

Novel activated carbon fiber cloth filter with functionalized silica nanoparticles for adsorption of toxic industrial chemicals

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Abstract Toxic industrial chemicals (TICs) are used for various civilian and military operations but can be hazardous to humans. There is interest in developing filters that can remove a wide range of airborne TICs (e.g., basic, acidic, organophosphate, and aromatic compounds) to reduce exposure in the work place and from terrorist attacks. A regenerable TIC air filter is particularly desirable because it removes the need for costly filter disposal and replacement. This study evaluates commercially available activated carbon fiber cloth (ACFC) and ACFC that has been modified with functionalized nanoparticles for their abilities to adsorb select TICs and for the regenerability of the adsorbents. The unmodified and modified ACFC samples were tested here for their ability to adsorb anhydrous ammonia (NH₃), hydrogen cyanide (HCN), and dimethyl methylphosphonate (DMMP) in dry and humid gas streams as representative basic, acidic, and organophosphate compounds, respectively. The unmodified ACFC was not an effective adsorbent for NH₃ (i.e., 2 and 3.1 mg NH₃/g ACFC at 1000 ppm_v NH₃ in dry and humid air respectively) or HCN (no detectable adsorption), but successfully adsorbed 840 mg DMMP/g ACFC at 100 ppm_v DMMP. The modified ACFC showed significant improvements for NH₃ and HCN adsorption in dried air (i.e., 19.5 mg NH $_3$ /g ACFC at 1000 ppm_v NH₃ and 4.7 mg HCN/g ACFC at 150 ppm_v HCN, respectively) and in humid air (i.e., 41.4 mg NH₃/g ACFC at 1000 ppm_v NH₃ and 1.6 mg

HCN/g ACFC at 50 ppm_v HCN, respectively) when compared to the unmodified sample. The modified ACFC also showed 2–5 % degradation of its initial dry mass after regeneration and then showed no detectable degradation. The modified ACFC also had similar electrical resistance to that of unmodified ACFC (i.e., within 1 %), indicating that it can be regenerated using electrothermal heating.

Keywords Activated carbon fiber cloth · Modified ACFC · Adsorption capacity · Electrothermal heating · Toxic industrial chemical · TIC · Regenerable air filter · Ammonia · Hydrogen cyanide · Dimethyl methylphosphonate

1 Introduction

Inhalation of airborne toxic industrial chemicals (TICs) can cause severe health effects such as lung or blood damage (OSHA 2013). There is an expanding need for the capture of TICs due to the potential risk of human exposure from an accidental or intentional release of these compounds. For example, during the mid-1990s sarin gas, an organophosphate, was intentionally released in both Tokyo and Matsumoto, Japan killing 21 people and hospitalizing hundreds of people (Masuda et al. 1995; Morita et al. 1995; Wikipedia 2013a, b). Due to the uncertainty of intentional releases of TICs, it is valuable to have a device that can effectively capture a wide range of TICs such as basic, acidic, and organophosphate compounds. Anhydrous ammonia (NH₃), hydrogen cyanide (HCN), and dimethyl methylphosphonate (DMMP) are representative basic, acidic, and organophosphate compounds, respectively. NH₃ is a choking agent that is volatile and colorless with a boiling point of -33.4 °C (Fingas 2002; Cotte-Rodriguez



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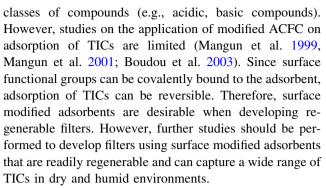
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et al. 2006) and water solubility of 531 g/l at 20 °C (Montgomery 2000). HCN is a chemical warfare agent that is categorized as a blood agent and is a volatile colorless compound, completely soluble in water (Romano et al. 2008), with a boiling point of 25.7 °C (Corn 2012). DMMP is a surrogate for anticholinesterase agents (nerve agents), such as tabun, sarin, and soman (Singer et al. 2005) and is a colorless liquid with a boiling point of 182 °C (Ellison 2008) and water solubility of 100 g/l at 21 °C (Chemical Book 2014).

