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1978 Intersectional Technical Paper Competition

Second Runnerup: Northern New England Section

Novel Sorbent Yarns and Fabrics Containing Active Carbon

THIS research was directed toward new approaches for incorporating active carbon in sorbent textile systems suitable for occupational protective clothing. The approaches were designed to overcome material deficiencies which became obvious in earlier work. We have included also pertinent background on active carbon technology.

Active Carbon Technology

Ordinary Forms and Uses Of Active Carbon

Active carbon is basically a substrate for sorbing gases and vapors as well as various solutes from gaseous or liquid solutions. It is normally available in various forms including fine powder, granules and fibers. The powdered and granular forms of active carbon are most commonly used for liquid or solution purification, but are also used for air purification in ventilation systems, respirators, protective clothing and cigarettes. Some of these air purification

ABSTRACT

Two promising textile systems were developed for use in permeable protective clothing to protect industrial and laboratory workers against toxic chemical vapors. In one system, active carbon powder was included in the cores of microporous polypropylene hollow filament yarns suitable for manufacture of clothing fabrics. In the other system, a layer of active carbon fabric was laminated between porous polypropylene nonwoven fabrics. Both systems offer durable fabrics which can sorb high levels of toxic vapors, provide resistance to sweat poisoning of the active carbon and minimize physiological heat stress.

KEY TERMS

Active Carbon
Carbon
Nonwoven Fabrics
Polypropylene Yarn
Protective Clothing

applications involve carbon powder which is dispersed and bound onto a suitable substrate, such as polyurethane foam, paper or nonwoven fabric. Fibrous forms of carbon are also being considered seriously for air purification because the fibers do not pack together as closely as the powder and no binders are required.

Active Carbon in Textile Systems

In a garment fabric system, active carbon fibers, yarns or powder can provide a potential means of protecting industrial and laboratory workers from vapors of toxic chemicals which may be sorbed by the body through the skin. However, the fabrics containing active carbon must not only protect against toxic chemicals but must also minimize the heat load imposed on the worker. Fabrics of fibrous materials can offer less physiological heat stress than air-impermeable coated fabrics because they can be made thin and with high air and water vapor transport.

When incorporating active carbon in a textile system, consideration must be given to methods of avoiding loss of carbon. It has so far proven impossible to hold carbon powder with a resin binder without reducing its toxic vapor sorptivity. The amount of carbon in the system must thus be increased to compensate for this loss of effectiveness. This problem is eliminated when active carbon yarns themselves are incorporated into a textile structure (1). However, by themselves, fibrous forms of active carbon in textile systems suffer from extremely low abrasion resistance. But by protecting the carbon fiber with an abrasion resistant covering which is air permeable (2), the wear properties could be improved.

New Research Approach For Active Carbon in Textile Systems

This report addresses three salient problems identified in previous work on the use of active carbon in textiles. The most obvious difficulty is the poor abra-

sion resistance of active carbon fibers and yarns when their fabric surfaces are exposed to normal wear and tear. Another is sweat poisoning, which refers to the fact that organic components dissolved or suspended in sweat are preferentially sorbed in the unprotected carbon and reduce its effectiveness. A third problem is the excessive insulation of present garment systems—e.g., carbon-impregnated foams—which not only increase sweat production but also cause heat prostration when such garments are worn for protracted periods in hot and humid environments.

In this research, both fiber and fabric approaches were developed to resolve or reduce the effects of these problems. In the fiber approach, microporous walled hollow fiber was used to encapsulate active carbon powder. This provided an abrasion resistant and potentially sweat protective covering for the carbon. In the fabric approach, active carbon woven fabric was laminated between nonwoven sheets which have an extraordinarily high liquid water resistance and high water vapor permeability. The laminate also provided the carbon fabric with abrasion resistance and presumably a barrier to liquid sweat. Both the fiber and fabric approaches have potential for minimizing physiological heat stress when incorporated into garments.

Preparation Of Active Carbon

Active carbon can be prepared from precursors such as bones, nut shells, fruit pits, coal, petroleum and synthetic or man-made fibers by pyrolysis to carbon in an inert atmosphere, followed by activation in an oxidizing gas atmosphere. The precursors are nonmelting and can be in any of several forms, such as powder, granules, fibers, yarns and fabric. Pyrolysis is usually carried out at temperatures up to 500C, while activation, which forms the pores and develops surface area, is carried out at a higher temperature in the vicinity of 1000C. The oxidizing gas for activation

is usually carbon dioxide or superheated steam. Prior to pyrolysis, some precursors require a lower temperature preoxidation step to develop the non-melting characteristic necessary for pyrolysis. This characteristic is critical for fibrous precursors because textile morphology must be retained and the material must not become stiff due to fiber and/or yarn interbonding.

