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Activated Carbon-Loaded Rayon Monofilaments

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Introduction

The threat of gas warfare in World War II prompted the development of a textile yarn containing activated carbon for use in gas protective clothing. Through cooperative research at several laboratories a fine filament rayon was made with 25–30% bound carbon.

However, yarn manufacturing costs were high because of: (*1*) expensive grinding of the carbon to less than 5 microns particle size, necessitated by the use of 3 to 5 mil diameter spinning orifices, (*2*) high filtration costs of the carbon–viscose mixture which was necessary to avoid plugging of the spinneret holes by the carbon, and (*3*) yarn processing difficulties engendered by the poisoning of the activated carbon by most textile finishing agents.

Work on the program ceased with the end of the war. There was no foreseeable peacetime use for an activated carbon textile fiber.

Later a patent[1] issued on the incorporation of activated carbon in cellophane.

The present effort originated with the idea to make an odor filter comprising fibers of cellulose-bound activated carbon for light industrial and residential air filtering systems.

The rather unique property of activated carbon—its ability to sorb a variety of gases and vapors—has not been exploited on a large scale in domestic air filtration for several reasons. In its most economical form, as a solid bed, activated carbon presents too much resistance to air flow. Residential air-moving systems permit a maximum pressure drop of 0.2 in. of water for air velocities of 300 ft./min. A 0.75-in. bed of carbon, 8 to 14 mesh (Tyler scale), develops 0.2 in. water pressure at 35 ft./min. air flow. At higher flow rates the pressure drop increases exponentially.

Attempts to avoid the pressure drop difficulty, through bonding the carbon to some type of perforated or open framework structure have been more or less successful. The problems with this approach include selecting a suitable binder and designing a suitable substrate for the carbon. The adhesive used must not poison the activated carbon, and the supporting framework must be open enough to allow a free air path and yet have an adequate surface area to permit high carbon loading. High carbon content is necessary to give a reasonably useful life to the filter.

The objectives of high carbon content, low pressure drop, and low manufacturing cost rule out the type of fine textile yarn spun during the war. The low denier (10–25 micron diameter) textile filaments, like solid bed carbon, cause too high a pressure drop in gas streams. The objectives mentioned above required a monofilament of the order of 500–1500 den. or $^{1}/_{64}$–$^{3}/_{64}$ in. diameter containing about 80% activated carbon.

Experimental: Preparation of Monofilaments Containing Activated Carbon

The preparation of the monofilaments involves obtaining a stable, aqueous dispersion of activated carbon which can be injected into the spinning dope (viscose) just prior to extrusion and spinning and processing the monofilament into a filter package ready for use.

1. Activated Carbon Dispersion. The aqueous, activated carbon dispersion contains 10–25% activated carbon. The particle size of the carbon should be at least 100% less than 70 microns. Thickening agents, such as carboxymethyl cellulose and sodium alginate, are used to keep the carbon uniformly suspended. They are added at the 1 to 2% level based on the dispersion weight.

2. Spinning and Processing the Monofilaments. The activated carbon dispersion is metered and mixed into the viscose stream after the viscose has been filtered and deaerated. At this point, the spinning departs from a normal viscose spinning operation, for if one extrudes a filament con-

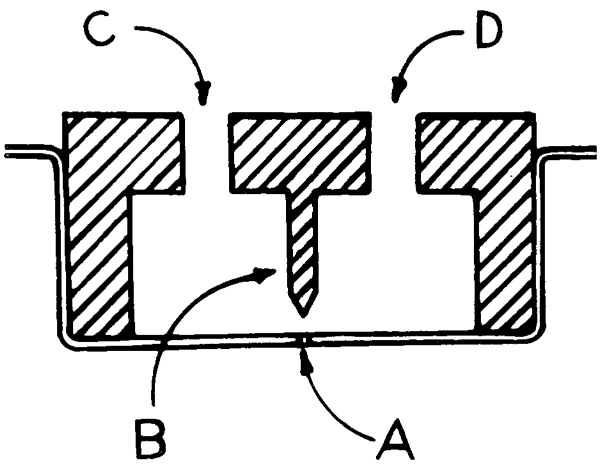

Fig. 1. Schematic diagram of conjugate jet (cross section).

