Nov. 5, 1968 C. E. WESTBROOK, JR., ET AL 3,409,020

TOBACCO SMOKE FILTER

Filed Feb. 24, 1965

FIG. 1

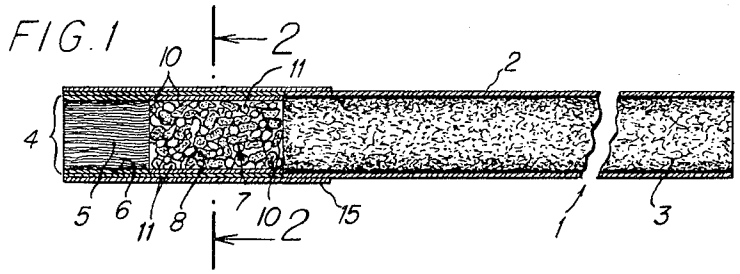


FIG. 2

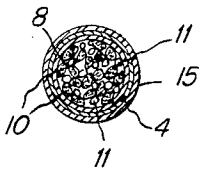


FIG. 3

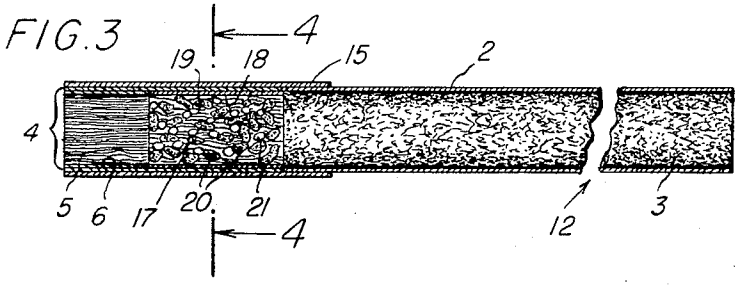


FIG. 4

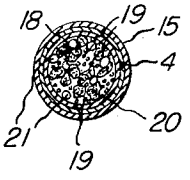


FIG. 5

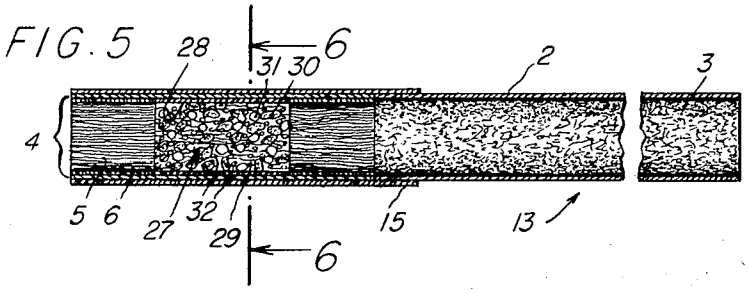


FIG. 6

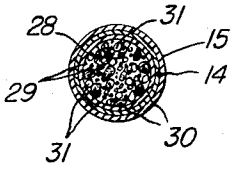


FIG. 7

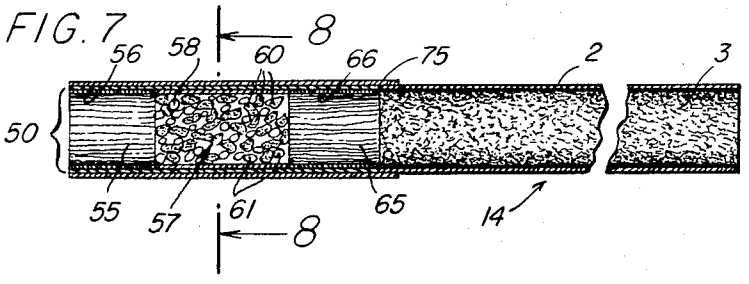
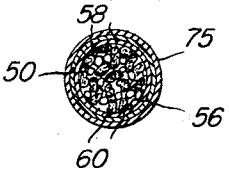


FIG. 8



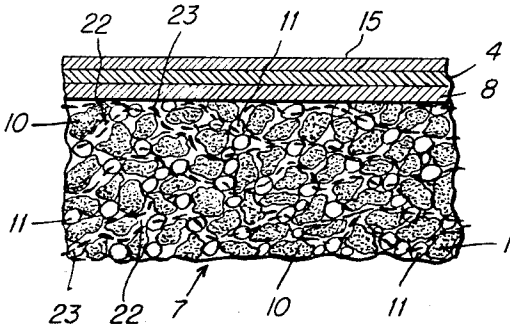


FIG. 9

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3,409,020

TOBACCO SMOKE FILTER

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ABSTRACT OF THE DISCLOSURE

This disclosure relates to a tobacco smoke filter comprising particles of activated carbon which are fused together with polyalkylene oxide particles, such as polyethylene oxide particles.

