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Breakthrough Curves for Toluene Adsorption on Different Types of Activated Carbon Fibers: Application in Respiratory Protection

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ABSTRACT

Activated carbon fibers (ACF) are considered viable alternative adsorbent materials in respirators because of their larger surface area, lighter weight, and fabric form. The purpose of this study was to characterize the breakthrough curves of toluene for different types of commercially available ACFs to understand their potential service lives in respirators. Two forms of ACF, cloth (AC) and felt (AF), with three surface areas each were tested. ACFs were challenged with six toluene concentrations (50–500 p.p.m.) at constant air temperature (23°C), relative humidity (50%), and air flow (16 l min^{-1}) at different bed depths. Breakthrough data were obtained using continuous monitoring by gas chromatography using a gas sampling valve. The ACF specific surface areas were measured by an automatic physisorption analyzer. Results showed unique shapes of breakthrough curves for each ACF form: AC demonstrated a gradual increase in breakthrough concentration, whereas AF showed abrupt increase in concentration from the breakpoint, which was attributed to the difference in fiber density between the forms. AF has steeper breakthrough curves compared with AC with similar specific surface area. AC exhibits higher 10% breakthrough times for a given bed depth due to higher mass per bed depth compared with AF, indicating more adsorption per bed depth with AC. ACF in respirators may be appropriate for use as protection in environments with toluene concentration at the Occupational Safety and Health Administration Permissible Exposure Limit, or during emergency escape for higher toluene concentrations. ACF has shown great potential for application in respiratory protection against toluene and in the development of thinner, lighter, and more efficient respirators.

KEYWORDS: activated carbon fiber; adsorption; breakthrough; respirators; respiratory protection; service life; toluene

INTRODUCTION

Granular activated carbon (GAC) is currently the standard adsorbent used in respiratory protection against volatile organic compounds (VOCs) and other gas phase contaminants. However, GAC has certain drawbacks, the primary being need for containment and attrition of the granular material (Feng *et al.*, 2005). Because of its granular nature, GAC needs to be contained in a cartridge or canister, which adds to the weight and bulkiness of the respirator. This tends to make respirators less comfortable to wear, which can result in poorer compliance in their use. Thus, there is a need for the development of more comfortable respirators to increase user compliance and improve worker protection against airborne contaminants.

Activated carbon fibers (ACF) have shown potential as alternative adsorbents for controlling VOCs by overcoming some of the drawbacks of GAC. ACFs are obtained from the carbonization and activation of polymeric fibers that can be prepared from novoloid, polyacrylonitrile, pitch and Rayon precursors, with diameters ranging from 10 to 20 μm (Petkovska *et al.*, 1991). This small fiber diameter allows homogeneous activation of the fibers, thus yielding an adsorbent material with a narrow pore size distribution in the micropore range (Feng *et al.*, 2005). ACFs have several advantages over traditional activated carbon adsorbents such as GAC, including their larger surface areas, higher adsorption capacities and rates, greater number of micropores, and faster thermal and mass transfer properties (Petkovska *et al.*, 1991; Tsai *et al.*, 2008; Balanay and Lungu, 2012). A comparison of the toluene adsorption between GAC and ACF types showed ACFs with BET (Brunauer, Emmett, and Teller) surface areas of 1500 $m^2\ g^{-1}$ to have higher adsorption capacities than GAC with a BET surface area of 1800 $m^2\ g^{-1}$ (Balanay *et al.*, 2011). ACF is also easier to handle than GAC as it can be manufactured in various forms, such as cloth and felt (Huang *et al.*, 2002). Among the various types of ACF, cloth forms were shown to have higher adsorption capacities and lower critical bed depths compared with the felt forms with similar specific surface areas (Balanay *et al.*, 2014).