Activated carbon fiber cloth (ACFC) has been shown to be a promising material for the removal of gaseous emissions (e.g., aromatic TICs such as benzene, toluene, and xylene) (Yao et al. 2009) and to be applicable in various scales and operating conditions (Ramirez et al. 2011; Le Cloirec 2012). ACFC has a large specific surface area $(800-2000 \text{ m}^2/\text{g})$ (Lo 2002) that is hydrophobic (Cal et al. 1997), which can be made more hydrophobic with hydrogen surface treatment (Sullivan et al. 2007). ACFC consists of 4-14 µm diameter woven fibers and is highly porous (0.35–0.95 cm³/g) with an average pore width of 0.6-1.5 nm (Lo 2002). The narrow pore-size distribution and large micropore volume, surface area, and micro-porosity of ACFC make it a desirable adsorbent for gas adsorption. Additionally, ACFC's high purity prevents unwanted chemical reactions that cause fires as experienced with granular activated carbon (GAC) (Zerbonia et al. 2001). The electrical properties of ACFC make it readily regenerable using electrothermal desorption (Petkovska et al. 1991). Furthermore, ACFC can be used upstream of a secondary air pollution control device such as a biofilter or a thermal oxidizer to control the concentration of the gas fed to the secondary device. The ACFC system can be operated based on user-defined constant or dynamic set-points to enhance the overall control efficiency (Emamipour et al. 2007; Hashisho et al. 2008).

Unmodified carbon-based adsorbents like GAC and ACFC cannot strongly adsorb all TICs. The ability of activated carbons to adsorb specific pollutants has been improved by modifying its pH, polarity, and reactivity using impregnation and/or surface treatment (Bansal and Goyal 2005). Impregnation is a process that involves treatment of activated carbon with a chemical reagent that can react with specific contaminants and thereby improve adsorption capacity for target pollutants such as HCN (Alder et al. 1988). However if the impregnants are consumed during the adsorption process, the adsorbent cannot be regenerated to its initial capacity with conventional thermal or pressure swing regeneration techniques. Chemical surface modification is a process in which chemical surface functionalities are added to carbon in the carbonization/activation process or by post-activation processing at high-temperature, in order to enhance its affinity toward specific



The intent of this research is to perform a comparative screening of the adsorption capacities of ACFC and ACFC modified with silica nanoparticles. It will serve as the impetus for further studies on a hybrid regenerable filter with unmodified and modified ACFC that has high removal efficiency for select TICs. The sample adsorption capacities are presented here for basic, acidic, and organophosphate compounds. The samples were tested in dry conditions and at 50 % relative humidity (RH) to simulate indoor conditions. Additionally, the electrical properties and regenerability of the samples are characterized. These results are important because they provide guidance for the development of new regenerable hybrid filters for the capture of TICs from gas streams.

2 Experimental

2.1 Filter

A mutlilayer ACFC filter was designed to adsorb a wide range of TICs using ACFC with surface area of 1600 m²/g (Kuraray Chemical, CH-900). The filter included alternating layers of unmodified and modified ACFC, which were each independently characterized. The modified ACFC layer was coated with functionalized silica nanoparticles. The modification was performed in a two-step process. For the initial step, a 15.2 cm \times 15.2 cm (6 in \times 6 in) activated carbon cloth was dipped in acid solution. Subsequently, the fabric was washed with deionized water and dried in an oven at 120 °C. The dried sample was immersed in 25 wt% tetraethoxysilane (TEOS)/75 wt% ethanol solution and dried in oven at 150 °C for 24 h. The precursor forms a porous silica network by a hydrolysis and condensation. This treatment increases the concentration of hydroxyl groups present on the surface of the activated carbon fiber.