This invention relates to a tobacco smoke filter. More particularly, the present invention relates to a tobacco smoke filter comprising particles of activated carbon fused with particles of a polyalkylene glycol.

Synthetic fibers, for example, cellulose esters, have found widespread use in tobacco smoke filters due to the ease with which they can be manufactured into filter rods on standard cigarette manufacturing equipment. These cellulose esters are generally employed in the form of crimped, continuous fibers or filaments. Filters made of cellulose acetate fibers function, in general, by removing a portion of the particulate matter from the smoke which passes through the fibers. The crimping or other physical positioning of the fibers within the filter serves to increase the surface area of the filaments which come in contact with the smoke. However, cellulose acetate filters do not remove any significant amounts of undesirable gas phase constituents from the tobacco smoke.

Some materials have also been applied to cellulose acetate filters in order to make the cellulose acetate fibers more effective in picking up the particulate matter in the smoke. Such materials may, for example, be starch, ground cellulose or the like, and are generally held in place by an adhesive or plasticizer such as dimethoxyethylphthalate, methylphthalylethylglycollate or triacetin. However, these materials do not materially alter the failure of the fibers to remove undesirable gas phase constituents from the smoke.

Other materials which are, in effect, self-bonding materials have been suggested for incorporation in synthetic fibers to be used in tobacco smoke filters. These materials include, for example, certain types of wax particles, such as paraffin or polyethylene wax, and certain sugar esters, such as sucrose acetate propionate. However, filters containing such additives also fail to remove any substantial amount of the undesirable elements which are present in the gas phase of the tobacco smoke.

Certain adsorbent particles, as exemplified by activated carbon, have also been employed in tobacco smoke filters. Such adsorbent particles have been employed in conjunction with crimped paper and serve to increase the effectiveness of such filters for removing the particulate matter in the smoke, as well as functioning to remove undesirable gas phase constitutents from the smoke. However, when activated carbon has been used in this manner, it has been employed in the form of very fine particles or dust, because it had been found that larger carbon particles are not completely satisfactory when incorporated with the paper. The larger particles have been found to be virtually impossible to employ in paper-type filters because they tend to tear and misshape the filters and to migrate from the filter during manufacture and storage.

Since paper-type filters have generally been found to be less efficient than synthetic fiber or tow-type filters,

activated carbon particles have also been suggested for incorporation in synthetic filaments to form tobacco smoke filters. However, it has been generally recognized that the particles of carbon will not by themselves adhere well to the synthetic fibers in the filter and adhesives such as triacetin, dimethoxyethylphthalate or methylphthalylethylglycollate have been employed to hold the particles on the fibers. Such adhesives have been found to poison or inactivate the activated carbon to a considerable extent.

Some of the disadvantages of the incorporating carbon particles in synthetic filaments have been overcome by the use of an improved adhesive mixture comprising polyvinyl pyrrolidone and a polyhydric alcohol, such as glycerol. Such an adhesive has made it possible to employ carbon particles over a wider range of sizes than had previously been possible. However, each such an improved adhesive has had only limited effectiveness.

Larger particles of carbon have been used in a separate filter section comprised solely of the carbon particles and this section has been placed between two noncarbon containing filter sections. This type of filter has an advantage over the previous filters in that the carbon particles need not be contacted with an adhesive to hold them on paper or tow. However, such filters have been found to create a different problem, in that the loose carbon particles tend to migrate from the section in which they are placed to other parts of the cigarette. Furthermore, this type of filter has been found to have the disadvantage that the carbon particles do no contact all of the tobacco smoke to the same degree. In such a filter, there is some settling of the carbon particles within the carbon-containing section of the filter during storage and transit of the cigarettes, creating an open space above the carbon. There is also a considerable amount of empty space around each of the carbon particles. Thus, a substantial percentage of the tobacco smoke, following a path of least resistance, can pass through these empty spaces in the filter without any significant contact between the smoke and the carbon. As will be more fully set forth below, the present invention overcomes this disadvantage and provides for intimate contact between substantially all of the tobacco smoke and the carbon particles.

