

See discussions, stats, and author profiles for this publication at: <https://www.researchgate.net/publication/255205363>

Next Generation Non-particulate Dry Nonwoven Pad for Chemical Warfare Agent Decontamination

ARTICLE *in* INDUSTRIAL & ENGINEERING CHEMISTRY RESEARCH · DECEMBER 2008

Impact Factor: 2.59 · DOI: 10.1021/ie801223b

CITATIONS

8

READS

90

10 AUTHORS, INCLUDING:



[Seshadri Ramkumar](#)

Texas Tech University

44 PUBLICATIONS 847 CITATIONS

SEE PROFILE



[Adam Love](#)

Roux Associates

26 PUBLICATIONS 223 CITATIONS

SEE PROFILE



[William Smith](#)

Lawrence Livermore National Laboratory

3 PUBLICATIONS 8 CITATIONS

SEE PROFILE



[Stephen Boyd Cox](#)

Research & Testing Laboratory

135 PUBLICATIONS 2,057 CITATIONS

SEE PROFILE

MATERIALS AND INTERFACES

Next-Generation Nonparticulate Dry Nonwoven Pad for Chemical Warfare Agent Decontamination

Seshadri S. Ramkumar,^{*,†} Adam H. Love,[‡] Utkarsh R. Sata,[†] Carolyn J. Koester,[‡] William J. Smith,[‡] Garrett A. Keating,[‡] Lawrence W. Hobbs,[§] Stephen B. Cox,[†] William M. Lagna,[∇] and Ronald J. Kendall[†]

The Institute of Environmental and Human Health, Texas Tech University, Lubbock, Texas 79409-1163, Forensic Science Center, Lawrence Livermore National Laboratory, PO Box 808, L-091, Livermore, California 94550, Hobbs Bonded Fibers, 200 South Commerce Drive, Waco, Texas 76710, and Edgewood Chemical Biological Center, U.S. Army Research, Development and Engineering Command, AMSRD- ECB-RT-D, 5183 Blackhawk Road, Aberdeen Proving Ground, Maryland 21010-5424

New, nonparticulate decontamination materials promise to reduce both military and civilian casualties by enabling individuals to decontaminate themselves and their equipment within minutes of exposure to chemical warfare agents or other toxic materials. One of the most promising decontaminating materials has been developed using a needle-punching nonwoven process to construct a nonparticulate composite fabric of multiple layers, including an inner layer of activated carbon fabric, which is well-suited for the decontamination of both personnel and equipment. This paper describes the development of a composite nonwoven pad and compares efficacy test results for this pad with results from testing other decontamination systems. The efficacy of the dry nonwoven fabric pad was demonstrated specifically for decontamination of the chemical warfare blister agent bis(2-chloroethyl)sulfide (HD or sulfur mustard). Gas chromatography/mass spectroscopy (GC/MS) results indicate that the composite fabric was capable of significantly reducing the vapor hazard from mustard liquid absorbed into the nonwoven dry fabric pad. The mustard adsorption efficiency of the nonwoven pad was significantly higher than that of particulate activated carbon and was similar to the currently fielded U.S. military M291 kit. The nonwoven pad has several advantages over other materials, especially its nonparticulate, yet flexible, construction. This composite fabric was also shown to be chemically compatible with potential toxic and hazardous liquids, which span a range of hydrophilic and hydrophobic chemicals, including a concentrated acid, an organic solvent, and a mild oxidant (bleach).

1. Introduction

Attacks on civilians and civilian infrastructure with chemicals, such as the attack by Aum Shinrikyo in Japan with the military nerve gas sarin¹ and recent attacks by Iraqi insurgents² with the industrial chemical chlorine, illustrate the need for decontamination systems for both civil and military defense. These decontamination systems must address a broad range of toxic military and industrial chemicals and be available to victims within minutes of a chemical accident or incident. These systems must also be inherently safe to use and noncorrosive if they are to be used on personnel and sensitive equipment.^{3–6} The need for an effective decontamination system that is capable of removing toxic chemicals and neutralizing the toxins to nontoxic byproducts has been highlighted by Lukey et al.⁷

Numerous strategies exist for decontamination after exposure to a liquid chemical, from physical removal of bulk liquid to chemical decomposition of the liquid into a nonhazardous form. Current physical decontaminants are capable of removing most liquids, including most toxic chemicals, from surfaces, while current reactive chemical decontaminants reduce the hazard for

a specific subset of chemicals, i.e., acids or chemical warfare agents. As a result, decontaminants that physically remove chemicals, such as diatomaceous earth and adsorbent carbon powders, have long been used in military systems for general-purpose decontamination of personnel and equipment. These powders readily disperse in the air and present respiratory hazards, both before and after they are used to decontaminate surfaces. Recently, many of the world's military organizations have fielded a reactive liquid decontamination solution (RSDL),⁷ which contains some water. RSDL is designed to reduce the hazard to personnel exposed to chemical warfare agents, such as the highly toxic nerve agents sarin (GB), soman (GD), and VX and the less toxic blister agent sulfur mustard (HD). Water is undesirable in general purpose decontamination systems because water damages sensitive materials, especially electronics. Therefore, new decontaminant approaches that are non-aqueous and devoid of loose particles are needed for personal and military equipment decontamination.⁶

Lukey et al.⁷ compared alternative decontamination systems, such as the particulate M291 Decontamination Kit, Sandia Foam, reactive skin decontamination lotion (RSDL), hypochlorite, diphotrine, and the reactive sponge. The M291 skin decontamination system is a particulate technology and consists of six individual pouches of carbonaceous reactive powder in a nonwoven fabric matrix. The particulate decontamination material in each pouch is a combination of adsorbent carbon, polystyrene polymer, and an ion-exchange resin. The M291 kit

* To whom correspondence should be addressed. Tel.: (806) 885 0228. Fax: (806) 885 2132. E-mail address: s.ramkumar@ttu.edu.