Characterization Of Active Carbon

For gas and vapor sorption applications, activation must produce a proper pore structure consisting of a distribution of two types of pores; i.e., macropores and micropores (3). Transitional pores normally considered to be a prerequisite for sorption in liquid systems are not required for gas and vapor sorption. These three pore types are classified according to size as follows: micropores having less than 30 Å diameter, transitional pores in the 30 Å to 2000 Å range, and macropores over 2000 Å diameter.

Formation of the small pores results in high surface areas on the order of 500-1200 m²/g. The macropores function as arteries and have a negligible

wall surface area in the range of 0.5-2 m²/g.

The potential sorptivity of active carbon for gases, vapors and liquids can be determined by conventional active carbon analyses for surface area, pore diameter and pore volume. The BET method is used to determine pore size distribution and surface area of macropores, transitional pores and micropores by means of nitrogen sorption and desorption isotherms. The iodine number can be used for determining surface area of pores with diameters greater than 10 Å. The mercury intrusion porosimeter at pressures up to 60,000 psi is used to determine macropore and transitional pore size distribution and the total pore volumes. The helium pycnometer provides density measurements which indicate the total volume of macropores, transitional pores and micropores. The liquid phase sorption capacity of active carbons can be determined by following the decrease of methylene blue or other dye concentration from solution. The Freundlich sorption isotherms thus obtained offer a quick comparison of different activated carbons. The methods for evaluating reactive sites depend on the specific technique of preparing the active carbon.

The pore surface of activated carbons not only has a carbon molecular asymmetry which causes the adsorption of gases, liquids and vapors, but also has functional groups containing hydrogen and oxygen. The hydrogen comes from the precursor and possibly from the superheated steam during activation; the oxygen comes from oxidation/activation processes. These elements are present in such forms as hydroxyl, peroxy, peroxide or carbonyl groups. These groups increase the sorptive potential of the activated carbon by providing further attractive forces toward sorbed molecules.

Kiselev et al. (4) modified the chemical nature of the carbon surface. They did not affect the surface area but significantly increased the number of functional groups. This was done by oxidizing the carbon for a short time in oxidizing agents; e.g., hydrogen peroxide, sodium hypochlorite, potassium permanganate. By this means, they made both hydrophobic and hydrophilic carbons.

Experimental

Test Procedures

Textile tests performed on the activated carbon filled hollow fibers and the laminated active carbon fabrics were in accordance with Federal Test Method Standard No. 191. A static CCl₄ vapor

sorption test was used as a preliminary screening tool to measure the level of activation of the carbon yarns. The specimen was allowed to sorb CCl₄ saturated vapor until equilibrium was attained; i.e., until it reached a maximum constant weight. The per cent weight increase was then calculated.

Sorption of CCl₄ vapors was also determined in a dynamic test method in which equilibrium conditions were not attained. The intent was to simulate a typical environment of toxic vapors at lower concentrations. The basic procedure involved the passing of the CCl₄ vapor through 100 cm² of the fabric sample at a concentration of 15 mg/l. at 33°C and 80% RH, and with a face velocity of 10 cm/min (flow rate 1 liter/min), and monitoring the effluent steam by passing it into a specific solvent. The dissolved vapor was then estimated colorimetrically in a developing agent system.

Two variations of this dynamic sorption procedure were used for fibers. In one method, fibers were arranged in parallel bundles (as in a fabric) and the bundles were laid at right angles to each other in layers. In the other method, a test tube was packed with fibers. In both cases, a CCl₄ vapor concentration of 5 mg/l. was used.

Preparation Of Yarn

The preparation of sorbent textile fibers was carried out by sheath-core spinning a slurry of active carbon powder into a wax/polypropylene blend hollow filament. The active carbon was regenerated and the walls made porous by extracting the carrier fluid from the slurry and the wax from fiber walls. Drawing was carried out to improve textile mechanical properties.

The sheath-core spinning was carried out by extruding the wax/polymer blend through the annulus of a spinneret comprising a hollow needle centered in an orifice (5). The carbon slurry was extruded simultaneously by means of a separate extruder feeding through the hollow needle into the core of the extruded polymer. Optimum high solids-loaded fluid slurries of active carbon were prepared while developing the pumping techniques and determining particle size required for their extrusion. It was eventually determined that slurries of pulverized samples (95% <325 mesh) classified through a 200-mesh screen could be pumped satisfactorily with a Zenith gear pump through spinnerets for considerable times without any noticeable interference with flow.

To achieve this product, slurries of three different active carbons in a variety of carrier fluids were investigated to

About ITPC

A POPULAR feature of AATCC conferences since 1940, the Inter-sectional Technical Paper Competition was established to provide local AATCC sections an opportunity to contribute original research findings to wet processing literature, and to give the sections a chance to outdo one another in an atmosphere of friendly competition. Papers are judged for originality, scientific value, practical value and presentation. The sections provide the judges whose balloting determines the order of finish in the competition.