Given its advantages, ACF is a good alternative adsorbent to consider in the development of thinner, lighter, and more efficient respirators because of their larger surface area, greater adsorption capacities, lighter weight, and convenient form factor (cloth and felt). Kudryavtseva *et al.* (1998) recognized the potential of ACFs in the development of comfortable, light-duty gas respirators and investigated the optimum properties of ACF for such application. Recent studies have investigated the possible application of various ACF types for respiratory protection against VOCs and concluded that ACFs have great potential for such application based on adsorption capacity and critical bed depth (Balanay *et al.*, 2011; Balanay *et al.*, 2014). Moreover, light N95-like respirators made of ACFs that are compact, easier to wear, and potentially regenerable (e.g. through thermal treatment) may be developed in the future. Such device of reduced size, complexity, and cost may be extremely useful as an escape mask for workers or the public, in case of accidental or intentional release of toxic gases and vapors (Balanay *et al.*, 2011). To provide adequate protection against high concentration exposures or longer term low-level exposures at or near the occupational exposure limit, adsorption characteristics of different types of commercially available ACFs must be understood across a wide range of concentrations for specific chemicals to determine potential levels of protection afforded when used in respiratory protection.

Toluene is one of the major indoor organic vapors and one of the most common VOCs in the workplace (Girman *et al.*, 1999; Larroque *et al.*, 2006). Several studies that tested ACF materials used toluene as a representative for the VOC group (Brasquet and Le Cloirec, 1997; Cheng *et al.*, 2004; Das *et al.*, 2004; Fournel *et al.*, 2005; Lorimier *et al.*, 2005). Additionally, toluene is important to characterize because, at exposures ranging from 200 to 1100 p.p.m., the adverse effects of toluene on humans range from eye, skin, and mucous membrane irritation, to drowsiness, nausea, and memory loss, to damaged liver and kidney, unconsciousness, or even death (Agency for Toxic Substances and Disease Registry, 2000). As its exposure limits, toluene has Occupational Safety and Health Administration (OSHA) Permissible Exposure Limit - Time-Weighted Average (PEL-TWA) of 200 p.p.m., Permissible Exposure Limit - Ceiling (PEL-C) of 300 p.p.m., PEL-Peak of 500 p.p.m., American Conference of Governmental Industrial Hygienists Threshold Limit Value (ACGIH TLV) of 20 p.p.m., and United Kingdom Workplace Exposure Limit (UK WEL) (8-h TWA) of 50 p.p.m. (Health and Safety Executive, 2011; OSHA, 2014). Toluene has lower adsorption capacity onto activated carbons compared with several VOCs, such as benzene, xylene, trichloroethylene, and tetrachloroethane (Hung *et al.*, 2007; Ramos *et al.*, 2010; Li *et al.*, 2012).

In a previous study, various types of ACFs were tested to determine their adsorption capacities and critical bed depths for toluene (Balanay *et al.*, 2014). These adsorption characteristics were derived from the ACFs' breakthrough characteristics. The purpose of this paper is to use this breakthrough data to obtain service life estimates of various commercially available ACF materials if used in respirators against toluene vapor.

METHODS

Materials

Two forms of ACF were used as adsorbents: woven cloth (AC) and non-woven felt (AF). For each form, three different manufacture-specified surface areas (1000 and 1500 m^2 g^{-1} for both AC and AF, 1800 m^2 g^{-1} AF, and 2000 m^2 g^{-1} AC) were tested for a total of six types of ACFs. ACF types were labeled based on their form and nominal surface area (Table 1).

AC10, AC15, AC20, and AF15 were manufactured from novoloid, a phenol aldehyde-based fiber, by American Kynol, Inc. (Pleasantville, NY, USA), whereas AF10 and AF18 were manufactured from viscose rayon fibers by Beijing Evergrow Resources Co. (Beijing, China). The ACFs were cut into 4-cm discs and thermally desorbed in an oven (Thermo Electron Corporation, Waltham, MA, USA) at 200°C overnight prior to testing to remove volatile impurities and moisture. Laboratory-grade toluene (Fisher Scientific, Waltham, MA, USA) was used without further purification as the adsorbate.

Characterization of ACF structural properties

Structural properties of these ACF materials have been described previously (Balanay *et al.*, 2014). The specific surface area and pore characteristics of the ACF samples were measured by nitrogen adsorption at 77 K using a Micromeritics ASAP 2020 automatic physisorption analyzer (Micromeritics Corp., Norcross, Georgia). Fiber organization of the ACF samples was examined by scanning electron microscopy (SEM) at ×203 magnification.

Table 1. Denotations of ACF types.

Form	Nominal[a] surface area, (m^2 g^{-1})			
	1000	1500	1800	2000
Cloth	AC10	AC15	—	AC20
Felt	AF10	AF15	AF18	—

[a]Manufacturer-specified.