[†] The Institute of Environmental and Human Health, Texas Tech University.

[‡] Forensic Science Center, Lawrence Livermore National Laboratory.

[§] Hobbs Bonded Fibers.

[∇] Edgewood Chemical Biological Center, U.S. Army Research.

serves the dual purpose of physical and chemical decontamination of the toxic agent. As stated earlier, the particulate nature of the powder poses potential secondary exposure health risks by creating inhalable particles and leaving particulate residues that can potentially result in secondary contamination and exposure on decontaminated areas, including skin and equipment surfaces.⁷

The available liquid decontamination alternatives generally target a limited set of chemicals (i.e., acids or bases or chemical warfare agents), and none are expected to decontaminate as broad a range of industrial chemicals as physical decontaminants. RSDL is a patented liquid formulation for skin decontamination.⁷ Applying RSDL to skin with a cotton swab effectively decontaminates nerve agents such as VX, and some thickened agents, including thickened VX and thickened mustard, which is a blister agent.^{7,8} Scrubbing the skin with an RSDL soaked sponge more effectively decontaminates unthickened mustard on skin than less-vigorous application of RSDL with swabs or syringes.⁷⁻⁹ In addition, RSDL leaves an oily residue on skin and can make soldiers uncomfortable while performing certain military activities.⁷ Sandia foam, which is also called MDF-100 foam, is a formulation of Sandia National Laboratories. When mixed at the point of application, the formulation generates a foam that changes to a liquid in 30 min. MDF-100 has been reported to perform well against nerve agents such as GD (35-fold protective ratio) and VX (72-fold protective ratio).⁷ The protective ratio is the ratio of LD₅₀ for cutaneous exposure in animals after and before decontamination. However, Lukey et al.⁷ have reported that other decontamination solutions are more effective and provide a greater than 100-fold protective ratio against nerve agents such as VX. Another product (diphoterine) has been reported to reduce 50% of the mustard applied to human skin samples, which was higher than 37% mustard reduction by soapy water and 32% by saline, but was lower than the percentage of mustard reduction (67%–79%) in an in vitro skin test conducted using diluted and undiluted bleach.^{10,11} In addition, diphoterine is known to cause slight skin irritation.⁷ Hypochlorite, diluted 10-fold from household bleach solution (0.5% concentration), is ineffective for agents such as VX, which require a significantly higher amount of hypochlorite for effective decontamination, but undiluted bleach solution (5% hypochlorite), which is effective for VX, is damaging to skin.

Recently, attention has focused on developing blotting or wiping systems as an alternative to particulate decontamination technologies. These studies have predominantly focused on solvent-soaked fabric systems wherein the solvent dissolves the chemical warfare agents and the fabric matrix serves as a carrier medium for the solvent. Kaiser et al.¹² have developed a multilayer solvent-soaked composite that has polymer films on top and bottom. The composite wipe consists of ethoxyperfluorobutane impregnated fabric layers with a vapor barrier on the top and a permeable film on the bottom. Guinea pigs contaminated with soman were wiped using the solvent-soaked sponges and the M291 decon kit. Results showed that the solvent impregnated sponges have higher protection ratios than M291 decon powder. The decontamination capability was fully attributed to the ethoxyperfluorobutane solvent, but the physical decontamination contribution from the fabric alone was not characterized. Most recently, Smith et al.¹³ have suggested the use of a two-part personal decontamination system for exposure to an unknown liquid chemical, including toxic industrial chemicals and chemical warfare agents. This system first removes the bulk of chemicals from the skin by blotting with

the dry sorbent pad described below and then removes residual chemicals by scrubbing with a liquid applicator (an open-cell polyethylene foam sponge) soaked with RSDL.¹⁴

A composite fabric matrix with both adsorbency and absorbency promises to be a more effective decontamination medium than generic adsorptive materials such as activated carbon. Smith et al.¹³ have indicated that the use of fabric systems is potentially advantageous for decontaminating sensitive body surfaces, including mucous membranes and wounds, in comparison to abrasive and difficult-to-remove particulate decontaminants.