Filters have also been made with adhesives to adhere the carbon particles together to form a disk or plug composed of carbon particles which are bonded together with a polyethylene resin. While such construction tends to overcome the problem of carbon particle migration, the polyethylene in such filters has been found to at least partially deactivate the carbon.

We have unexpectedly discovered that outstandingly superior results can be obtained in the filtration of tobacco smoke by employing a filter comprising activated carbon particles which are bonded to particles of polyalkylene oxide.

It is an object of the present invention to provide an improved filter for tobacco smoke.

It is a further object of the present invention to provide an improved filter for cigarettes, cigars, cigarillos, cigaritos and the like, said filter having particles of activated carbon dispersed therein in such a manner that the tobacco smoke is intimately contacted with said particles of activated carbon.

It is another object of the present invention to provide a tobacco smoke filter wherein activated carbon particles are maintained in place in the filter in a substantially non-poisoned state.

A further object of the present invention is to provide a filter containing a material which holds particles of activated carbon in place within the filter but which does not mask or otherwise deactivate the activated carbon.

It is a still further object of the present invention to provide a filter ingredient which not only holds the acti-

vated carbon in place but which, itself, effects the selective removal of certain constituents from the tobacco smoke.

It is another object of the present invention to provide a filter which provides a cooler smoke than would otherwise be obtained.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

Accordingly, the present invention comprises a filter for tobacco smoke wherein particles of activated carbon are held in place in the filter by being fused or bonded with particles or a polyalkylene oxide, wherein the polyalkylene oxide, itself, functions as an active filtering agent.

While it is possible to employ the polyalkylene oxide particles to not only bond carbon particles to the polyalkylene oxide particles, themselves, but also to hold carbon particles to paper-type or to tow-type filters, the present invention is particularly directed to an especially effective filter comprised of carbon particles bonded to polyalkylene oxide particles. Although the preferred filter may be used in conjunction with paper-type or tow-type filters, it does not itself contain any paper or tow. In such a preferred filter, the polyalkylene oxide particles, which will be described in more detail hereinafter, combine with the particles of activated carbon to form a rigid structure which is capable of high adsorption of the undesirable gas phase elements of tobacco smoke as well as high removal of the particulate matter of the smoke without the losses or inefficiencies which have generally been involved with other tobacco smoke filters.

By employing the filter of the present invention, the carbon particles are held by the polyalkylene oxide in a desired configuration within a filter structure for smoking articles, such as cigarettes, and can provide for intimate and uniform contact by the tobacco smoke with both the carbon particles and the polyalkylene oxide in the filter element. The configuration of the filter of the present invention is such that the carbon particles are not poisoned or deactivated by the presence of the polyalkylene oxide. In addition, the polyalkylene oxide serves itself to remove undesirable elements from the tobacco smoke. Furthermore, the filter of the present invention does not have any significant amount of undesirable taste or aroma imparted thereto by the ingredients of the filter.

A further important advantage of the present filter is the reduction which can be obtained in smoke temperature so that the present filter can provide a smoke exit temperature which is only slightly above room temperature and therefore more desirable to the smoker.

Thus, the polyalkylene oxide particles serve to selectively remove certain gaseous phase components from the tobacco smoke and to provide an additional degree of cooling to the tobacco smoke as well as to bind the carbon particles together or to a carbon or tow base without adversely affecting the character or the quality of the smoke which passes through the filter and without adversely affecting the ability of the carbon itself to adsorb or absorb the undesirable constituents of the tobacco smoke. In addition, the polyalkylene oxide particles are compatible with materials generally employed in filter manufacture and have no objectionable taste or odor.

For a further understanding of the present invention, reference may be made to the attached drawing, which forms a part of the present application.

In the drawing, FIG. 1 is a longitudinal sectional view of a cigarette having a filter which is a preferred form of the present invention.