In the 1978 competition at Anaheim, Calif., in November, the Northern New England Section won second runnerup honors with the accompanying study on the use of active carbon in permeable polypropylene fabrics for clothing designed to protect workers against toxic vapors. The Delaware Valley Section took first place with its report on how disperse dyes affect certain flammability characteristics of polyester (TCC, December, p18). The Northern Piedmont Section was first runnerup with its study on the use of Karl Fischer reagent to determine the water content of a broad range of textile specialty chemicals (TCC, December, p 27). The Pacific Section placed fourth with a report on the effect surfactants and heat have on the dyeing and physical properties of cotton, nylon and polyester.

attain the highest solids loading (Table I), and maximum carbon tetrachloride sorption after regeneration, per volume of slurry (Table II). It is seen that triethylene glycol (TEG) gave the highest pigment density for all three carbons. With respect to the sorption capacity of the slurries after regeneration, slurries of Amoco PX21, a newly developed petroleum based material, in the triethylene glycol showed the highest sorption capacity per cubic centimeter of slurry by far, and especially so if the absorption measurements were carried out on carbons equilibrated with air of moderate humidity.

Furthermore, studies of the packing factors in the slurries gave a good measure of the accessible void volume between the particles from which maximum potential solids loadings were estimated. At 0.31 g/cm³ solids loading (the maximum achieved), PX21 is near its maximum solids loading and in air gives about 2.5 times the absorption capacity per cubic centimeter of slurry of Barneby-Cheney XZ: i.e., 0.63 g carbon tetrachloride/cm³ for PX 21 vs. 0.24 g carbon tetrachloride/cm³ for XZ.

Both monofilament and multifilament hollow fiber yarns filled with PX21 and TEG slurry were spun. Two types of hollow filament spinnerets were used. These were a monofil spinneret with straight-through flow for the slurry, and a 12-hole spinneret for preparation of the multifilament yarn.

The yarns were extruded at 165-175C down a 15-ft cooling stack at high draw-down ratios (>100:1) typical of usual spinning operations, taken up at 500 to 1000 ft/min, and then drawn 5:1 over a heated draw bar in a separate step. Drawing temperatures of 95C appeared to be satisfactory for the yarns.

The extraction and activation process for the yarns was accomplished with one or two pentane bath extractions before subsequent methanol and pentane steps. The initial pentane treatment, carried out by soaking the yarns in warm pentane (30C) for several hours, extracted the wax from the fiber, making the fiber walls porous. The walls were thus readily permeable to methanol, so that additional soakings in warm methanol (60C) removed the TEG from the slurry. A final extraction in a Soxhlet apparatus permitted contact of the carbon with pure pentane to insure complete desorption of the TEG from the carbon surface. The yarns were then dried at 110C in a vacuum to remove the pentane to complete the carbon reactivation. A photograph of a filament cross-section is shown in Fig. 1.

Preparation Of Fabric

The Kimberly-Clark Corp. recently

Table I—Carrier Fluid Testing of PX and XZ Carbons

Carbon Type	Carrier Fluid Type	Carbon Weight (g)	Carrier Fluid Weight (g)	Slurry Density (g/cm ³)	Slurry Volume (cm ³) ^a	Pigment Density (g/cm ³) ^b
PX21 ^c	TEG ^d	2.7	10	1.27	10.0	0.27
PX21	TEG	2.8	10	1.20	10.7	0.26
PX21	Water	2.6	10	1.13	11.2	0.23
PX21	Freon E3	1.6	10	1.87	6.2	0.26
PX21	Xylene	3.2	10	1.04	12.7	0.25
PX21	LOPS ^e	2.9	10	0.99	13.0	0.22
PX24 ^c	TEG ^d	6.5	10	1.30	12.7	0.51
PX24	Freon E3	3.9	10	1.67	8.3	0.43
PX24	LOPS ^e	7.0	10	1.09	15.6	0.45
PX24	Xylene	4.5	10	1.11	13.1	0.34
BC-XZ ^f	TEG ^d	8.4	10	1.44	12.8	0.66
BC-XZ	Water	8.8	10	1.30	14.5	0.61

^aSlurry volume calculated as (carbon weight + carrier fluid weight)/slurry density. ^bPigment density calculated as carbon weight/slurry volume. ^cAmoco petroleum based active carbon. ^dTriethylene glycol. ^eLOPS = Low odor paraffin solvent (a refined kerosene from Ashland Chemical Co.). ^fBarneby-Cheney coconut charcoal.