Given the shortcomings of currently fielded particulate decontamination systems, there has been renewed interest in constructing decontamination pads and wipes with activated carbon fabrics. Kaiser et al.¹⁵ have studied the relationship between adsorbent material characteristics, such as pore volume and pore size distribution, and the adsorption potential of commercially available activated carbon samples wetted with hydrofluoroether solvent. Their work focused on the adsorption of a chemical warfare agent simulant (2-chloroethyl ethyl sulfide) dissolved in a solvent, rather than on the adsorption of an actual undiluted chemical warfare agent. In solvent-wetted activated carbon, the solvent influences the adsorption of chemical molecules onto the adsorbent carbon. This influence is evident from the correlation between higher adsorption capacities of activated carbons and higher concentrations of mesopores in the carbons.¹⁵ In addition, the adsorption from liquid phases increases as the pore diameter increases, which has been attributed to the larger size of molecules being adsorbed from a liquid phase.¹⁵ Smaller pore diameters suffice for the adsorption of toxic chemicals from a gaseous vapor phase. Activated carbons exhibit different adsorption dynamics when they are challenged by chemical warfare agents in vapor phases, compared to challenges by chemical warfare agents in liquid phases. Activated carbon with a microporous pore structure (pore diameter of <2 nm) is optimal for sequestering agent vapor and a mesoporous pore structure (~2.9 nm) is optimal for the removal of liquid chemical warfare agents.¹⁵

Recently, there have been some developments in new reactive decontamination formulations for sponge wipes based on cholinesterase and oxidative oxime reactivity for use in skin decontamination. Gordon et al.¹⁶ have been successful in developing polyurethane sponges with cholinesterase and other chemical warfare agent decontaminating enzymes. Their reactive sponge is a polyurethane matrix that contains immobilized cholinesterase (ChE) enzymes.¹⁶ The reactive sponge has been found to be stable for extended periods at high temperature and can be reused due to the presence of oximes.¹⁶ Although these sponges can detoxify and prevent secondary contamination, their efficiency can be enhanced by the presence of highly adsorbent cores, as in the case of the multilayered nonwoven pad reported in this paper. In addition, Gordon et al.¹⁷ have evaluated the decontamination efficacy of reactive-enzyme-immobilized sponges for agents such as GD and VX. In comparison with other formulations that contain water, such as RSDL, the reactive sponge was equally effective for VX and rather more effective in the case of GD.¹⁷ In addition, the reactive sponge outperformed the carbon powder wipe in the M291 kit, in regard to its decontamination potential in detoxification of GD and VX.¹⁷ A detailed overview of the current personal protective and decontamination technologies has been provided by Ramkumar et al.¹⁸

The aforementioned discussion illustrates the characteristics that can be optimized to develop dry pads and wipes that are efficacious for industrial chemicals as well as chemical warfare

agents. The dry pad or wipe would have both absorbent and adsorbent capabilities that remove bulk chemicals and retain toxic vapors. It is essential to find an alternate technology that will be devoid of loose particles, contain no free water, and be useful for both sensitive military equipment and personal decontamination. The need for such water-free nonaqueous and nonparticulate dry detoxifying technologies has been recognized by the U.S. military.⁶ There has been limited information from studies with dry pads or wipes using neat chemical warfare agents and the associated efficacy of such items for their agent vapor retention capabilities.

This paper presents results on the development of a multi-layered dry pad, containing a core of activated carbon adsorbent, and its retention capability for the chemical warfare agent sulfur mustard. Sulfur mustard (HD) was chosen for initial characterization of the wipe primarily because this blister agent is less soluble in water than the more-common nerve agents sarin and soman. Hence, physical absorption of bulk sulfur mustard into a wipe was expected to be more effective than destruction by any of the reactive aqueous-based decontamination alternatives previously described. A dry nonwoven absorbent-adsorbent pad that contained a core of activated carbon adsorbent has been developed. This nonparticulate pad provides enhanced decontamination capabilities for a broad range of chemicals by both absorbing liquids and adsorbing vapors. The flexible pad can decontaminate both sensitive surfaces of the human body, including skin, mucous membranes and wounds, and surfaces of sensitive electronic equipment.¹⁹ The dry absorbent-adsorbent pad provides more decontamination functions than a regular activated carbon pad. The nonparticulate and nonwoven dry pad physically decontaminates toxic mustard by absorbing the bulk liquid and capturing off-gassing vapors. The absorbent layers on the top and bottom of the core activated carbon layer quickly remove the bulk liquid chemical warfare agent when a pad is pressed against a surface. The middle nonwoven activated carbon core captures toxic vapors from the absorbed liquid. The surrounding absorbent layers also prevent the linting and shredding of activated carbon, thereby preventing secondary contamination. The dry nonwoven composite pad addresses one of the immediate requirements of the U.S. government for military and civilian defense,⁶ namely, an effective decontamination system with broad application to the decontamination of both individuals and sensitive equipment.

2. Experimental Methods and Materials

2.1. Needle-punching Technology for Manufacturing Nonwoven Fabrics. Needle-punching nonwoven technology was used to manufacture the three-layered nonwoven pad, which consists of absorbent fabrics on the top and bottom with activated carbon nonwoven fabric in the middle. The top and bottom absorbent fabrics were individually needle-punched and then assembled with the adsorbent activated carbon in the middle. The three layers were then needle-punched to form the flexible nonwoven pad. Needle-punching technology uses a series of barbed needles to interlock fibers to produce well-integrated and coherent fabrics.^{20,21} Needle-punching technology converts fiber into fabric without processes such as spinning, winding, and weaving. In the needle-punching technology, fibers are first opened and then blended in the hopper feeder. These fibers are then separated into single fibers by a set of revolving rollers in a process referred to as carding, which uses worker and stripper rollers. The individualized fibers coming out of the carding process are aligned in one direction as a partially coherent web. Several layers of carded webs are piled up to