2.2 Experimental apparatus

The experimental apparatus that was used for adsorption experiments consisted of a gas/vapor generation system and a gravimetric balance (Fig. 1). The gas/vapor generation



system consisted of a gas drier/purifier used to purify the carrier gas stream, certified compressed gas cylinders to provide gas streams containing NH₃ or HCN in N₂, a syringe pump (KD Scientific, Model 200) used to supply DMMP at a constant rate, mass flow controllers (Tylan, FC-280) to control the gas flow rates, and a membrane humidifier (Perma Pure, MD-110-12P) to control the RH of the gas stream. For NH₃ and HCN, the calibrated standard gas (i.e., specified concentration ± 5 % by volume) was diluted with controlled RH ultra-high-purity (UHP) N₂ to obtain the desired gas stream composition before entering the balance. DMMP generation involved injecting liquid DMMP with a syringe pump at a constant rate into a controlled RH UHP N₂ gas stream to obtain the desired concentration before entering the balance. Mass flow controllers were calibrated with a gas flow meter (Bios DryCal, DC-2). The experimental temperatures were controlled by adjusting the electrical power that was applied to an electrical resistance heater that was wrapped around the sample chamber, with a variable alternating current (AC) transformer (Variac). The temperature of the sample was measured with a thermocouple (Omega, Type K). Adsorbed mass was determined with an electronic gravimetric balance (Cahn Inc., Model C-2000) with a 0.1 µg detection limit.

2.3 Experimental procedure

2.3.1 Adsorption capacity measurement

The adsorption capacities of the samples were determined gravimetrically. Before each test, the gravimetric balance was zeroed and calibrated with a 10 mg standard mass. The initial mass of the adsorbent sample was then measured

Fig. 1 Schematic of adsorption system experimental apparatus

electrodes (Fig. 2b). The vessel was purged with 2 l/min N₂ for 30 min while voltage (Fig. 2c) was applied across the Exhaust Gravimetric balance . Sample MFC UHP-N2 **MFC** Purifier Humidifier Mixture Syringe Pump

MFC: Mass Flow Controller TIC: Toxic Industrial Chemical

MFC

UHP: Ultra High Purity

while 0.3-0.5 1/min of dry UHP N₂ passed through the sample chamber. The sample was heated to 70-80 °C until the mass was stable (± 0.05 mg) indicating the removal of volatile adsorbed material. The sample was then cooled to ambient temperature (20-24 °C) and the mass of the adsorbent was recorded. A humidified flow rate of 0.3-0.5 1/min UHP N₂ was then passed through the sample chamber and the increase in the sample mass due to water vapor (H₂O) adsorption was recorded after the mass was stable $(\pm 0.01 \text{ mg})$ for 20 min. Then, 0.3–0.5 l/min gas stream with a select TIC concentration at the same RH was provided to the gravimetric balance. The increase in the adsorbent mass was recorded after the mass was stable $(\pm 0.01 \text{ mg})$ for 20 min.

2.3.2 Resistivity measurement

The resistivity values of the modified and unmodified ACFC samples were determined, because this property affects regenerability of the samples when using electrothermal heating. Both, modified ACFC and unmodified ACFC samples, were cut into 4 cm × 8 cm rectangular strips. The initial mass of each strip was recorded, and then the mass was recorded after oven heating at 110 °C for >8 h. Areal density was calculated based on the mass that was measured after heating.

Adsorbent resistivity was determined for each sample based on resistance measurements of the rectangular samples (4 cm × 8 cm) of adsorbent in a separate 1.85 1 interior volume Pyrex vessel (Fig. 2). Each rectangle (Fig. 2a) was supported across its width by stainless steel

Coil

 \bigcirc



Thermocouple

and heater

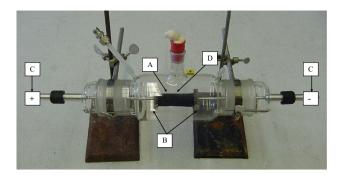


Fig. 2 Apparatus for electrical resistance measurements of ACFC rectangles

ACFC with a direct current (DC) power supply (Tenma, Model 72-2085) to maintain the ACFC at 150 $^{\circ}$ C to desorb volatile adsorbates (e.g., H₂O) from the ACFC. Temperature of the ACFC was measured with a Type K thermocouple (Fig. 2d, 1.6 mm diameter, Omega Inc.) that was attached to the ACFC at the center of the rectangle.