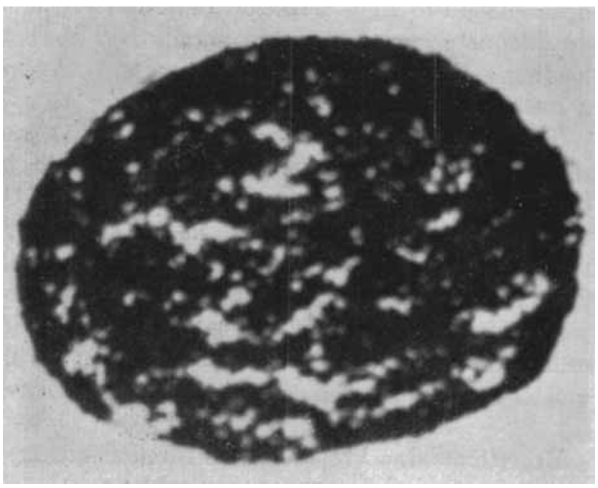

Fig. 2. Normal filament containing 80% activated carbon. 90×.

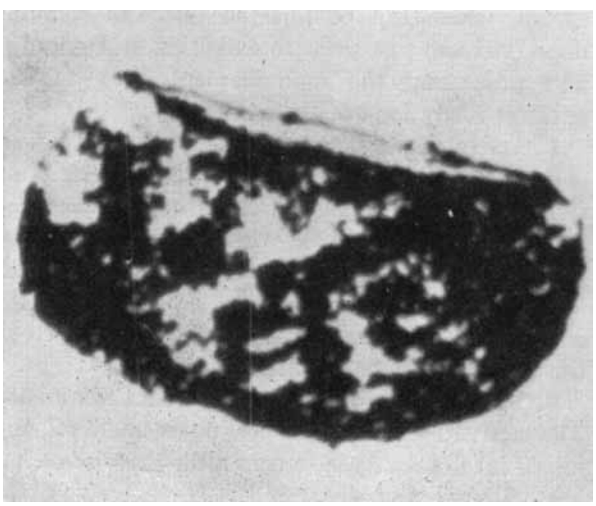

Fig. 3. Conjugate filament containing 80% activated carbon. 90×.

taining large amounts of carbon (70–80%) under normal conditions,[2] a brittle filament results which must be handled carefully to avoid breakage. To reinforce the filament, a pure cellulose backbone is added.[3] This is accomplished by using a conjugate jet,[4] a cross section of which is shown schematically in Figure 1. The carbon-loaded viscose stream flows through the channel C to the jet hole A. A plain viscose stream flows through channel D to the jet hole A. A divider or septum B separates the two streams until they join in the jet hole. The effect is shown dramatically in the cross-section photomicrographs of the filaments.

Figure 2 shows a normal filament containing 80% activated carbon. With the exception of void spaces the cross section is uniform. The origin of the voids is indeterminate. Undoubtedly some arise from cross section preparation; others may be due to stresses set up during drying of the monofilament.

Figure 3 shows a conjugate filament containing 80% activated carbon. Examination reveals the backbone of pure cellulose. It has collapsed on deswelling and drying as would a plain rayon filament. The carbon-loaded portion, because of the restraint imposed by the carbon, does not shrink.