FIG. 2 is a cross-sectional view of the cigarette shown in FIG. 1, taken along the lines **2—2**.

FIG. 3 is a longitudinal sectional view of a modification of the present invention.

FIG. 4 is a cross-sectional view of the cigarette shown in FIG. 3, taken along the lines **4—4**.

FIG. 5 is a longitudinal sectional view of a further modification of the present invention.

FIG. 6 is a cross-sectional view of the cigarette shown in FIG. 5, taken along the lines **6—6**.

FIG. 7 is a longitudinal sectional view of an additional modification of the present invention.

FIG. 8 is a cross-sectional view of the cigarette shown in FIG. 7, taken along the lines **8—8**.

FIG. 9 is a magnified view of a portion of the cigarette shown in FIGS. 1 and 2, showing in greater detail the body of filtering material prepared in accordance with the preferred form of the present invention.

Referring further to FIGS. 1 and 2, there is shown a cigarette **1** having a paper cylinder **2**. Shredded tobacco **3** is contained within paper cylinder **2**. Paper cylinder **4** is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder **2**. Paper cylinder **4** generally defines the filter unit of cigarette **1**. Paper cylinder **4** contains a carbon free filter section **5**, which is preferably tow, which is contained within paper cylinder **6**. Carbon-containing section **7** contained within paper cylinder **8** comprises carbon particles **10** which are held in place by polyethylene oxide particles **11**. The filter unit, which is defined by paper cylinder **4**, is joined to the filler section, which is defined by paper cylinder **2**, by paper cylinder **15**, which covers all of paper cylinder **4** and a portion of paper cylinder **2**.

Referring further to FIGS. 3 and 4, there is shown a cigarette **12** having a paper cylinder **2**. Shredded tobacco **3** is contained within paper cylinder **2**. Paper cylinder **4** is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder **2**. Paper cylinder **4** generally defines the filter unit of cigarette **12**. Paper cylinder **4** contains a carbon-free filter section **5**, which is preferably tow, which is contained within paper cylinder **6**. Carbon-containing section **17** contained within paper cylinder **18** comprises tow **19** and carbon particles **20** which are held in place on the tow by polyethylene oxide particles **21**. The filter unit, which is defined by paper cylinder **4**, is joined to the filler section, which is defined by paper cylinder **2**, by paper cylinder **15**, which covers all of paper cylinder **4** and a portion of paper cylinder **2**.

Referring further to FIGS. 5 and 6, there is shown a cigarette **13** having a paper cylinder **2**. Shredded tobacco **3** is contained within paper cylinder **2**. Paper cylinder **4** is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder **2**. Paper cylinder **4** generally defines the filter unit of cigarette **13**. Paper cylinder **4** contains a carbon-free filter section **5**, which is preferably tow, which is contained within paper cylinder **65**. Carbon-containing section **27** contained within paper cylinder **28** comprises tow **29** and carbon particles **30** and polyethylene oxide particles **31**. Tow particles **29** are coated with a coating of polyethylene oxide **32**, which serves to hold the carbon particles **30** and the polyethylene oxide particles **31** in position on tow **29**. Polyethylene oxide particles **31** also serve to hold the carbon particles **30** in position in the filter. The filter unit, which is defined by paper cylinder **4**, is joined to the filler section, which is defined by paper cylinder **2**, by paper cylinder **15**, which covers all of paper cylinder **4** and a portion of paper cylinder **2**.

FIGS. 7 and 8 illustrate another form of the present invention wherein there is shown a cigarette **14** having a paper cylinder **2**. Shredded tobacco **3** is contained within paper cylinder **2**. Paper cylinder **50** is positioned at the other end of the cigarette from the shredded tobacco and abuts one end of paper cylinder **2**. Paper cylinder **50** generally defines the filter unit of cigarette **14**. Paper cylinder **50** contains a carbon-free filter section **55**, which is preferably tow, which is contained within paper cylinder **56**. Carbon-containing section **57** contained within paper cylinder **58** comprises carbon particles **60** which are held in place by polyethylene oxide particles **61**. Carbon-free section **65** contained within paper cylinder **66** is positioned between section **57** and filler or shredded tobacco **3** serves

to isolate the carbon-containing section from the filler as well as to provide additional particulate matter filtration. The filter unit, which is defined by paper cylinder **50**, is joined to the filler section, which is defined by paper cylinder **2**, by paper cylinder **75**, which covers all of paper cylinder **50** and a portion of paper cylinder **2**.