The ACFC was then heated from 50 to 150 °C in 20 °C increments. Current and voltage were measured concomitantly with a multimeter (Fluke, Model 45). Current and voltage were used to calculate resistance and then resistivity ($\rho(T)$, (Ω -m)) was calculated based on the relationship between resistance, ACFC geometry, and temperature (Sullivan et al. 2001).

$$\rho = R \frac{CSA_{Eff}}{L}, \tag{1}$$

where R = resistance (Ω), L = length of ACFC (m) parallel to current flow, and CSA_{Eff} = effective cross sectional area perpendicular to flow of electric current (m², Eq. 2).

$$CSA_{Eff} = \frac{D_A}{D_f}W, \qquad (2)$$

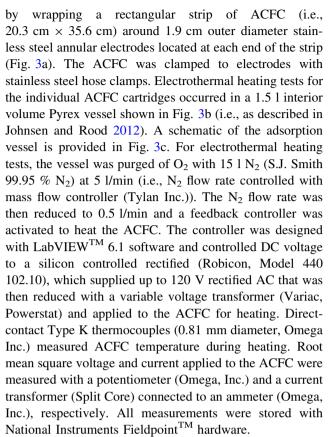
where D_A = areal density of ACFC (g/m²), D_f = density of graphitic carbon (Hayes and Joseph 1981) (g/m³) = 1500 kg/m³, and W = width of ACFC (m) perpendicular to flow of electric current. A linear least square regression between resistivity and temperature was used to determine resistivity dependence on temperature.

$$\rho(T) = \rho_{R}[1 + \alpha(T - T_{R})], \tag{3}$$

where ρ_R resistivity at the reference temperature, $\alpha=$ thermal coefficient (°C⁻¹), which is the change in resistivity for a 1 °C change in temperature, T= temperature (°C), and $T_R=$ reference temperature (0 °C).

2.3.3 Electrothermal heating

Annular ACFC cartridges (i.e., modified and unmodified ACFC) were constructed for electrothermal heating tests



Four heating cycles were performed on each ACFC cartridge that involved electrothermally heating to 150 °C and then maintaining this temperature for at least 5 min to allow the cartridge temperature to stabilize before allowing the cartridge to cool to ambient temperature (25 °C). The temperature controller applied a select voltage (15 V AC with electrical current varying based on electrical resistance, which decreased with increasing temperature) until the ACFC reached 140 °C and then reduced the voltage to stabilize the ACFC at 150 °C. Hence steady-state values of the ACFC's resistance were determined at 150 °C and all transient values (i.e., total energy and time required to heat ACFC to 150 °C) were determined from the start of the heating cycle until the temperature was within 10 % of the set-point of 140 °C.

3 Results

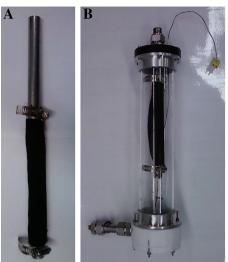
3.1 TIC adsorption

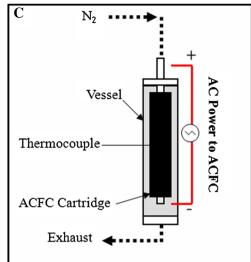
3.1.1 NH₃ adsorption and desorption

The unmodified and modified ACFC were tested for H₂O adsorption and 1000 ppm_v NH₃ adsorption at RH values of <1 and 50 % (Fig. 4). The modified ACFC sample has an order of magnitude higher adsorption capacity of NH₃ and



Fig. 3 Photograph of ACFC cartridge made with modified ACFC **a** and photograph **b** and schematic **c** of ACFC vessel





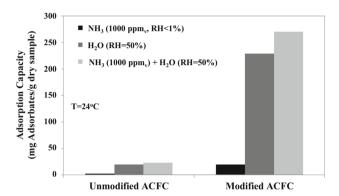


Fig. 4 Adsorption capacity of unmodified and modified ACFC for $NH_3,\,H_2O$ and then NH_3 plus H_2O

 H_2O when compared to unmodified ACFC. The increase in the adsorption capacity is attributed to formation of an adduct between NH_3 and the silica functional group (Blomfield and Little 1973).