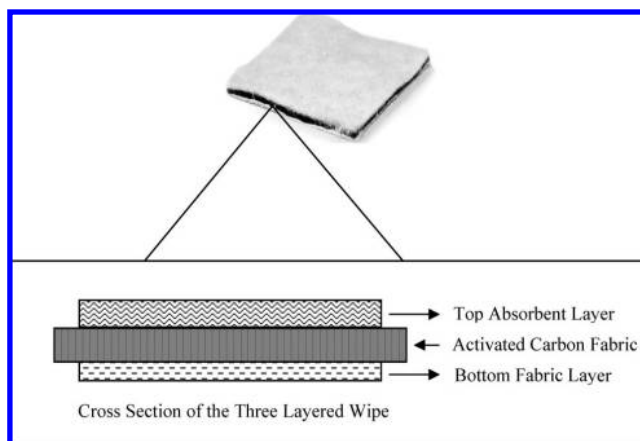


Figure 1. Nonwoven decontamination pad.

obtain the necessary weight and are subsequently oriented in the cross direction by the crosslapper. Crosslapping provides the final nonwoven fabric with a good balance of strength in both directions. The cross-lapped web is then subsequently needle-punched using a series of needles to produce an interlocked fabric. The fibers in needle-punched fabrics are interlocked by the punching action of a series of barbed needles. The needles interlock the fibers without seriously damaging them. The needle-punched fabric, because of the mechanical interlocking, is a more flexible fabric than other fabrics bonded by thermal or laminating processes. Needle-punching technology is suitable for developing heavy weight fabrics and composites. More recently, Roedel and Ramkumar²¹ have developed lightweight fabrics from different fibers, such as polyester and cotton. The development of such fabrics proves that needle-punching technology is versatile and can be used to develop multipurpose composites, such as chemical protective suit liners.^{20,21} The use of needlepunching to develop activated carbon nonwoven composite fabrics is different from the bonding technique adopted by Kaiser et al.¹² The needle-punching technology provides an enhanced composite structure, both in terms of adsorption characteristics and flexibility.

2.2. Nonwoven Composite Pad. The three-layered nonwoven composite pad was developed at Hobbs Bonded Fibers (Waco, TX), using a manufacturing-scale needle-punching line. Viscose fibers, polyester fibers, and the nonwoven activated carbon fabric were procured from three different commercial sources. The viscose fibers in the top fabric were single-needle-punched (top to bottom) to provide liquid absorption capability. The middle porous activated carbon nonwoven fabric serves as the adsorbent layer. The bottom polyester fabric, prepared by single needlepunching, like the top fabric, enhances the overall structural integrity and strength of the composite.

The activated carbon nonwoven fabric was evaluated for its surface area and pore size distribution by Professional Analytical and Consulting Services (Coraopolis, PA). The multipoint Brunauer–Emmett–Teller (BET) surface area was determined to be 1.071 m²/g, and the pore size ranged from 0.5 nm to 2 nm.²² The large surface area and small pores show that the activated carbon fibers are microporous in nature and can be effectively used to adsorb vapors.

The three layers were then needle-punched together to form the composite pad. Figure 1 shows the multilayered pad with top and bottom absorbent layers. Scanning electron microscopy (SEM) of the multilayered pad (Figure 2) shows that the middle activated carbon layer remains intact and is not damaged by the needling process. Needle-punching has two primary advan-

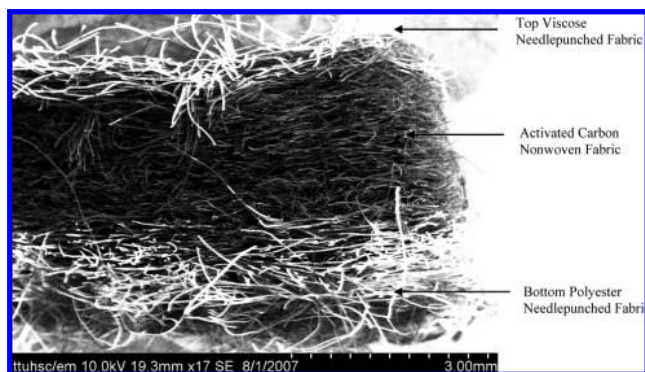


Figure 2. SEM image of the three-layered composite with unshredded activated carbon nonwoven fabric in the middle.

Table 1. Details of Materials Used^a

physical property	Type of Material		
	viscose nonwoven fabric	polyester nonwoven fabric	nonwoven activated carbon
weight	100 g/m ²	80 g/m ²	140 g/m ²
fiber denier	3	6	
fiber staple length	50.8 mm	50.8 mm	
BET surface area			1071 m ² /g ^b

^a Viscose and polyester fiber details were provided by each manufacturer. ^b Surface area data for nonwoven activated carbon were obtained from Professional Analytical and Consulting Services.²²

tages: (i) prevention of shredding of the brittle activated carbon fibers and (ii) retention of open pores. The physical attachment of the fabric layers enables the pores of the activated carbon fibers to remain open and active, whereas resin bonding typically seals the pores.

The details of materials used for developing the nonwoven pad are given in Table 1.