Breakthrough determination

Breakthrough determination has been described previously (Balanay *et al.*, 2014). ACF materials were challenged with toluene in a stainless steel chamber, with an internal diameter of 4 cm, at constant airflow (16 l min^{-1}), temperature (23 ± 0.5°C), and relative humidity (RH; 50 ± 10%) at varied bed depths. Breakthrough curves of toluene were obtained at 50, 100, 200, 300, 400, and 500 p.p.m. for each ACF type.

Toluene vapor was generated by continuous injection of liquid toluene into a pre-conditioned air stream using an Aladdin-1000 automated syringe pump (World Precision Instruments, Sarasota, FL, USA). Dry air was supplied to a Miller-Nelson Model HCS-401 instrument (Assay Technology, Inc., Livermore, CA, USA), which controlled the flow, temperature, and RH as specified previously. Breakthrough of toluene was determined by continuous monitoring of the effluent gas with a gas chromatograph (Agilent Model 6850, Agilent Technologies, Alpharette, GA, USA) equipped with a gas sampling loop and flame ionization detector. A 5-point calibration curve was established using the syringe pump to generate five known concentrations. The gas chromatograph sampling valve was used to sample the known concentration several times at each calibration point in the same manner as breakthrough experiments. This experimental set-up for breakthrough determination has been used in previous studies (Balanay *et al.*, 2011, 2014).

Breakthrough curves of toluene were obtained for each ACF type at different adsorbent bed depth (i.e. discrete layers of ACF) per toluene concentration. The 10% breakthrough time or breakpoint (i.e. time, in minutes, at which the toluene concentration in the effluent rises to $C/C_0 = 0.1$, wherein C = effluent or exit concentration and C_0 = inlet or initial challenge concentration) was used to make estimates of service life for respiratory protection.

RESULTS AND DISCUSSION

ACF structural properties

The structural properties of each ACF type were summarized in Table 2, including weave type, thickness, density, and mass per depth. The non-woven AF types are shown to be thicker than the woven AC types and were less dense (0.05–0.06 g cm^{-3}) than the AC types (0.22–0.34 g cm^{-3}). Other characteristics of the ACF materials (i.e. BET surface area and microporosity) were also measured previously, showing that the specific surface area of the AC is similar to its AF counterpart (Balanay *et al.*, 2014).

The SEM images in Fig. 1 show the difference in the inter-fiber structures between the cloth and felt forms, as represented by AC15 and AF15, respectively. The AC is composed of woven yarns of twisted fibers (Fig. 1a), whereas the AF is composed of non-woven, randomly distributed fibers (Fig. 1b). This gives the AC ~5-fold density of the AF.

Breakthrough characteristics by ACF form

The breakthrough curves show unique shapes for each ACF form. Fig. 2 shows sample breakthrough curves representing AC (Fig. 2a) and AF (Fig. 2b) types at 500-p.p.m. toluene and at four different bed depths by number of layers. With AC, the increase in toluene concentration occurs slowly from the breakpoint. Such gradual increase is more prominent as the number of adsorbent layers increase. With AF, the increase in toluene concentration occurs abruptly from the time the breakpoint starts. However, the increase in concentration slows down as 100% breakthrough is approached. This trend becomes more prominent as the challenge concentration decreases.

When comparing AC and AF with similar specific surface areas at the same number of layers, it takes a shorter time for toluene to approach near saturation (i.e. $C/C_0 = 0.90$) from the start of the breakpoint with AF compared with AC, as demonstrated by the steeper slope of the breakthrough curves for AF. The gradual increase in the effluent concentration for AC, particularly at higher number of layers, suggests a larger mass transfer resistance and a layering effect for AC. In contrast, the abrupt increase in the effluent concentration at the breakpoint for AF indicates that the micropores across the bed depth were equally available and fill simultaneously, demonstrating the lack of layering effect for AF. These observations may be attributed to the difference in fiber density between the cloth and felt. As shown in the SEM images, the AC has tightly

Table 2. Structural properties by ACF type.