Cyclic H_2O (RH = 50 %) then NH₃ plus H_2O $(1000 \text{ ppm}_{v} \text{ at } \text{RH} = 50 \%) \text{ adsorption/desorption tests}$ occurred to determine the additional mass gain due to NH₃ adsorption after adsorption of H₂O (Figs. 5, 6). The initial dry mass of the modified sample decreased by 2 % after three adsorption/desorption cycles and then no detectable degradation was observed after additional adsorption/desorption cycles. The H₂O adsorption capacity increased by 16 % after three adsorption/desorption cycles and then remained stable while NH₃ plus H₂O adsorption capacity increased by 5 % and then remained stable. The change in adsorption capacity over the first three adsorption cycles is attributed to the reaction of NH3 with ACFCs functional groups, and loss of sorbent from electrothermal heating. The steady-state adsorption capacity for NH₃ (total adsorption capacity minus H₂O adsorption capacity) onto the

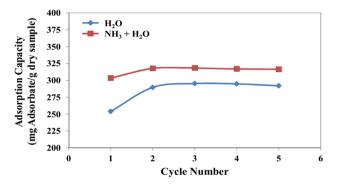


Fig. 5 Adsorption capacity of $\rm H_2O~(RH=50~\%)$ and $\rm H_2O~plus~NH_3$ (RH = 50 %, 1000 ppm $_{\rm v}$ NH $_{\rm 3}$) for modified ACFC during five adsorption cycles

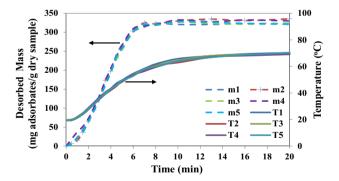


Fig. 6 Five regeneration cycles for modified ACFC inside the gravimetric balance. m_x and T_x describe desorbed mass and temperature for xth regeneration cycle, respectively

modified ACFC is larger than that of the unmodified ACFC by a factor of six demonstrating an improved adsorbent for NH₃. With regards to ACFC regenerability, the time required for the sample to reach its final dry mass (within 2 %) was the same for all five desorption cycles indicating



consistent regeneration behavior after each adsorption cycles (Fig. 6). These results are encouraging and confirm potential use of functionalized silica nanoparticles to enhance the adsorption capacity of ACFC for basic compounds and demonstrate regenerability of the modified ACFC.

3.1.2 HCN adsorption

Adsorption of HCN by unmodified ACFC indicated no detectable adsorption (<0.01 mg/g sample) of HCN at HCN concentrations of 150 ppm_v at dry conditions, while the modified ACFC adsorbed 4.7 mg HCN/g ACFC. The modified ACFC was also tested at lower HCN concentrations of 50 and 100 ppm_v (Fig. 7) to further characterize its ability to adsorb HCN. There is a strong linear correlation $(R^2 = 0.99)$ between HCN adsorption capacity onto the modified ACFC and HCN concentration for the conditions tested. Interestingly, extrapolation of these data to 1000 ppm_v HCN in dry N₂ results in an HCN adsorption capacity of 27 mg HCN/g ACFC, which is the same order of magnitude as the NH₃ adsorption capacity (19.5 mg NH₃/g ACFC at 1000 ppm_v). The enhancement of adsorption capacity of modified ACFC compared to the unmodified ACFC is attributed to hydrogen bond formation between HCN and the proton present on the hydroxyl groups of the modified ACFC (Rasko et al. 2002).