2.3. Methods. 2.3.1. Evaluating Efficacy of the Nonwoven Composite Pad. Evaluations of the efficacy of the nonwoven composite pad were performed at the Forensic Science Center at Lawrence Livermore National Laboratory (LLNL). The nonwoven composite dry pad was initially evaluated, along with other sorbents, by measuring the relative amounts of mustard, bis(2-chloroethyl)sulfide, that would be adsorbed after being deposited on the material. The mustard used for this evaluation was synthesized at LLNL; its purity was verified to be $\geq 99\%$ by gas chromatography/mass spectrometry (GC/MS). The other materials tested were powdered activated carbon (PAC) (Mallinckrodt Chemical Corp.) and the M291 kit (U.S. Department of Defense).

The materials were weighed and placed in individual, 40-mL, VOA (volatile organic analysis) vials. The amount of sorbent used was sufficient to ensure that the liquid mustard droplet fell on the sorbent surface. Three replicate vials were prepared for each adsorbent. Identical, 40-mL VOA vials without sorbent were used as positive control experiments. Using a 10- μ L Hamilton syringe, 5 μ L of mustard were dropped onto each sorbent surface. The VOA vials, containing the sorbent and the mustard, were capped and held for ~ 24 h, at ambient temperature, for equilibration. The headspace of each vial was sampled using a 50- μ L, gas-tight Hamilton syringe and the vapor was injected directly into a GC/MS system for quantification. GC/MS analysis was performed using an Agilent Model 5973 GC/MS system (Agilent Technologies, Palo Alto, CA), and quantification was performed using peak areas of the m/z 109 ion of sulfur mustard.

These experiments were conducted in two sets; therefore, separate controls were utilized for each set. Control 1 and the nonwoven pad analysis were performed as one set and Control 2, the M291, and the PAC were analyzed as the second set. Results for each material were normalized to the control vapor concentration to appropriately represent the associated reduction in vapor concentration. The calibration curve for mustard was linear in the concentration range of all the vials measured; therefore, normalized GC/MS peak areas represent normalized concentrations.

2.3.2. Evaluating the Chemical Compatibility of a Dry Nonwoven Composite Pad. Other characteristics of the composite pad were also examined. The compatibility of the composite pad with other chemicals was to ensure that the use of the decontamination pad, even for unknown chemicals, is unlikely to cause harm. The compatibility tests address potential concerns about reactivity of the composite pad to bleach, especially after saturation with liquid chemical warfare agent, and the disintegration of pads when they contact aggressive acids or solvents. To check for the release of vapors containing mustard by bleach treatment after use, 4-cm² pads previously exposed to mustard were dipped in 10% sodium hypochlorite (bleach) and observed for any adverse reactivity, as evidenced by either heat or vapor production. To check for reactivity and disintegration during use, unexposed pieces of 4 cm² pads were soaked in 70% nitric acid and *p*-xylene, which is an organic solvent, and were observed for reactivity, heat production, and material degradation.

2.3.3. Evaluating the Liquid Absorption Capacity of a Dry Nonwoven Composite Pad. One additional simple test was performed to determine the capacity of the composite pad for hydrophilic and hydrophobic liquid absorbency. The area that a pad can decontaminate is proportional to its liquid absorptive capacity. To measure the capacity of a pad, the pad was cut into 4-cm² pieces, each weighing ~ 110 mg, and the thickness of the piece was measured with a micrometer. Each piece of a pad was weighed using an analytical balance (Model AT 201, Mettler Toledo, Columbus, OH), and then was soaked in liquid (either water or *p*-xylene) for 1 min and allowed to briefly drain for 10–15 s. The amount of residual liquid absorbed into the soaked piece was then determined gravimetrically by weighing the drained piece, using the same analytical balance, and then subtracting the weight of the dry pad.

3. Results and Discussion

3.1. Comparative Analysis for Performance Evaluation of Decontamination Materials. The relative performance of the composite pad for adsorbing mustard vapor, compared to both the PAC and the M291, is shown in Figure 3. The best sorbents maintain the lowest concentrations of mustard vapors in the vial headspace. The mustard vapor concentration is normalized to the control from the associated set, to indicate the relative reduction in vapor concentration. The average GC/MS peak areas and the standard deviations are shown in Table 2. The standard deviation was determined from three replicate values. The results in Figure 3 show that the composite pad is able to reduce the concentration of mustard vapors by more than three orders of magnitude. Analysis of variance (ANOVA) was used to compare log-transformed peak areas, which is a measure of adsorption, among decontamination systems. Log-transformation was used to alleviate violations of the assumptions of ANOVA (i.e., normality and additivity). First, adsorption of the nonwoven pad was compared to its control value. Then, adsorption of M291, PAC, and their control values were