Figure 4 shows a concentric type of composite filament. The core of plain cellulose appears crescent-shaped and separates from the outer ring, leaving a large internal void that apparently cracks through to the surface. The ring-core filament because of this is fragile. The side-by-side structure (Fig. 3) is tougher and permits

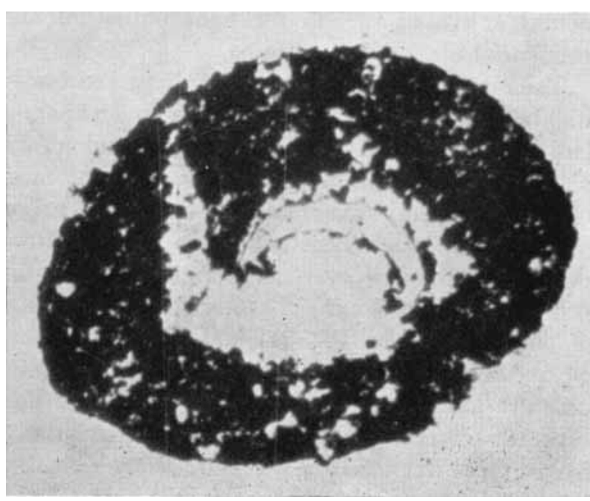

Fig. 4. Concentric type of composite filament. 90×.

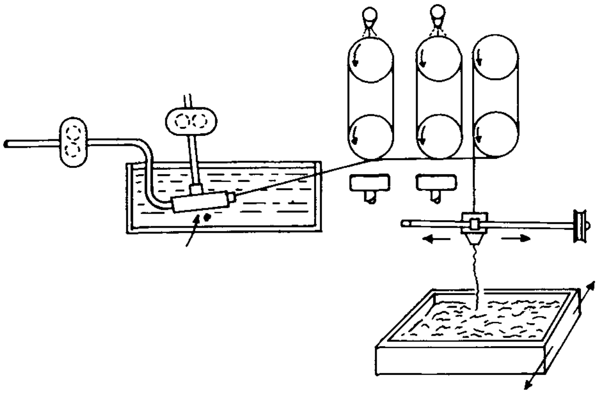

Fig. 5. Apparatus for spinning and processing the monofilaments.

easier processing; it is the type discussed throughout the rest of the paper.

After extrusion into a conventional spin bath (see Fig. 5) the monofilament passes over a series of rotating, thread-advancing reels where it is washed thoroughly with hot water. It is partially dried on heated reels and then dropped into a filter frame via a double traversing mechanism which lays the filament down in a random manner. Standard filter frames are used to collect the monofilaments; after collection, the filter package is dried further in a hot air oven.

Results: Properties of the Monofilaments containing Activated Carbon

The gas or odor sorptive properties of the monofilaments depend on whether or not the filament is conjugated, on the extent of loading and on the nature of the carbon.

1. Effect of Conjugation on CCl_4 Sorption. The activity or capacity of an activated carbon is defined as the weight per cent of CCl_4 that a particular sample can sorb under standard conditions. The activity is measured by passing clean, dry air saturated with CCl_4 at 0°C. over a fixed weight of carbon at 25°C. until the sample no longer increases in weight. Table I shows the capacities of conjugated and normal monofilaments. The conjugated samples are superior and approach the activity of "raw" carbon.

Presumably the thinner film of cellulose covering the carbon in the conjugate samples renders the carbon more accessible to the CCl_4 gas.

The same apparatus, fitted with a halide ion detector is used to calculate the gas adsorption rate. The CCl_4 concentration in the inlet air is held constant, and the concentration of CCl_4 in the exit air is measured with time. Plotting the ratio of the concentration against time produces a sigmoidal adsorption curve. For simplicity, the time for the exit air to reach a CCl_4 concentration of 10% of that in the inlet air is used as a comparative measure of adsorption rate. This is shown in Table I as the 10% break time. Again, the sorption rates of the conjugated filaments are higher. Thus, in addition to easier filament handling, conjugation results in much improved sorption performance.

TABLE I
CCl_4 Gas Adsorptivity of Conjugated vs. Regular Monofilaments

	Carbon, %	CCl_4 Capacity, %[a]	10% Break time, sec.[b]
Regular	82.9	49.4	111
Conjugated			
Side by side	82.4	58.0	358
Concentric	84.3	62.0	375
"Raw" carbon	100	65.1	380

[a] Equilibrium CCl_4 adsorption, wt.-% based on carbon content.
[b] Time for CCl_4 content of effluent gas stream to equal 10% of that in the inlet gas stream.