HCN adsorption capacity was also determined at RH = 50 % by performing an H₂O adsorption cycle at RH = 50 % followed by an adsorption cycle of a gas stream containing 50 ppm_v HCN and H₂O (RH = 50 %) at $20 \,^{\circ}$ C. The HCN adsorption capacity (total adsorption capacity minus H₂O adsorption capacity) at RH = 50 % was 1.6 mg HCN/g ACFC, which is 20 % lower than HCN adsorption capacity of 2 mg HCN/g ACFC at dry conditions. This is attributed to competitive adsorption of HCN and H₂O in presence of hydrophilic groups on the modified ACFC. Thus, even at RH = 50 %, the modified ACFC

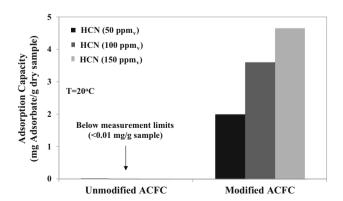


Fig. 7 HCN Adsorption capacity for unmodified and modified ACFC

adsorbs HCN (1.6 mg/g) while there was no detectable HCN adsorption with the unmodified ACFC demonstrating that the modified ACFC is an improved adsorbent for HCN.

3.1.3 DMMP adsorption

100 ppm_v DMMP was adsorbed onto unmodified and modified ACFC at 20 °C and 50 % RH (Fig. 8). The unmodified ACFC has an adsorption capacity (total adsorption capacity minus H₂O adsorption capacity) of 824 mg DMMP/g ACFC. The modified ACFC had an adsorption capacity (total adsorption capacity minus H₂O adsorption capacity) of 219 mg DMMP/g ACFC, which is 27 % of the adsorption capacity for the unmodified sample. The method to calculate the adsorption capacity (total adsorption capacity minus H₂O adsorption capacity) is not necessarily accurate due to possible interactions between the TICs and H₂O during multicomponent adsorption. However, this difference in adsorption capacity might be attributed to DMMP being less hydrophilic compared to NH₃ and HCN, as higher concentrations of hydrophilic groups are present on modified ACFC resulting in more adsorbed H₂O (Fig. 4). Thus, the unmodified layer of the mutli-layer filter is expected to contribute more towards DMMP adsorption.

3.2 Electrothermal regeneration of filter

Unmodified and modified ACFC were electrothermally heated to compare their regeneration heating requirements. Table 1 shows the mass, areal density and resistivity parameters of each ACFC sample. The areal density of the modified sample was 44 % larger than that of the unmodified sample. This increase in areal density is attributed to silica nanoparticles that were deposited on the surface of ACFC. The resistivity of the modified sample was 92 % higher than that of the unmodified sample. The electrical resistance of the ACFC cartridges at 150 °C and the energy

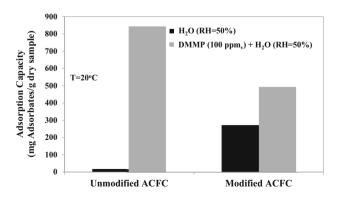


Fig. 8 Adsorption capacity of unmodified and modified ACFC for $100~\text{ppm}_{\scriptscriptstyle V}$ DMMP



Table 1 Mass, areal density, and resistivity parameters of 4 cm × 8 cm rectangular strips of unmodified and modified ACFC^a

Sample	Mass	Areal density (g/m ²)	$\rho_R \; (\Omega\text{-m})$	α (°C ⁻¹)
Unmodified ACFC	0.47 ± 0.02	148 ± 5	$1.48 \times 10^{-3} \pm 0.21 \times 10^{-3}$	$-2.83 \times 10^{-3} \pm 0.41 \times 10^{-3}$
Modified ACFC	0.68 ± 0.02	213 ± 6	$2.64 \times 10^{-3} \pm 0.01 \times 10^{-3}$	$-2.53 \times 10^{-3} \pm 0.12 \times 10^{-3}$