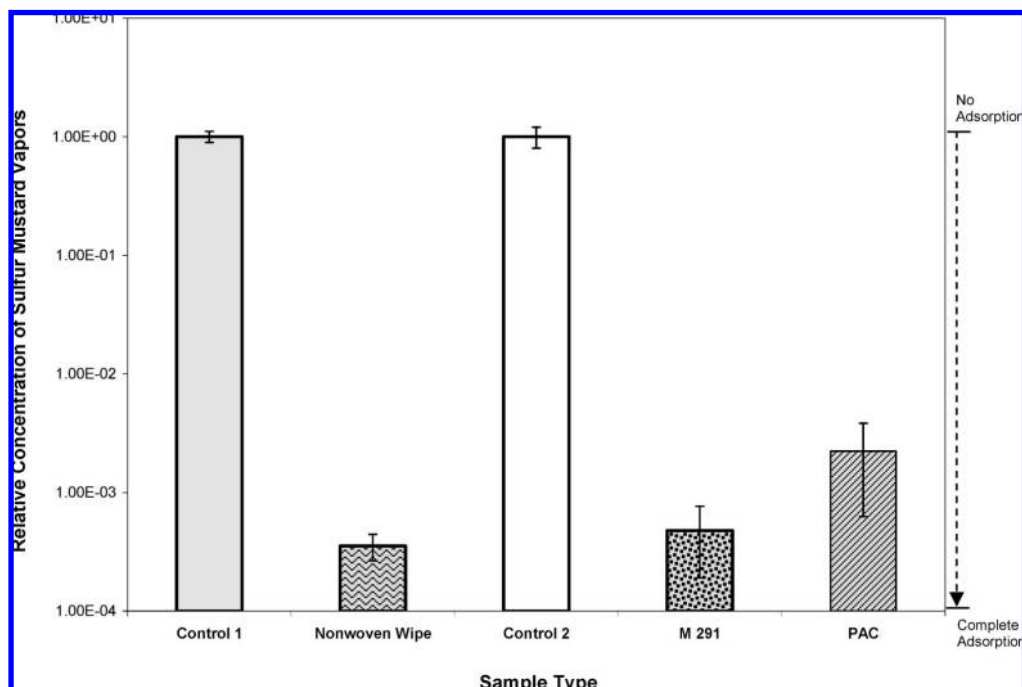


Figure 3. Adsorption of sulfur mustard vapors by various adsorbent materials.

Table 2. GC/MS Peak Area for *m/z* 109 of Sulfur Mustard (HD)^a

decon type	average peak area	Average Normalized Peak Area Using Respective Mean Controls ^b	
		control 1	control 2
control 1	3.50×10^7 (3.76×10^6)	1.00×10^0 (1.07×10^{-1})	
nonwoven wipe	1.24×10^4 (3.12×10^3)	3.55×10^{-4} (8.92×10^{-5})	
control 2	3.77×10^7 (7.50×10^6)		1.00×10^0 (1.99×10^{-1})
M 291	1.80×10^4 (1.08×10^4)		4.78×10^{-4} (2.87×10^{-4})
PAC	8.40×10^4 (6.05×10^4)		2.23×10^{-3} (1.60×10^{-3})

^a Values given in parentheses indicate the respective standard deviations, which correspond to three repeats. ^b Individual GC/MS peak areas associated with the vapor concentration of HD in the headspace of the vials that contain decontaminant samples are normalized by their respective control peak area values, which represent the vapor concentration of the agent in the headspace of the vials that contain the CW agent HD and no decontamination sample.

compared. Finally, arcsin-transformed adsorption efficiencies of the three decontamination systems were compared. For these analyses, adsorption efficiency was measured as the percent reduction of the control peak area of the three different decontamination systems, relative to their respective controls. Tukey's test was used to conduct post hoc multiple comparisons. All analyses were conducted using the R environment²³ (R Development Core Team, 2006). The nonwoven pad significantly increased adsorption, as reflected by lower peak areas, compared to its control ($p < 0.001$). The p value is a statistical measure that indicates the probability that the observed differences could have arisen due to random variation. For example, if $p < 0.05$, there is a <5% chance that the observed differences could be due to random variation. Similarly, both the M291 ($p < 0.001$) and PAC ($p < 0.001$) significantly increased adsorption. When the three decontamination systems were compared, adsorption efficiency of the nonwoven pad was significantly better than that of PAC ($p = 0.041$) and was similar to M291 ($p = 0.952$). In addition, variability among the three replicate estimates of nonwoven pad adsorption efficiency was less than those of the three M291 and PAC replicates (see Figure 4).

3.2. Chemical Compatibility of Dry Nonwoven Composite Pad. Table 3 shows the results from observations of chemical compatibility of the composite pad with the three chemicals

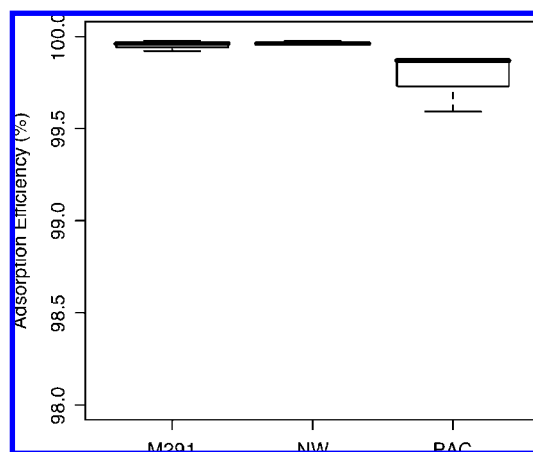


Figure 4. Adsorption efficiency of different decontamination materials. (NW denotes the nonwoven composite pad.)

tested, in which the pad demonstrated no adverse reactivity or decomposition with either bleach, nitric acid, or *p*-xylene.