With particular reference to FIG. 9, which illustrates in detail a preferred form of the present invention, carbon particles **10** are positioned within concentric cylinders **8**, **4** and **15** and are held in position within cylinder **8** by the portions of the polyethylene oxide particles **11** which are fused to the carbon particles and to other polyethylene oxide particles to form a cohesive structure.

It will be seen from FIG. 9 that the particles of carbon and the particles of polyethylene oxide **11** form channel-like spaces **22**. These channel-like spaces generally define a plurality of tortuous paths, several of which are indicated by dotted lines **23**, along which the tobacco smoke travels. During such travel, the tobacco smoke encounters the relatively large carbon particles **10** and the relatively large polyethylene oxide particles **11** and is diverted thereby through adjacent passages. In this manner, the smoke not only passes around each particle with substantial contact occurring but also is forced through a more complex passage whereby there is intimate contact with both polyethylene oxide particles **11** and carbon particles **10** and is thereby thoroughly exposed to their combined action, as will be set forth later in this specification.

It will be understood that the materials employed in the cigarettes shown in the drawing are merely illustrative. For example, the carbon particles shown and the polyalkylene oxide particles described are merely representative of such materials and the paper coverings for the respective filter sections may also be varied from the arrangements shown in the drawing, dependent upon various factors, such as manufacturing convenience.

In order to prepare the tobacco smoke filter of the present invention, polyalkylene oxide resin particles in various mesh sizes and shapes can, for example, be admixed with activated carbon particles and thereafter the mixture is heated in order to partially fuse or bind the two types of particles together. Alternatively, the mixture may be applied to tow or to filter paper which may, if desired, be coated with a coating of polyethylene oxide, and may then be bonded together and to the tow or paper by heating to partially fuse the polyalkylene oxide. The mixture can be placed in a filter tube and then fused or can first be partially fused and, when cooled, packed into filter tubes and the resulting tubes are then heated to further fuse the particles together.

The particles can also be more completely fused together initially to form a rigid or semi-rigid structure which can then be cut into a desired size and shape for incorporation within a paper cylinder or the like to be employed as a filter.

As used heretofore and throughout this specification, unless otherwise stated, the term "particles" when employed to describe the polyalkylene oxide is intended to cover all of the various solid or semi-solid forms which the polyalkylene oxide may take. The polyalkylene oxide may have a molecular weight between about 70,000 and 5,000,000 and greater can be employed in the invention. The preferred molecular weight of the polyethylene oxide is from about 3,000,000 to about 5,000,000. Preferably, the polyalkylene oxide is used in a mesh size of from −1 to +60 (United States Standard), although satisfactory results can be obtained when only about 90% of the polyalkylene oxide particles are within the range, the remainder being between about 4 and 400 mesh.

The activated carbon particles which are employed in accordance with the present invention may have a particle size of from −10 to +150 mesh (United States Standard). Preferably, the majority of the carbon particles and the majority of the polyalkylene oxide particles have the same particle size distribution and both types of particles are substantially all in the range of −10 to +60 mesh. The particles may be roughly spherical in shape or may have other regular or irregular shapes, depending on how they are produced.

The activated carbon which is employed may be one of various known types, such as coke base carbon, petroleum base carbon, wood base carbon or nutshell carbon, or any other suitable carbon.

Preferably, the carbon employed is a coconut-type carbon. Such carbons have a somewhat irregular shape, and are therefore held somewhat more easily in position by the particles of polyethylene oxide. They also afford good contact and good pick up of undesirable gas phase constituents.

The polyalkylene oxide may be polyethylene oxide or a copolymer of ethylene oxide with less than 50% by weight of propylene oxide, i.e., oxides containing both $-C_2H_4O-$ and $-C_3H_6O-$ groups, and may be also mono- or diesters of such polyalkylene oxides, for example, the methoxy esters of polyethylene oxides. As used herein, the term "polyalkylene oxide" is intended to include all such materials, including the esters, having molecular weight of from about 70,000 to about 5,000,000.