^a Average ± standard deviation

Table 2 Electrothermal heating tests with ACFC cartridges^a

Sample	Energy for heating to 140 °C (kJ/g ACFC)	Resistance at 150 °C (Ω)
Unmodified ACFC	0.61 ± 0.02	4.20 ± 0.04
Modified ACFC	0.43 ± 0.01	4.16 ± 0.09

^a Average ± standard deviation

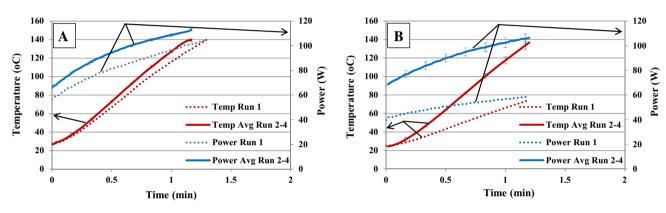


Fig. 9 Temperature and power profiles during electrothermal heating of unmodified ACFC (a) and modified ACFC (b)

required for heating the ACFC cartridges to 140 °C are described in Table 2. Temperature and power profiles for electrothermal heating of the ACFC cartridges are shown in Fig. 9. Vertical bars on the curves for run 2–4 indicate standard deviations.

The first heating cycle required more energy than the following cycles for each sample due to the additional energy required to heat and desorb previously adsorbed material (e.g., water). This also resulted in longer electrothermal heating times. The resistance of the ACFC also changes with adsorbed water and that has impact on heating profile. The modified ACFC was more hydrophilic and had more adsorbed water than the unmodified ACFC so the differences in the energy and heating time that are described by the temperature profiles, between having initially adsorbed water and being freshly regenerated were more defined. After the first heating cycle, water was desorbed from the sample and hence power and temperature profiles look similar for unmodified and modified samples for run 2–4.

The modified ACFC consumed a similar total energy for heating (kJ) (within 2 %) and had similar steady-state

resistance values (Ω) (within 1 %) to unmodified ACFC, despite the larger total mass of the modified ACFC. This resulted in a smaller normalized energy consumption (kJ/g ACFC). Based on these results the unmodified and modified ACFC samples performed similarly (i.e., same order of magnitude for total heating energy and resistance) during the electrothermal regeneration tests demonstrating it is feasible to regenerate the filter with resistive heating.

4 Summary and conclusions

Commercially available ACFC was modified with silica nanoparticles to increase its adsorption capacity for common TICs. The adsorption capacity (total adsorption capacity (NH $_3$ plus H $_2$ O) minus H $_2$ O adsorption capacity) for NH $_3$ increased by a factor of 13 for modified ACFC compared to unmodified ACFC at 50 % RH. Further, modified ACFC adsorbed 4.7 mg HCN/g ACFC at 150 ppm $_v$ while unmodified ACFC did not show detectable adsorption for HCN. The adsorption capacity (total adsorption capacity (DMMP plus H $_2$ O) minus H $_2$ O adsorption capacity) of



modified ACFC for DMMP was 73 % smaller when compared to unmodified ACFC at 50 % RH.

The modified ACFC was also tested for electrothermal regeneration capability by performing five NH₃ adsorption/ desorption cycles. The adsorption capacity of modified ACFC stabilized after the third cycle. The modified ACFC also has electrical properties, which are similar (i.e., electrical resistance values at 150 °C within 1 %) to unmodified ACFC, indicating that it can be heated to achieve electrothermal regeneration. The modified ACFC shows potential to be used in conjunction with unmodified ACFC in a hybrid multi-layered regenerable filter that can capture basic, acidic, and organophosphate TICs such as NH₃, HCN, and DMMP (on modified/unmodified ACFC) and aromatic TICs such as benzene, toluene, and xylene (on unmodified ACFC as previously published). Therefore, the overall filter shows promise to adsorb a wide range of toxic compounds. Additionally, the unmodified and modified ACFC samples can be regenerated with electrothermal heating, suggesting the proposed filter can be regenerated, reducing maintenance costs for filter replacement.

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