ACF type	Weave type	Thickness (cm)	Density[a] ($g\ cm^{-3}$)	Mass per bed depth[a] ($g\ cm^{-1}$)
AC10	Woven	0.063	0.34 ± 0.00	4.30 ± 0.05
AC15	Woven	0.063	0.28 ± 0.00	3.46 ± 0.06
AC20	Woven	0.063	0.22 ± 0.01	2.79 ± 0.07
AF10	Non-woven	0.200	0.06 ± 0.00	0.75 ± 0.01
AF15	Non-woven	0.300	0.06 ± 0.00	0.78 ± 0.02
AF18	Non-woven	0.275	0.05 ± 0.00	0.68 ± 0.02

[a]Mass of each ACF type was measured at four different bed depths.

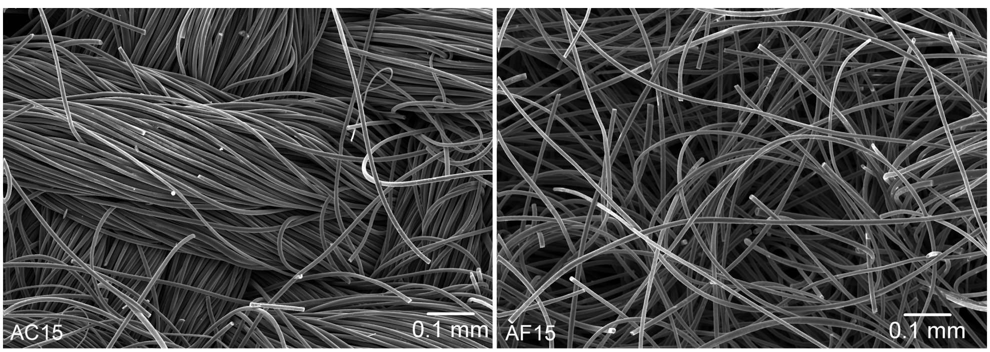

Figure 1 SEM images of (a) AC15 and (b) AF15 at ×203 magnifications.

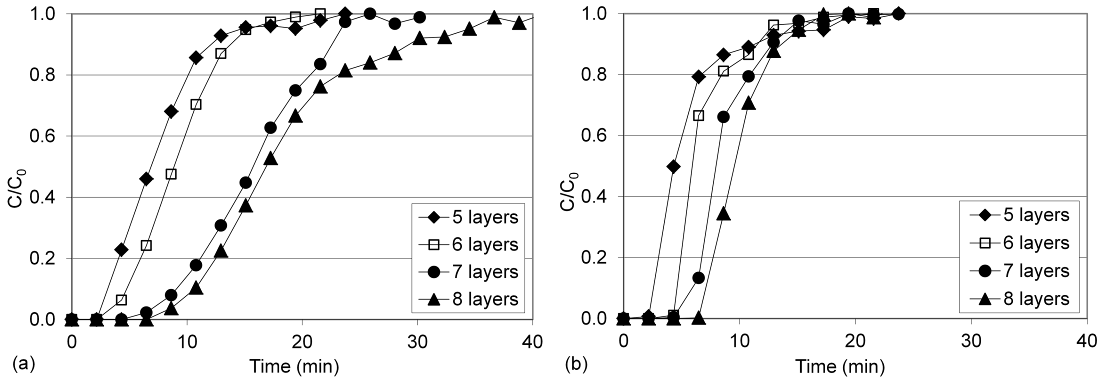

Figure 2 Experimental breakthrough curves for (a) AC10 and (b) AF10 by number of layers at C_0 = 500-p.p.m. toluene.

woven fibers and are denser than the AF, which has loosely packed fibers that are distributed randomly. Because of the AF's lower density and looser packing, each fiber within the adsorbent bed is more equally available for diffusive adsorption of contaminants from the air flow and the adsorbent loads nearly uniformly. Conversely, in AC, the tight, organized weaving of the cloth causes more interaction between the fibers and air flow, allowing each fiber more opportunity to remove contaminants on a single pass. As the adsorbent bed approaches saturation, the downstream layers are less saturated than the upstream layers, and it takes times for the remaining layers to reach full saturation, as demonstrated by the slower increase to near saturation after the breakpoint (Fig. 2).