3.3. Liquid Adsorption Capacity of Dry Nonwoven Composite Pad. Table 4 shows the pad absorption capacity for hydrophilic (water) and hydrophobic (*p*-xylene) liquids. The composite pad was able to absorb 14 times its own weight in water and 11 times its own weight in *p*-xylene. This capacity is similar to microfiber cloth products that have been evaluated

Table 3. Compatibility of Nonwoven Dry Pad with Chemicals

parameter	value/comment
material	nonwoven dry pad (4 cm ²)
compatible with bleach?	yes
compatible with nitric acid?	yes
compatible with <i>p</i> -xylene?	yes

Table 4. Liquid Absorption Capacity of Nonwoven Dry Wipe

parameter	value
material	nonwoven dry pad (4 cm ²)
thickness (cm)	0.46
water absorption	
g of H ₂ O per g of material	14.3
g of H ₂ O per cm ³ of dry material	1.06
<i>p</i> -xylene absorption	
g of <i>p</i> -xylene per g of material	11.8
g of xylene per cm ³ of material	0.84

previously, which are designed for high liquid capacity (~10 times their own weight of liquid).

A quantitative understanding of the specific mechanistic influence of the components of the pad on the absorption and adsorption capabilities is outside the scope of this paper. Nonetheless, the general performance of the pad can be associated with specific characteristics of the dry pad. The viscose and polyester top and bottom layers enable the wicking and absorption of bulk liquid. The overall construction of the nonwoven fabric results in a liquid holding capacity that is high, compared to its weight and consistent with other microfiber cloths. The adsorption capability of the fabric is associated with the activated carbon fabric core layer and the pore size distribution of the activated carbon nonwoven fabric is well-suited for vapor adsorption and retention.

4. Conclusions

Based on these initial evaluations, the development of a nonparticulate dry decontamination composite pad using needle-punching nonwoven technology seems to have significant promise for the next generation of dry decontamination systems. The needle-punching nonwoven technology was shown to be capable of constructing a flexible pad without any shredding of the activated carbon core, while retaining sufficient flexibility to enter into the crevices of objects to be decontaminated. The pad has many of the characteristics desired: (i) absorption of significant quantities of bulk liquid chemicals without adverse reactivity or disintegration and (ii) affinity for and capacity to adsorb and retain chemical vapor. These characteristics are important improvements over the existing technology for the military, the M291 kit. Additional testing is required to verify the performance of the composite pad on a wider array of potential threats.

The nonwoven composite fabric was evaluated here as a dry nonreactive pad. The fabric also has the potential to serve as a platform technology for including additional functionality to make reactive and self-cleaning pads and wipes. Nonwoven composite fabrics could also have additional forms and applications related to individual protection (e.g., clothing) or collective protection (e.g., tents). This approach to construction of the next generation of decontamination systems deserves to be seriously considered. These pads that are robust and sufficiently flexible to conform to difficult shapes and spaces, absorb large quantities of liquids, adsorb toxic vapors, and are compatible with a wide range of toxic or hazardous chemicals.

Acknowledgment

The evaluation of this composite wipe was part of a DHS-funded project, managed by the Technical Support Working Group (TSWG), to develop a Low-Cost Personal Decontamination System. The project was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory (under Contract W-7405-Eng-48). S.R. gratefully acknowledges the U.S. Army Research, the Development and Engineering Command of the U.S. Department of Defense, for broad-based support of his research on nonwoven fabrics for personal protection and decontamination. S.R. also acknowledges the Food and Fibers Research Grant Program of Texas, The CH Foundation of Lubbock, Cotton Foundation, South Plains Foundation, Lubbock, ICRC of CASNR-TTU and Plains Cotton Growers, Inc. for supporting his nonwovens research at Texas Tech University. Garfield Purdon and Andrew Burczyk of Defence Research and Development Canada—Suffield, Philip O'Dell of O'Dell Engineering, and Nayla Feghali of E-Z-EM, Inc., provided valuable insights into the design and performance requirements for the dry pad.

