.4 (USEPA 2000). ACA does not include the cost of auxiliary equipment, resulting in a low capital investment cost estimate, but the annualized cost is within 10% of

Table 1. Summary of Annual Costs for a Granular Activated Carbon (GAC) System Compared to an Activated-Carbon-Fiber Cloth (ACFC) System Designed to Control a $68 \text{ m}^3/\text{min}$ Dry Air Stream Containing 710 ppmv Toluene^a

Description	GAC (dollars)	ACFC (dollars)	GAC by ACA (dollars)
Adsorbent and adsorber vessels	76,060	86,209	
Auxiliary equipment	18,608	18,608	
Purchased equipment cost (includes 18% for tax, etc.)	111,708	123,684	66,640
Installation	33,512	37,105	
Indirect costs	34,629	38,342	
Total capital investment	179,850	199,131	107,291
Labor	9,315	9,315	
Maintenance	17,280	17,280	
Replacement of adsorbent	989	—	
Electricity	1,881	9,971	
Steam	5,635	—	
Cooling water	497	—	
Overhead	15,957	15,957	
Administrative costs and taxes	7,194	7,965	
Capital recovery cost	19,411	21,864	
Total annual cost	78,162	82,353	71,704

Note: ACA = Air Compliance Advisor, version 7.4.

^a15-year amortization schedule.

the corresponding cost calculated for the GAC system when using the EPA Air Pollution Control Cost Manual (USEPA and Mussatti 2002).

The cost to purchase the toluene that is captured and recovered for this application is \$249,759 compared to the annual cost of the ACFC system of \$82,353. The value of toluene recovered is based on 100% capture and recovery efficiency, continuous operation of the source, and a cost of toluene = \$2.29/L. The toluene is reagent grade, manufactured for the paint industry (Qi 2002). A HAP/VOC capture and recovery system using ACFC and rapid ED is projected to be cost competitive with traditional GAC adsorption technology with the GAC system costing \$78,162. This cost estimate was developed without the benefit of optimization of ACFC/ED technology.

The ACFC system uses much less adsorbent than the GAC system, but the packing density is much lower, resulting in a larger vessel for the ACFC system. For the bench-scale ACFC system, the packing density was $72 \text{ kg}/\text{m}^3$. The total mass of adsorbent for the ACFC system was 36.0 versus 1,175.5 kg for the GAC system. The adsorbent replacement cost for the ACFC system was neglected because most of the need to replace carbon in a GAC system is due to the contamination/degradation related to steam regeneration. In the ACFC system, only the pollutant comes in contact with the adsorbate, and regeneration takes place under an inert N_2 atmosphere. The ACFC system uses 52% of the energy needed for steam regeneration (54.5 versus 104 MJ/h) but the cost of energy for regeneration is higher for the ACFC system due to the difference in the cost of energy for electricity compared to that for natural gas.

There are several factors which merit additional consideration of the ACFC system. For the steam generation cost estimates, a factor of 20% of the fuel cost is included to account for the capital and operating costs in producing the steam. If in-plant steam is not already available, the factor of 20% is likely to be grossly inadequate for a dedicated boiler for small adsorption systems. Also, the ACFC system has no liquid waste stream and conserves

water resources. To capture many commonly used HAPs/VOCs, an ACFC adsorber and electrothermal desorber system is readily scaleable, and is constructed without any moving parts except the valves that control fluid flows. Elimination of the need for equipment such as steam generators, decanters, or inert gas heaters allows an ACFC unit to be readily scalable as a smaller or larger unit and increases its mechanical reliability compared to conventional adsorption/recovery systems. The elimination of any utility requirements except electrical power facilitates operation in commercial, industrial, and other remote types of applications. Recovery of water miscible solvents by a steam-regenerated GAC system requires costly and energy-intensive distillation, making an ACFC/ED system significantly more economical for this application. ACFC/ED technology is ideal for recovering ultrapure compounds because the adsorbent is initially free of contamination and because the regeneration process does not introduce contaminants to the adsorbate. GAC systems are not a practical option for adsorbates that initiate fires within the adsorber, and noncarbonaceous adsorption systems are typically significantly more costly than GAC systems.

This paper documents very encouraging proof-of-concept results, with process efficiency and economics improving with optimization. Lower-cost ACFCs are available and could also significantly improve the economic analysis for ACFC systems. The ACFC/ED technology has strong potential to be the technology of choice for capture and recovery of water-miscible compounds or compounds susceptible to contamination/fires as observed with GAC systems using steam regeneration.

Summary and Conclusions

An automated bench-scale activated carbon fiber cloth adsorber with rapid electrothermal desorption was developed to capture and recover hazardous air pollutants and volatile organic compounds from air streams. ED causes such rapid concentrated release of the adsorbate from the ACFC that 70–80% of the desorbed vapor condenses within the adsorber and drains from the vessel during regeneration, allowing recovery of liquid HAPs/VOCs without the use of auxiliary cooling or steam. This system reduces N_2 consumption by 93% compared to its predecessor, because N_2 is only required to provide an inert atmosphere during ED. Condensation of HAP/VOC occurs at room temperature by passive transfer of heat to the walls of the container, so the need to supply ancillary cooling is eliminated for compounds with relative pressures ≤ 0.1 at 20°C. Proof-of-concept tests verified the capability of the system to adsorb and then desorb these compounds without the use of a cryogenic condenser and provided scale-up data for a pilot-scale unit.

Comparison to the previous system that was similar but did not allow for in-vessel condensation reveals that the heating portion of the regeneration cycle is reduced by 97%, while the cooling step remains unchanged at 20–30 min, yielding an overall decrease in the duration of a regeneration cycle of 67%. The N_2 flow rate during regeneration is reduced to 1% of the total gas flow rate that passes through the adsorber during the adsorption cycle. The new design allows a 500% increase in throughput of the adsorber when treating 1,000 ppmv MEK in air due to a reduction of cycle times. Modification of the system resulted in cost savings of 86% for the cost to capture and recover the MEK compared to the previous system.

Continuous adsorption, desorption, condensation, and recovery of MEK and other select solvents, with concentrations of approximately 1,000 ppmv, has been demonstrated in the labora-

tory in a much simpler system than for conventional (e.g., steam- or heated N_2 -regenerated) adsorption systems. Economic analysis shows the ACFC/ED technology to be cost competitive with conventional GAC systems, while providing a simpler system, reducing energy consumption, and conserving water resources. This ACFC adsorber with ED technology extends the application of carbon adsorption systems to situations that were previously economically and physically impractical.

Acknowledgments

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