Effect of toluene concentration on breakthrough characteristics

The effect of toluene concentration on the breakthrough characteristics of AC and AF is shown in Fig. 3 for a fixed bed depth (five layers of ACF). The trends in this data are represented by AC15 and AF15. The breakthrough time of toluene through a fixed number of ACF layers increases as the toluene challenge concentration decreases, as expected. However, such increase in breakthrough time is not linear with the concentration. Greater increase in breakthrough time at the lower concentrations compared with that at higher concentrations was observed with the same proportional change in challenge concentration, as is evidenced by the non-linear trends shown in Fig. 4. The steepness of the breakthrough curves (Fig. 3) increase with toluene concentration, demonstrating decreasing saturation times. The increase in both the breakthrough and saturation times with the decrease in toluene challenge concentration indicates a longer service life for ACF at lower toluene concentrations. The uncharacteristic curve shapes of AF-15, particularly at 50-p.p.m. toluene (Fig. 3b), is not fully understood at this time, but is suspected to be due to the low bulk density of the AF material, given its 'spongier' characteristic compared with the AC type. The AF structure may have allowed toluene molecules to readily penetrate through the AF bed non-uniformly, without fully saturating each layer. At a low concentration, certain regions in the material becomes completely saturated, subsequently allowing the toluene molecules to travel through the regions still holding some unspent capacity. Thus, the breakthrough curve is sharp as penetration starts but becomes elongated as certain regions in the material slowly become saturated.

Using 50% RH as a constant parameter may have resulted to shorter breakthrough times and steeper curves compared with much lower RH (e.g. 10% RH) due to competition for adsorption sites between toluene and water molecules. Such adsorption site competition between molecules may have also contributed to the uncharacteristic breakthrough curves of AF materials. Conversely, toluene adsorption capacities are expected to be higher at 50% RH than if challenged at higher RH (e.g. 90% RH) for the same reasons as stated previously.

10% Breakthrough time

The 10% breakthrough times were compared among ACF types at a given bed depth, with the purpose of

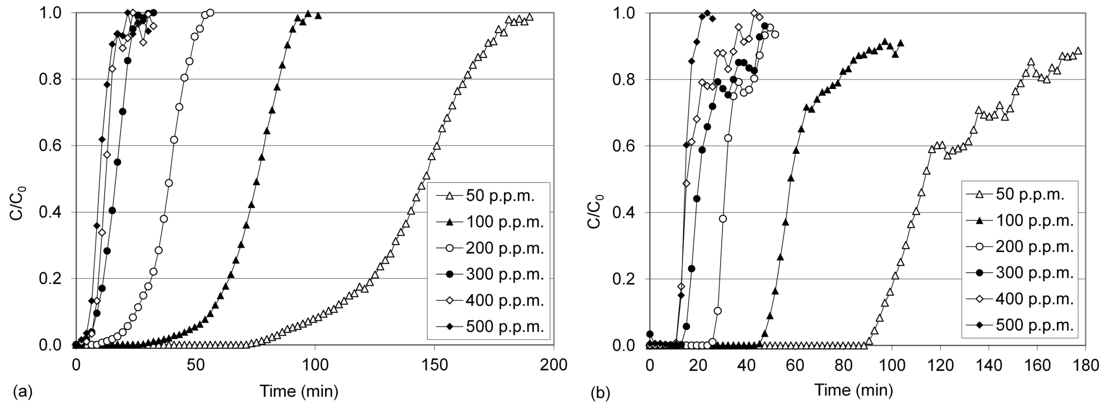

Figure 3 Experimental breakthrough curves for (a) AC15 and (b) AF15 by toluene concentration at number of ACF layers = 5.

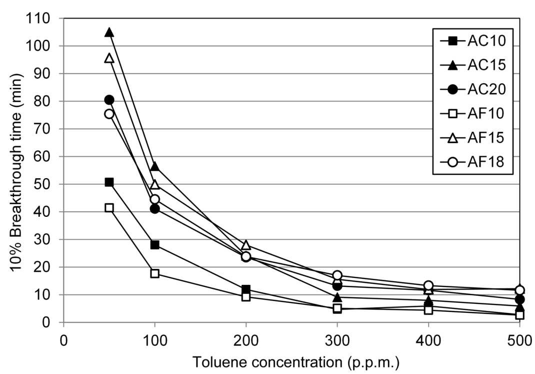

Figure 4 10% Breakthrough time as a function of toluene concentration by ACF type at number of ACF layers = 5.