Illustrative of such materials are polyethylene oxides which have the general formula:

$$HO(-C_2H_4-O-)_xH$$

wherein x is an integer having a value of from about 1,600 to about 115,000. Such materials may be prepared, generally, by polymerizing alkaline oxides by conventional methods. For example, ethylene oxide can be reacted in accordance with the following equations to yield the polymer:

(1) $$HC\overset{O}{\diagdown\diagup}CH + H_2O \longrightarrow HO-CH_2-CH_2-OH$$

(2) $$HO-CH_2-CH_2-OH + n(HC\overset{O}{\diagdown\diagup}CH) \longrightarrow HO(-CH_2-CH_2-O-)_{n+1}H$$

Particularly preferred polyalkylene oxides are water-soluble solid polyethylene oxide and copolymers containing at least 50 weight percent of ethylene oxide in copolymerized form with up to 50 weight percent of a second lower olefin oxide, for example, propylene oxide, butylene oxide, and the like.

In a most preferred embodiment of the present invention, polyethylene oxide and/or the above defined copolymers should have a reduced viscosity value in the range of from about 1.0 to about 75 to even higher, and most preferably should have a reduced viscosity of from about 2 to about 60. Reduced viscosity is an indirect measurement of the molecular weight of the polymer and it is a value obtained by dividing the specific viscosity by the concentration of the alkylene oxide polymer in the solution, the concentration being measured in grams of polymer per 100 millileters of solvent at a given temperature. The specific viscosity is obtained by dividing the difference between the viscosity of the solution and the viscosity of the solvent by the viscosity of the solvent. The reduced viscosities herein referred to are measured at a concentration of 0.2 gram of polyalkylene oxide in 100 millileters of acetonitrile at 30° C.

Although, in accordance with one embodiment of the present invention, as is illustrated in FIGS. 5 and 6, the poly (ethylene oxide) can be in the form of a film on tow or paper, when the film can be employed in conjunction with polyethylene oxide particles, it is preferably in the form of particles which function by means of fusing portions of their surfaces to hold themselves and carbon in a cohesive filter structure.

Solid alkylene oxide polymers can be prepared by certain metal carbonate catalysts, such as calcium carbonate, barium carbonate, strontium carbonate and the like. These metal carbonate catalysts can be employed in

concentrations in the range from about 0.3 to 3 parts by weight per 100 parts by weight of alkylene oxide. The polymerization reaction can be conducted in the liquid phase at a temperautre in the range from about 70° C. to about 150° C. It is preferred that the metal carbonate catalyst contain not more than one part by weight of non-sorbed water per 100 parts by weight of monomer, and at least 0.01 part by weight of sorbed water per 100 parts by weight of catalyst. It is also preferred that the carbonate catalyst be free from ions which reduced their catalytic activity such as, for example, chlorate and thiosulfate ions. Additional details regarding the production of polyalkylene oxide can be found in the disclosure in United State Patent 3,032,445 and the disclosure in the United States applications which are referred to therein.

The temperature and time for the fusion and heating of a mixture of polyalkylene oxide particles and carbon particles may vary according to the degree of fusion and rigidity which is desired and according to the molecular weight of the polyethylene oxide. However, the preferred temperature range is 90 to 120° C., although temperatures as low as 65° C. will work, so long as the polyalkylene oxide partially fuses with the carbon. The preferred time range when connective heating is employed is from about 10 minutes to about one hour. The use of microwave heating techniques can bring the time requirements down to the order of a few seconds. As an alternative, the polyalkylene oxide particle-carbon particle mixture may be fused by contacting the mixture with fine droplets of water at room temperature and thereafter be shaped into the desired form.

As used herein, the term "fused," when applied to the polyalkylene oxide particles, unless otherwise indicated, means joined by means of the polyalkylene oxide, itself. The joining or fusing will be between polyalkylene oxide particles themselves and between polyalkylene oxide particles and carbon particles. It may also be between either of those types of particles and tow or paper, if desired. The fusing may be by means of softening the polyalkylene oxide to the point where it will serve as such an adhesive. This "softening" can be accomplished, as indicated above, by heating the polyalkylene oxide particles to partially melt or soften them. The softening may also be accomplished by contacting the particles with water to "water-soften" the outer surface of the polyalkylene oxide particles. The softened particles can then be joined to the polyalkylene oxide particle. Although the softened particles may be tacky enough to fuse the particles to each other and to the carbon particles, generally the adhesive connection is established after the partially melted or water softened portion is hardened, for example, by cooling the melted particles or by drying the water softened particles.

