

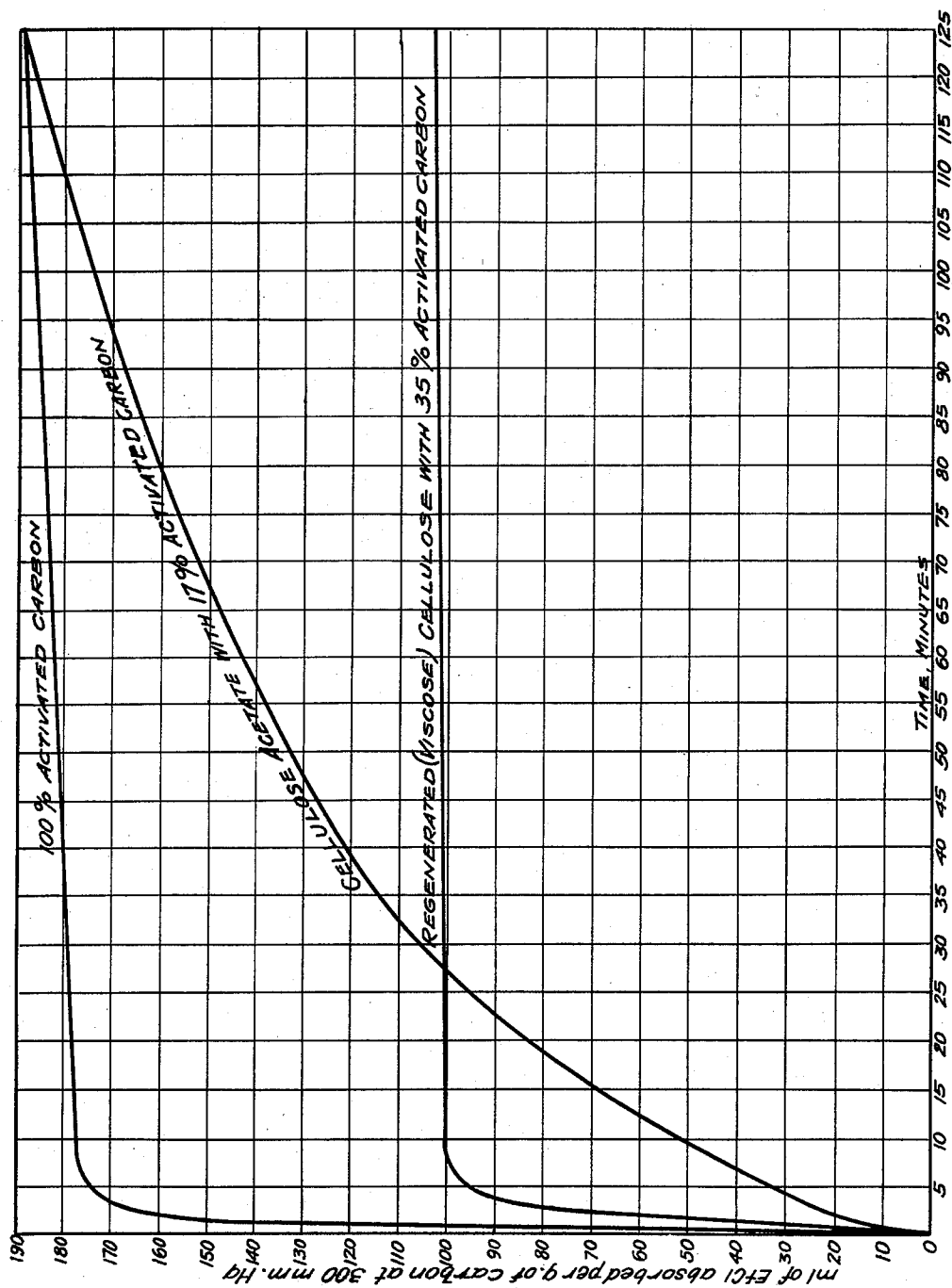
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FILTER MEDIUM

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1

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FILTER MEDIUM

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1 Claim. (Cl. 183—4)

The invention described herein may be manufactured and used by or for the Government, for governmental purposes, without payment to us of any royalty thereon.