characterizing the potential respiratory protection against airborne toluene at different concentrations for each ACF type (Fig. 5). Given that the wearer's breathing results in a face velocity similar to that in the test condition (i.e. 0.21 m s^{-1}), at an adsorbent bed depth = 0.5 cm and surface area = 12.6 cm^2 (based on the 4-cm ACF discs), using ACs in a respirator may afford protection to the wearer from 11 to 19 min before 10% breakthrough of 500-p.p.m. toluene occurs and from 134 to 190 min for 50-p.p.m. toluene. AFs in a respirator at an adsorbent bed depth = 1.5 cm and surface area = 12.6 cm^2 may afford protection to the wearer from 3 to 15 min before 10% breakthrough of 500-p.p.m. toluene occurs and from 41 to 96 min for 50-p.p.m. toluene. If the area of the ACF adsorbent is increased to that of a typical small-size N95 respirator (~165 cm^2), such ACF respirator may provide ~13 times longer performance if the adsorbent bed depth and air velocity through the adsorbent are the same as in these tests and if there is uniform adsorption throughout the entire respirator. This would mean that AC may provide 143–247 min of respiratory protection at 500-p.p.m. toluene and 1742–2470 min at 50-p.p.m. toluene; whereas AF may afford protection from 39 to 195 min at 500-p.p.m. toluene and 533–1248 min at 50-p.p.m. toluene. For each concentration, the 10% breakthrough time generally increases as the ACF-specific surface area increases (Fig. 5), which may be attributed to the increasing micropore volume.

Breakthrough times of toluene as a function of bed depth are shown for the various ACF types at two different concentrations (Fig. 6). The bed depth tested for AC ranged from 0.25 to 0.50 cm (4–8 layers) and for AF ranged from 0.83 to 2.10 cm (3–8 layers). Given the same bed depth, AC types exhibit a higher breakthrough time compared with AF, as illustrated with the 50- and 500-p.p.m. toluene trials shown in Fig. 6. This can be attributed to the more dense form of AC than AF, as described previously. Thus, for an equivalent studied bed depth, the mass of AC is higher than the mass of AF, as shown in Table 2. Consequently, as breakthrough time increases with the bed depth, the rate of increase is higher for AC than for AF. The ACF with the lowest specific surface area, either cloth (i.e. AC10) or felt (i.e. AF10) form, had the lowest breakthrough time at a given bed depth for both toluene concentrations.

Figure 7 shows the toluene breakthrough times of ACF types as a function of adsorbent mass at

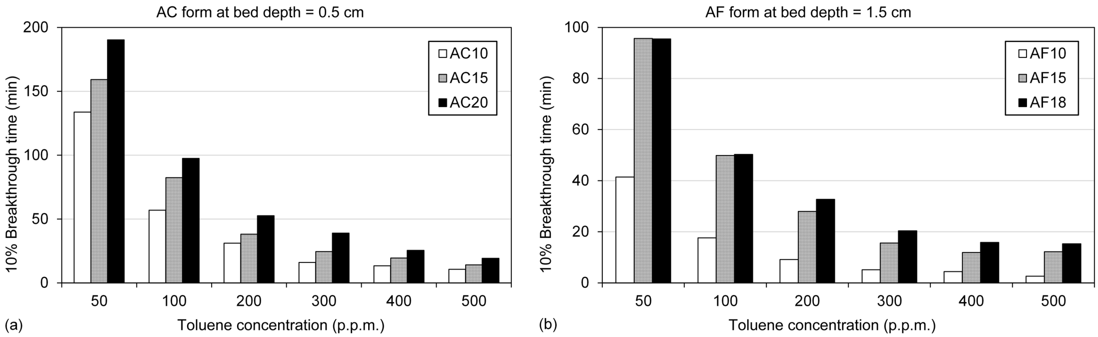

Figure 5 10% Breakthrough time of toluene by ACF type: (a) AC form and (b) AF form.