Table II—Sorption Capacity of Activated Carbon Dispersions

Carbon	Dispersion Type	Pigment Density (g/cm ³)	Sorption Medium	Sorption Capacity of Powder, Wt. Basis (g CCl ₄ /g)	Sorption Capacity of Dispersion, Vol. Basis (g CCl ₄ /cm ³)
XZ	Bulk Powder	0.45	CCl ₄	0.51	0.23
			Air-CCl ₄ ^a	0.4	0.2
PX21	Bulk Powder	0.30	CCl ₄	0.51	0.33
			Air-CCl ₄ ^a	0.4	0.24
			CCl ₄	2.10	0.63
			Air-CCl ₄ ^a	2.09	0.63
PX24	Bulk Powder	0.31	CCl ₄	2.10	0.63
			Air-CCl ₄ ^a	2.10	0.63
			CCl ₄	—	—
			Air-CCl ₄ ^a	0.69	0.39
PX24	TEG ^b	—	CCl ₄	—	—
			Air-CCl ₄ ^a	—	—
			CCl ₄	—	—

^aThese experiments were carried out on samples dried under vacuum, then tested with the static CCl₄ vapor sorption method.

^bAfter regeneration.

introduced its Evolution II polypropylene nonwoven fabric composite. It is of two-ply construction, consisting of a spunbonded nonwoven fabric layer



Fig. 1. Microporous polypropylene hollow filament filled with active PX21 carbon powder. ×1500.

which provides strength, and a melt-blown layer (microfibers) which provides an extraordinarily high hydrostatic resistance without application of a water resistant finish. The object of laminating active carbon fabric between two sheets of Evolution II was to provide durability and a barrier to the penetration of liquid sweat; i.e., to eliminate or minimize sweat poisoning of the active carbon. Furthermore, such laminates can be thinner than an active carbon-impregnated polyurethane foam laminate (0.08 in. thick) presently in use and, therefore, produce less thermal insulation in garments.

The 3.8 oz/yd² active carbon fabric selected was produced by F.A.P. Maggs (Chemical Defense Establishment, Porton Down, Salisbury, Wilts., United Kingdom) from a woven viscose precursor fabric. It was laminated with spot bonding between two sheets of 1.5 oz/yd² Evolution II with the spunbonded layers acting as facing sheets.

Table III—Active Carbon-Filled Microporous Hollow Multifilament Yarn

Characteristic	
Yarn	10 filaments
Denier (den/filament)	6 (60 total)
Tenacity (g/den)	2.5
Elongation to Break (%)	30
Amoco PX21 Filled (% by wt)	~30
Filament Wall Thickness (mm)	0.04
Carbon Tetrachloride Sorption	
Static Method (% by wt)	49
Dynamic Method (mg/cm ²)	1.0 ^a
	159 mg/2.7 g ^b

^aEstimated for four-layer 2.7 g sample.
^bSample packed in tube.

Further details cannot be given because proprietary processes were used.

Results and Discussion

Activated Carbon-Filled Hollow Fiber

Multifilament sorbent yarns with the properties shown in Table III were produced by this filled hollow fiber approach. The textile properties of the yarns are quite adequate for achieving knitted and woven fabrics.

The sorption results show the sorptive character of these yarns, and, because of the good mechanical properties, durable and comfortable sorptive fabrics from these yarns should be attainable. The hydrophobic nature of the polypropylene sheath should also help resist sweat poisoning.

Laminated Spunbonded/Meltblown (SB/MB) Polypropylene Carbon Fabric

This composite SM/MB polypropylene nonwoven fabric laminate has the properties shown in Table IV. Appreci-

Table IV—Properties of Carbon Fabric Laminates

SB/MB Laminates	Weight (oz/yd ²)	Air Permeability (ft ³ /min/ft ²)	Thickness (in.)	Hydrostatic Resistance (cm)	Dynamic CCl ₄ Vapor Sorption (mg/cm ²)
With carbon fabric	6.2	16	0.045	97	2.2
Without carbon fabric (control)	3.3	29	0.025	45	—

able air permeability and reduced thickness (in comparison with 0.08 in. thickness in presently used active carbon-impregnated polyurethane foam laminates) indicate potentially good garment comfort. Presumably, the hydrostatic resistance indicates good liquid sweat resistance. The CCl₄ vapor sorption level of 2.2 mg/cm² is sufficient for a new protective clothing material, in so far as only 1.2 mg/cm² has been satisfactory in the presently used active carbon impregnated polyurethane foam laminate.

Conclusions

Active carbon powder can be successfully incorporated in yarns of hollow microporous polypropylene filaments suitable for weaving or knitting into sorbent textiles.

Active carbon fabrics can be successfully laminated between spunbonded/meltblown polypropylene nonwoven fabrics to make another sorbent textile system.

Both textile systems offer sorption capacity for garments which can protect workers against toxic chemical vapors.

These garment fabrics might not be subject to sweat poisoning of the active carbon because of their hydrophobic nature.

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