This invention relates generally to an adsorbent filter medium useful for filtering and treating fluids. More particularly, it relates to a method of preparing extruded filaments containing an efficient adsorption agent, such as activated carbon or charcoal, in such a manner that a fabric woven from the threads or yarns of the filaments is effective in removing vapor contaminants from air filtered through the fabric.

An object of this invention is to provide artificial films and filaments having incorporated integrally a finely divided adsorbent in suitable amount so that the films or filaments may be employed in forming a filtering medium or membrane of adequate structural strength.

Another object is to provide an adsorbent fabric capable of completely removing physiologically harmful vapor contaminants from air while the air permeates through the fabric, so that the fabric may be made into a protective clothing for affording protection against vesicant vapors without giving discomfort to the wearer. Also further objects will become apparent from the following description:

In the manufacture of artificial filaments for use in various textiles, a spinning solution is extruded through small orifices into a coagulant. The composition of the spinning solution depends upon the type of filament to be made, for instance, viscose (cellulose xanthate), cellulose acetate, vinyl polymers, and various other filament and film forming resinous polymers, which in general are of the linear type. The coagulant will be the special one required for each spinning solution, e.g. an acid bath is used for viscose in wet spinning and air for dry spinning of thermoplastic ester polymers. It is known that methods have been developed for incorporating certain materials in the spinning solution or coagulating liquid and in various other steps in the processes for manufacturing fabrics and films. In general these foreign materials are introduced in very small amounts to the extent of about 1% of the weight of thread for modifying the finish, as for example, in delustering. It will become readily apparent that the present invention is not concerned primarily with the ordinary qualities of a textile such as its appearance, finish or feel but is concerned with a method of incorporating finely divided adsorbent particles into a plastic formed into a fabric uniformly effective over its breadth as a filtering medium, yet having the desirable characteristics of a cloth fabric with reference to flexibility, wearing qualities, strength and permeability.

The manner in which the objects of this invention are accomplished is demonstrated by the following examples, which are not intended to be limiting. The first example illustrates how an adsorbent is used with a viscose solution, which is subjected to wet spinning or wet coagulation, and the second example illustrates how the ad-

2

sorbent is used with an ester or thermoplastic resin forming substance.

Finely ground activated carbon or charcoal having a colloidal particle size in the neighborhood of one or several microns preferably an average size less than three microns, is mixed with a viscose solution, with or without the aid of dispersing or wetting agents (such agents not being required), the amount of carbon so used varying from about 10% up to the largest quantities which can be incorporated without destroying the yarn properties, i.e., about 50%, the more effective amounts having been found to be above 20% by weight on the basis of the dry cellulose in the spun filaments.

It has been found expedient to grind the carbon by a dry grinding process known as "micronizing"; and the suspension obtained with the powdered active carbon thus formed has been found to be extremely stable and efficient in the subsequent spinning operation. A viscose suspension thus obtained may be spun in the usual manner.

By exercising precautions during spinning and finishing of the viscose suspension so as not to introduce foreign substances which would poison the active carbon, the resultant filaments, threads, and yarns obtained after a usual viscose desulfiding and finishing process is found to retain in the active carbon all or nearly all of its original activity. The threads or yarns may be used in all normal textile operations, and the woven fabric, felt or other articles made from such thread or yarn possess adsorption properties comparable to the adsorption properties of the pure active carbon on the active carbon weight basis.

A typical example of a satisfactory protective cloth was prepared under the following conditions:

(1) An activated charcoal of a type prepared for use in gas masks was pulverized until in the powder obtained, 90% of the carbon particles were one to 3 microns in size. This powdered activated carbon in an amount calculated to be 35% by weight of the dry cellulose, was mixed in a viscose solution containing 7.7% cellulose and 6.7% caustic soda (NaOH), the incorporation being effected without the use of assisting agents in a high speed mixer. The further customary steps of aging, treatment with carbon bisulfide, dissolving in caustic and ripening were performed on the mixture to obtain a viscose solution having the desirable spinning maturity changes before the spinning operation. The resulting solution may be filtered through usual filtration apparatus employed without substantial removal of the suspended carbon.