Literature Cited

- (1) Tu, A. T. Toxicological and Chemical Aspects of Sarin Terrorism in Japan in 1994 and 1945. *Toxin Rev.* **2007**, 26 (3), 231.
- (2) Weitz, R.; Al-Marashi, I.; Khalid, H. Chlorine as a Terrorist Weapon in Iraq. WMD Insights, U.S. Department of Defense Threat Reduction Agency (DTRA), May 2007 (15). (http://www.wmdinsights.com/PDF/WMDInsights_May07Issue.pdf, as accessed on April 15, 2008.)
- (3) U.S. Department of Defense (DoD). Chemical and Biological Defense Program, Annual Report to Congress, 2006. (<http://www.acq.osd.mil/cp/nbc06/cbdpreporttocongress2006.pdf>, as accessed on September 17, 2007.)
- (4) U.S. Department of Defense (DoD). Chemical and Biological Defense Program, Annual Report to Congress, 2007. (<http://www.acq.osd.mil/cp/cbdreports/cbdpreporttocongress2007.pdf>, as accessed on September 17, 2007.)
- (5) Gurudatt, K.; Tripathi, V. S.; Sen, A. K. Adsorbent Carbon Fabrics: New Generation Armour for Toxic Chemicals. *Defense Sci. J.* **1997**, 47 (2), 239–250.
- (6) U.S. Department of Defense (DoD). Chemical and Biological Defense Program, Annual Report to Congress, March 2005. (<http://www.acq.osd.mil/cp/nbc05/cbdpreporttocongress2005.pdf>, as accessed on September 17, 2007.)
- (7) Lukey, B. J.; Hurst, G. C.; Gordon, R. K.; Doctor, B. P.; Clarkson, E. IV.; Slife, H. F. Six Current or Potential Skin Decontaminants for Chemical Warfare Agent Exposure—A Literature Review. In *Pharmacological Perspectives of Toxic Chemicals and Their Antidotes*; Flora, S. J. S., Romano, J. A., Baskin, S. I., Sekhar, K., Eds.; Narosa Publishing House: New Delhi, India, 2004; p 13.
- (8) van Hooidonk, C.; van der Weil, H. J.; Langerberg, J. P. Comparison of the Efficacy of Skin Decontaminations II. In-vivo Text—Final Report, Prins Marutis Laboratory, TNO Report PML, 1996.
- (9) Bide, R. W.; Burczyk, A. F.; Risk, D. J. Comparison of skin decontaminants for HD: Canadian Reactive Skin Decontaminant Lotion, Canadian Decontaminating Mitt and US Skin Decontaminant Kit. In *Proceedings of the 1996 Medical Defense Biosciences Review, Bioscience '96*; U.S. Army Medical Research and Development Command, 1996.
- (10) Gerasimo, P.; Blomet, J.; Mathieu, L. Diphtherine Decontamination of Cl₄[−] Sulfur Mustard Contaminated Human Skin Fragments *in vitro*. *The Toxicologist* **2000**, 54 (1), 152.
- (11) Dolzine, T. W.; Logan, T. In *Proceedings of the 1998 Medical Defense Biosciences Review, Bioscience '98*; U.S. Army Medical Research and Development Command, 1998, No. AD MOO 1167.
- (12) Kaiser, R.; Gordon, R. K.; Clarkson, E. Novel Decontamination Wipes. In *Proceedings of the 2002 Joint Service Scientific Conference on Chemical & Biological Defense Research*, Hunt Valley, MD, 2002.
- (13) Smith, W. J.; Love, A. H.; Koester, C. J.; Purdon, J. G.; O'Dell, P.; Bearinger, J. P.; Keating, G. A.; Noy, A.; Verce, M. Evaluating the

Efficacy of Low-Cost Personal Decontamination System (LPDS) Formulations for Sulfur Mustard and Assorted TICs. In *Proceedings of DECON 2005*, Joint Program Manager for Decontamination, Tucson, AZ, 2005.

(14) Burczyk, A. F.; Purdon J. G.; Chenier C. The Canadian Reactive Skin Decontamination Lotion Polymeric Foam Material Applicator System. In *Proceedings of the 1998 Medical Defense Biosciences Review, Bioscience '96*; U.S. Army Medical Research and Development Command, 1996.

(15) Kaiser, R.; Kulczyk, A.; Rich, D.; Ronald, J. W.; Minicucci, J.; MacIver, B. Effect of Pore Size Distribution of Commercial Activated Carbon Fabrics on the Adsorption of CWA Simulants from the Liquid Phase. *Ind. Eng. Chem. Res.* **2007**, *46* (19), 6126.

(16) Gordon, R. K.; Feaster, S. R.; Russell, A. J.; LeJeune, K. E.; Maxwell, D. M.; Lenz, D. E.; Ross, M.; Doctor, B. P. Organophosphate skin decontamination using immobilized enzymes. *Chem.-Biol. Interact.* **1999**, *119* (120), 463.

(17) Gordon, R. K.; Owens, R. R.; Askins, L. Y.; Baker, K.; Ratcliffe, R. H.; Doctor, B. P.; Clarkson, E. D.; Schulz, S.; Railer, R.; Sigler, M.; Thomas, E.; Ault, K.; and Mitcheltree, L. W. Chemical, Biological, and Radiological Decontamination and Detoxification Using Polyurethane Sponges. In *Proceedings of the 2006 Scientific Conference on Chemical and Biological Defense Research*, Hunt Valley, MD, 2006.

(18) Ramkumar, S. S.; Sata, U. R.; Hussain, M. M. Personal Protective Fabric Technologies for Chemical Countermeasures. In *Advances in Biological and Chemical Terrorism Countermeasures*; Kendall, R. J., Presley, S. M., Austin, G. P., Smith, P. N., Eds.; Taylor and Francis Group: New York, 2008; *Ch.* 8, p 203.

(19) Sata, U. R.; Ramkumar, S. S. New Developments with Nonwoven Decontamination Wipes. In *Proceedings of International Nonwovens Technical Conference 2007*, Atlanta, GA, 2007.

(20) Ramkumar, S. S. Development of Protective Clothing Substrates. A New Approach. *AATCC Rev.* **2002**, *2* (2), 28.

(21) Roedel, C.; Ramkumar, S. S. Development and the Study of the Surface Mechanical Properties of H1 Technology Needle punched Nonwoven Substrates. *Textile Res. J.* **2003**, *73* (5), 381.

(22) Test report, Professional Analytical and Consulting Services, Coraopolis, PA 15108, January 5, 2008.

(23) R Development Core Team. R: A language and environment for statistical computing, R Foundation for Statistical Computing: Vienna, Austria 2007. (<http://www.R-project.org>, as accessed on April 15, 2008.)

Received for review August 8, 2008

Revised manuscript received September 22, 2008

Accepted September 24, 2008

IE801223B