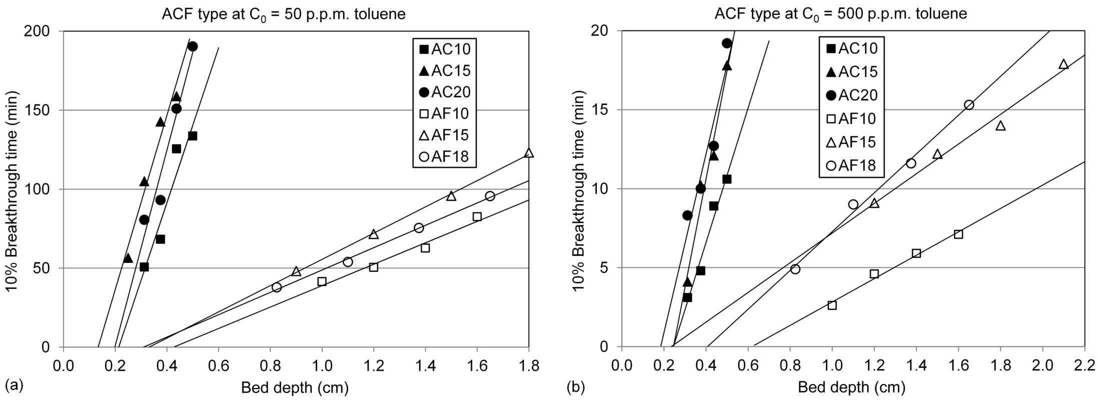

Figure 6 10% Breakthrough time of toluene as a function of bed depth by ACF type at (a) C_0 = 50 p.p.m. and (b) 500 p.p.m.

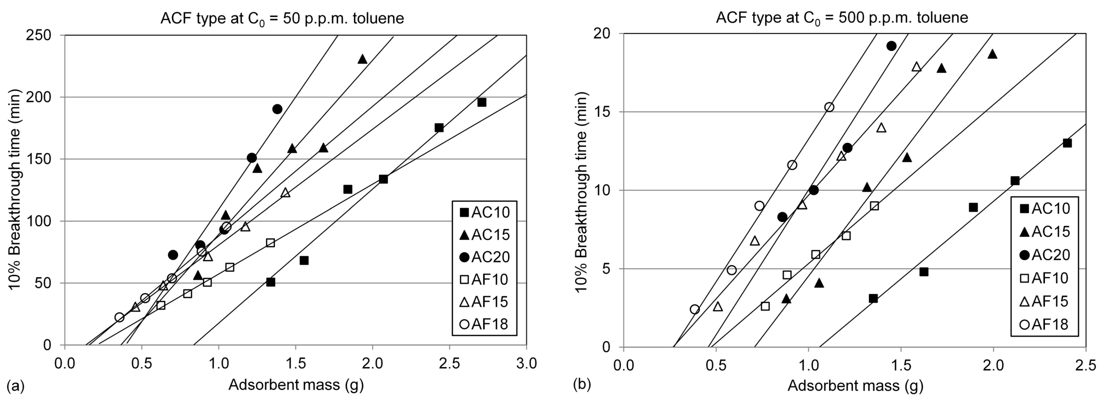

Figure 7 10% Breakthrough time of toluene as a function of mass by ACF type at (a) C_0 = 50 p.p.m. and (b) 500 p.p.m.

two concentrations, which demonstrates varying performance of AC versus AF types. Given the same mass (e.g. 1.5 g), all AF types show higher breakthrough times compared with the AC types at 500-p.p.m. toluene, whereas AC types of higher specific surface area (AC15 and AC20) shows higher breakthrough times compared with their AF counterparts (AF15 and AF18) at 50-p.p.m. toluene. Interestingly, at 50-p.p.m. toluene, AC10 exhibits a higher breakthrough time than AF10 when adsorbent mass reaches 2.1 g. However, bed depth is given more emphasis in this study than adsorbent mass because there are practical limitations to how thick a respirator cartridge or N95-like mask could be without impeding the wearer's vision or comfort.

Application of breakthrough characteristics to respiratory protection

The extent of respiratory protection that the studied ACF types may provide against airborne contaminants may be described based on their breakthrough characteristics. Both breakthrough and saturation times increased as toluene challenge concentration decreased, indicating a longer service life for ACF at lower toluene concentrations. At the UK WEL of 50 p.p.m., an N95-like ACF respirator composed of a 0.5-cm AC may have a service life of at least 28 h, and one composed of a 1.5-cm AF may have a service life of at least 8 h. At OSHA PEL of 200 p.p.m., the AC may provide protection against toluene for at least 6 h, and AF for at least 2 h. Protection afforded by ACF respirators against toluene at higher concentrations will last for a shorter period. At 500 p.p.m., the Immediately Dangerous to Life or Health (IDLH) value for toluene, the same N95-like AC respirator may provide protection for at least 140 min, whereas the AF respirator for at least 30 min, given a face velocity and RH similar to the test condition. Thus, some of these ACF types may be suitable for emergency purposes, such as escaping from an area with high-volume spills resulting in high ambient concentration of toluene. However, in an emergency situation, breathing rates are expected to be higher than normal use conditions due to panic and/ or high levels of physical exertion, which is expected to decrease the protection time during escape scenarios. From a safety standpoint, ACF is indicated to be more suitable for use at lower ambient concentrations in providing longer respiratory protection against toluene. However, increasing the bed depth of these ACF materials in respirators should provide longer protection against toluene.