(2) The spinning took place in a wetting or coagulant bath comprised of 11% sulfuric acid, 16% sodium sulfide, 1% zinc sulfate, and several parts per million of wetting agent, although it was not found material to have the wetting agent present. An ordinary spinneret and spinning velocity were used. The filament size was 1.25 denier. The filament size has been found to be extremely important and the effectiveness of the retained active carbon is closely related thereto. The finer the filament size the higher the activity.

(3) The crude yarn obtained was subsequently washed free of acid and salt, desulfided in the usual sodium sulfide solution, about 0.6%, subsequently washed free of sulfide, acidified by sulfuric acid, and washed again.

No soap or finishing agents usually employed in the manufacture of rayon filaments were used, since it was considered that they may have a harmful effect.

Where it is deemed necessary to incorporate a finishing agent to act as a lubricant, ethylene glycol or other glycols, glycerine or similar simple chemical compounds rather than inorganic soaps have been employed. Min-

eral oil has been found satisfactory, particularly because it can be subsequently readily removed.

Yarn made in accordance with the foregoing procedure was quilled and woven on a standard box loom into a fabric as filling. No lubricant or other foreign materials were used in the weaving operation. Samples of the adsorbent yarn were woven and knit by many of the usual methods into various articles. The yarns were found to process by all the textile methods. In the preparation of a fabric having a high strength and particularly favorable weaving construction the face of the fabric showed mainly the protective adsorbent black yarn used as filling, and the backing of the fabric was mainly an appropriate amount of interwoven cotton yarn.

This fabric had the following approximate specifications:

Weight of fabric.....	0.21 g./sq. in.
Weight of activated carbon....	0.029 g./sq. in.
Picks/inch.....	36.
Size of cotton in filling.....	36 p-16 coned filling.
Size of rayon in filling.....	1000 denier, 35% activated carbon black yarn.
Sley.....	108.
Size of cotton in warp.....	23 coned yarn.

Physical data was obtained on the fabric using recognized test methods. The results of these tests at 70° F.-65% R.H. are as follows:

Abrasion test.—The carbon fiber side of the samples abraded against standard abrasion cloth withstood abrasion for 1400 to 1460 revolutions.

Tensile strength:

By the strip method—	Lbs. per inch
Average warp.....	92
Average fill.....	37
Grab method—	Lbs.
Average warp.....	123
Average fill.....	52

Tear strength:

ASTM trapezoid method D39—	Lbs.
Average warp.....	19.0
Average fill.....	12.2

Air permeability.—Average of 82.5 cubic feet of air passed per minute per square foot fabric at 0.5 in. water pressure differential.

The described fabric was tested for mustard vapor resistance by standardized dynamic tests in the "as received" condition, and after being made into jackets and worn by personnel for 144 hours.

Cloth cut from the arm pits, backs, and fronts of the worn jackets were also tested for mustard resistance after a dry cleaning treatment of 5 extractions with acetone in a Soxhlet extractor, after laundering in accordance with the Quartermaster mobile formula using low titre wool soap. In the dynamic test method, air saturated with mustard (dichlorethyl sulfide) vapor was passed at the rate of 400 cc. per minute through the test specimen of the fabric cut out as a disc 3 inches in diameter. The results of all the mustard vapor resistance tests are summarized as follows:

Although there were considerable variations in results on the different jackets after the various treatments, as was to be expected, the greatest average activity loss was 3% in the jackets laundered by the Q.M. formula using low titre wool soap. Practically no change in mustard vapor resistance occurred in 144 hours of wear. The garments laundered in accordance with the Q.M. formula using the Nacconol detergent showed gains in activity up 34% and those treated with acetone after 144 hours of wear showed gains in absorbent activity up to 20%.

From the results of the tests, it is concluded that the adsorbent fabric can be subjected to a wide variety of treatments, including wear, laundering and organic solvent dry cleaning, without any substantial loss of

adsorption activity. In general, over 80% of the total original activity of the carbon was preserved.