2. Effect of Carbon Concentration on CCl_4 Sorption. Table II shows the effect of carbon concentration. As the carbon content increases, higher capacities and sorption rates are noticed. This again is a matter of the carbon's greater availability to the gas as the cellulose film around the carbon becomes thinner. The 80–85% level of carbon represents the maximum practical range of concentration consistent with processing and good mechanical properties.

The third factor affecting adsorptivity of gases is the carbon type. The sorptivity of a particular carbon is carried over into the monofilament. Carbons designed for gas sorption show approxi-

TABLE II
Effect of Carbon Concentration in Monofilament on CCl_4 Gas Adsorptivity

Carbon in Monofil, %	CCl_4 Capacity, %	10% Break time, sec.
35	37	210
60	53	325
80	60	375
100 (raw carbon)	62	450

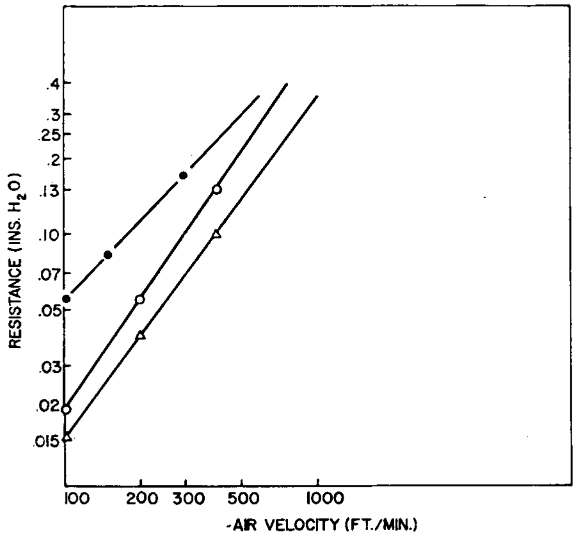

Fig. 6. Air resistance of carbon monofilament rayon filters: (●) 0.50 lb. activated carbon-loaded monofilaments; (O) 0.33 lb. activated carbon-loaded monofilaments; (△) glass wool dust filter. Filters are 12 × 12 × 1 in.

mately twice the adsorptive capacity and rate of the "powdered" carbons. The gas carbons in general have a smaller and more uniform pore size, and are more abrasion resistant, and have a higher density. The powdered carbons are used chiefly in municipal water purification systems and in other liquid phase applications.

The carbons used in this work are coal-derived gas carbons from the Pittsburgh Chemical Company, to whom we are indebted for the adsorption data shown.

It is difficult to extrapolate from laboratory data to actual filter use condition. An "average" odor parameter for residences is rather difficult to obtain. However, in another paper in this symposium, Turk[5] has discussed his findings on the performance of carbon-loaded monofilaments.

3. Air Resistance of Activated Carbon-Loaded Monofilament Filters. The resistance of filters made with carbon loaded monofilaments is dependent on packing density. This is shown in Figure 6, in which air resistance is plotted against air velocity for filters packed to 0.33 and 0.5 lb. of monofilament/ft.^{2} of area and 1 in. of depth. A commercial glass wool dust stop serves as a comparison. Thus, 0.5 lb. of monofilament containing 80% carbon meets the air resistance requirement for home use.

Conclusion

It is believed that this product will find many important end uses in air purification. For the first time activated carbon can be presented to air streams in a nondusting form, in reasonable quantities, with unimpaired activity, and with low resistance to gas flow.

References

1. Cornwell, R. T. K., E. Dunn, and A. W. Hunter (American Viscose Corp.), U. S. Pat. 2,574,533 (Nov. 11, 1951).
2. Braunlich, R. H. (American Viscose Corp.), U. S. Pat. 2,923,378 (Feb. 2, 1960).
3. McDowell, R. L. (American Viscose Corp.), patents pending.
4. Kulp, M., F. F. Morehead, W. A. Sisson, and W. Webb (American Viscose Corp.), U. S. Pat. 2,386,173 (Oct. 2, 1945).
5. Turk, A., and A. Berlin, *J. Appl. Polymer Sci.*, **5**, 668 (1961).