It is important to note that the service lives identified for the N95-like ACF respirators were based on 10% breakthrough of challenge toluene concentrations. Certain respirator certification assessments for organic vapor service life may have used lower percentage breakthrough, such as 0.5% of 1000-p.p.m. carbon tetrachloride in NIOSH testing (National Personal Protective Technology Laboratory [NPPTL], 2006). Testing the ACF respirators using these certification conditions or criteria may result in shorter service lives, and thus shorter protection times. Moreover, ACF materials were tested in this study using a constant unidirectional airflow, humidity, and temperature. In realistic workplace scenarios, ACF respirators are exposed to bi-directional airflow and humidity fluctuations due to worker breathing and thus, respirator service lives may change. Breakthrough times have been demonstrated to significantly decrease when airflow across activated carbon adsorbents was changed from fixed flow to sinusoidal flow (Suzin *et al.*, 2000; Nir *et al.*, 2002; Linders *et al.*, 2003) The effect of sinusoidal airflow on toluene adsorption onto ACF respirators needs further investigation to determine the extent of decrease in breakthrough times.

CONCLUSIONS

Based on the breakthrough characteristics of toluene, particularly the breakthrough and saturation times, ACF may provide sufficient respiratory protection in environments with lower toluene concentrations (i.e. 50 p.p.m.) for extended periods. Comparing the ACF forms (AC versus AF), AC provides longer protection against toluene than AF at equivalent bed depth based on its longer breakthrough time. This is likely attributable to the denser form of AC compared with the non-woven form of the AF, and therefore greater mass of adsorbent carbon present. Moreover, at a given toluene concentration, the 10% breakthrough time generally increases as the ACF specific surface area increases. This is explained by the increasing microporosity of each ACF form as the specific surface area increases. ACF may also be appropriate for use as short-term respiratory protection in performing quick tasks or during emergency escape for higher toluene concentrations. ACF, particularly AC with the highest specific surface area, has a good potential for application in the development of thinner, lighter, and more efficient respirators.

Adsorption capacity and critical bed depth are other important adsorption characteristics in assessing the potential of ACF in respiratory protection application, and were investigated and described in a previous study (Balanay *et al.*, 2014). However, pressure drop or breathing resistance across the respirator is another factor that must be considered, as it is one of the requirements tested by NIOSH and European Standards for respirator use approval (CEN, 2001, 2004; NPPTL, 2012a,b). This may be a concern for

AC due to the resistance to flow caused by its tightly woven fibers. Using a face velocity (i.e. 0.19 m s^{-1} based on a 6.9-cm cartridge diameter) similar to that used in NIOSH inhalation and exhalation resistance tests for chemical cartridge respirators (NPPTL, 2012a,b), preliminary experiments on ACF materials (4-cm discs) demonstrated that, at certain number of layers (i.e. 2–10), AC types had higher average pressure drop values ranging from 25 to 78 mm H_2O compared with those of the AF types ranging from 17 to 57 mm H_2O. Among the AF types, pressure drop also varied depending on the density of the adsorbent material (i.e. less dense AF types had lower pressure drop). Compared with the NIOSH maximum breathing resistance requirements of 40 (inhalation) and 20 (exhalation) mm H_2O, certain AF types had acceptable pressure drop. Adsorption of the ACF is determined by its mass, which in turn is related to its bulk density, whereas the pressure drop across the ACF is determined by its permeability. The permeability of the adsorbent can be controlled by changing its bulk density. In this consideration, a compromise between the bulk density and permeability must be reached. The pressure drop across ACFs in an actual respirator cartridge or filter must be further studied to determine the optimum dimensions, thickness, and density of ACF materials that will result in acceptably breathable respirators (Balanay *et al.*, 2014).

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