Evidently the filaments of the adsorbent yarn are built up of bundles of cellulose molecules joined together end to end in chains containing a number of cellulose molecules, and the active adsorbent is bonded to and set in these bundles as a permanent part thereof so it cannot be rubbed out or washed off.

The fabric made with the adsorbent yarn may have any desired type of reinforcing threads including cotton, rayon, or even chemically resistant filament such as spun glass, metal wire, asbestos, and the like. Accordingly, adsorbent fabrics may be made for utilization in various chemical processes where it is desired to have the benefits of a finely divided adsorbent retained integrally in a fabric. Such a fabric, for example, may be made in the form of an endless belt to be passed cyclicly through an adsorption zone and a desorption zone. Such a fabric may be used also as a fixed filtering membrane.

Use may also be made of the viscose cellulose suspension of the activated adsorbent for preparing adsorbent films by the usual methods of extrusion or casting. Similar films were prepared with suspensions of activated carbon in cellulose acetate, and in solutions of copolymers or vinyl chloride and vinyl acetate by evaporation of their respective solvents, e.g., acetone. These films were found to retain high adsorption values of the activated carbon. Threads were prepared by extruding the solutions into water, and similarly, high adsorption values were obtained.

One method of determining the relative adsorption values for the various adsorbent plastic compositions consists in determining the amount of ethyl chloride adsorbed over various periods by a given weight of active adsorbent in the adsorbent plastic as compared to the adsorption value of the adsorbent per se. Results of such tests are illustrated in the accompanying drawing, which shows the adsorption characteristics of the micronized activated carbon by itself as compared to the values in cellulose acetate and in viscose cellulose. The curves indicate that the adsorbent cellulose acetate combination has some very interesting and useful properties in that substantially all the activity of the carbon is retained, although the adsorption proceeds more slowly. The sample of the adsorbent viscose rayon containing 35% of activated carbon is shown to retain more than 50% of the adsorption value of the activated carbon.

From tests on varieties of adsorbent plastics that were prepared, it is indicated that the preferred types of plastic forming materials to be used are of the thermoplastic class characterized by high polymer substances having long chain structures, and which are suitable for extrusion or injection molding. The incorporation of the finely divided adsorbents into the plastics, unlike the use of fillers is made in such a manner as to have a large amount of surface area of the adsorbent exposed to gases or fluids which are able to permeate into the adsorbent plastic so as to retain as much as possible of the adsorption value of the finely divided adsorbent.

Among other types of plastic forming substances to be used in making the adsorbent plastic may be mentioned acrylic esters, such as methyl methacrylate; long chain amino acids, or diamines with dibasic acids, such as in nylon polyamides; and others depending upon the purpose of the adsorbent plastic. Various other adsorbents may be used, as for example, finely divided silica gel, activated aluminum, and similar effective adsorbents.

The adsorbent plastics may be set into various shapes or forms, as for example, filaments, foils, films, sheets, granules, beads, tubing, etc. It is important to remember, however, that the higher adsorption activity is obtained with greater surface exposure; therefore, in a final hardened form the adsorbent plastics are preferably thin porous conglomerates. In these various shapes and forms the adsorbent plastics may be used in purifying liquids,

5

gases, or vapors, with simplification in the removal or separation of the finely divided adsorbent as an integral part of the plastic from the treated fluid. In certain treatments the adsorbent plastic may function as a catalytic agent and in others it may be considered as an absorption agent. Also, the porous plastic may serve as a semi-permeable dialyzing membrane which selectively allows one fluid substance to diffuse more readily than another, and thus bring about a separation of the mixed fluid substances. The finely divided adsorbents may be activated or be impregnated with an adsorption or a catalytic promoting agent before dispersion in a plastic forming liquid or after setting of the adsorbent plastic. The adsorbent plastics formed by spinning, extruding, or molding are herein described as having a self-sustaining form in that the plastic is formed without any supporting base in contradistinction to impregnated and coated materials.

The invention has been described with reference to certain illustrative examples, but it is to be understood that modifications come within the spirit and scope thereof.

We claim:

6

A protective fabric consisting essentially of textile fibers spun from a viscose solution having suspended therein more than 10% by weight of activated carbon having an average particle size of less than three microns.

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