Synopsis

Rayon monofilaments containing 80% activated carbon were prepared. They are an effective medium for adsorbing noxious odors. High grade gas-adsorbing carbons are intimately bound in a cellulose matrix by mixing the carbon in viscose (sodium cellulose xanthate solution) and extruding the mixture through a special conjugate jet as monofilaments into a rayon coagulating bath. The special jet permits a backbone of plain viscose to be spun as a spine support for the carbon-loaded portion of the monofilament to give it added strength. The diameters of the dry, nondusting monofils can be varied greatly. For air cleaning purposes the nonfilaments range from $^1/_{64}$ to $^3/_{64}$ in. in diameter. Although imbedded in cellulose, the carbon's capacity to remove malodors remains high. In addition, when made into a filter unit, the resistance to air flow through the unit is low. The novel use of regenerated cellulose as a carrier for large quantities of activated carbon indicates that other materials (catalysts, ion exchange resins, etc.) can be similarly bound in cellulose and still retain a high level of their original performance.

Résumé

On a préparé des monofilaments de rayonne renfermant, 80% de carbone actif. C'est un moyen efficace pour adsorber les odeurs nuisibles. Les carbones adsorbant des teneurs élevées de gaz sont intimement liés à la matrice cellulosique en mélangeant le carbone à la viscose (solution de xanthate de cellulose sodé) et en extrudant le mélange sous forme de monofilaments dans un bain coagulant la rayonne. Ce filage spécial permet de filer une chaîne de viscose ordinaire comme support pour la partie chargée au carbone du monofilament, ce qui lui donne une solidité accrue. Les diamètres des monofils sèchés et dépoussièrés peuvent varier dans de grandes proportions. Pour la purification de l'air les monofilaments ont un diamètre de $^1/_{64}$ à $^3/_{64}$ de pouce. Bien qu'absorbés dans la cellulose, la capacité du carbone à adsorber les mauvaises odeurs demeure élevée. De plus lorsqu'on fait passer l'air dans ce matériau sous forme de filtre, la résistance au courant d'air à travers cette unité est faible. Le nouvel emploi de la cellulose régénérée, porteuse de grandes quantités de carbone activé, montre que d'autres substances (catalyseurs, résines échangeuses d'ions, etc.) peuvent de

même être liées à la cellulose sans en modifier grandement les propriétes originales.

Zusammenfassung

Rayon-Einzelfasern mit einem Gehalt an Aktivkohle von 80% wurden dargestellt. Sie bilden ein wirksames Medium zur Adsorption übler Gerüche. Durch Vermischen der Kohle mit Viskose (Lösung von Natriumcellulosexanthogenat) und Extrusion der Mischung durch eine Spezialzwillingsdüse als Einzelfasern in ein Rayonfällbad werden hochaktive Adsorptionskohlen fest in eine Cellulosematrix gebunden. Die Spezialdüse erlaubt das Spinnen eines normalen Viskosefadens als Träger um dem kohlehältigen Teil der Einzelfaser zusätzliche Festigkeit zu verleihen. Der Durchmesser der trockenen, staubfreien Einzelfäden kann stark variiert werden. Für Luftreinigungszwecke liegt der Durchmesser der Einzelfäden in Bereich von $^1/_{64}$ bis $^3/_{64}$ inch. Trotz der Einbettung in Cellulose behält die Kohle eine hohe Kapazität zur Entfernung übler Gerüche. Ausserdem besitzen daraus hergestellte Filtereinheiten einen niedrigen Widerstand gegen das Durchströmen von Luft. Die neuartige Verwendung von regenerierter Cellulose als Träger für grosse Mengen von Aktivkohle lässt erwarten, dass andere Materialien (Katalysatoren, Ionenaustauscherharze etc.) in ähnlicher Weise unter Beibehaltung eines hohen Anteils ihrer ursprünglichen Fähigkeit an Cellulose gebunden werden kann.