In filter plugs which embody the present invention, from about 20 to 100 parts, by weight, and preferably from about 40 to 100 parts, by weight, of activated carbon are employed per 100 parts of polyalkylene oxide. Below 100 parts of polyethylene oxide per 100 parts of carbon, the fusion does not provide completely satisfactory results. Preferably, when the filter plug is to be employed in a cigarette or similar product, sufficient carbon should be present to provide from about 50 to about 150 mg. of carbon per cigarette.

Synthetic fibers, when employed in accordance with the present invention, are preferably cellulose acetate fibers and may be cellulose acetate tow or yarn having a total denier of from about 25,000 to about 75,000, and preferably from about 30,000 to about 50,000, and having a denier per filament of from about 1 to about 16, and preferably from about 3 to about 6. Preferably, the cellulose acetate fibers or filaments are generally longitudinally aligned and coextensive with the length of the filter element and have portions thereof which are crimped into a different alignment from the general orientation of the filaments, whereby the filaments have certain portions which somewhat intermesh, as illustrated in the drawing. While cellulose acetate fibers are the preferred materials, other cellulose esters, for example, cellulose butyrate, or other types of synthetic fibers and mixtures of synthetic fibers may also be employed as the fibers of the present invention.

Filter plugs embodying the present invention may contain other additives, for example, other adsorbents, such as silica gel, rice starch, activated aluminas and the like.

Filter plugs embodying the present invention will generally be from about 4 to 25 mm. in length, when employed in cigarettes or the like. The plugs are preferably incorporated in combination with an outer carbon-free plug, which may be a paper type plug or a tow type plug. Preferably, the outer plug is a cellulose acetate crimped fiber type plug having a total denier of from about 25,000 to 100,000, a denier per fiber of from about 1 to 16, a plug length of from about 4 to 20 mm. and a maximum RTD (resistance to draw) of about 2 inches of water.

An inner plug positioned between the tobacco and the carbon-containing plug may also be employed, if desired. This inner plug may be similar in nature to the outer plug.

Both the inner plug and the outer plug, when employed, may have incorporated therein polyethylene oxides, triacetin, various waxes, and the other additives and similar materials and may also include various flavorants, if desired.

It is to be understood, however, that when an inner plug and an outer plug are employed in combination with a carbon-containing filter of the present invention, the inner and outer plugs need not be of the same construction or contain the same additives, if any. Thus, for example, the outer plug may contain various flavorants which are not present in the inner plug.

While we do not wish to be bound by any particular theory, we believe the unexpectedly superior properties of the present filter are due at least in part to the following reasons:

(1) The carbon particles are bound with the polyethylene oxide particles to form a relatively rigid structure which has no undesirable void spaces and which does not lose any substantial number of particles due to their migration (caused, for example, by mechanical settling or by the drawing of smoke through the filter).

(2) The bonding action of the polyethylene oxide particles does not interfere with the adsorptive properties of the carbon particles and also function themselves as absorbents.

(3) The polyethylene oxide particles serve a particularly unexpected function, in that, due to their selective removal of water-solubles and certain organic gaseous components from the smoke, the carbon particles are rendered more effective for the removal of the other gaseous phase components from the smoke. These properties are in addition to the ability of both the carbon and the polyethylene oxide to remove particulate matter from the smoke.

For a further understanding of the present invention, reference may be had to the following examples, which merely serve to illustrate certain forms of the present invention.

Example 1

Two hundred milligrams of a polyethylene oxide resin (designated as Polyox WSR1H–3, DS7–34–Z) having a molecular weight of about four million, a density of about 32.7 pounds per cubic foot and a viscosity of about 2500 centipoises (in aqueous solution of one weight percent concentration at 25° C.) were employed in the form of particles having a size of 30 to 60 mesh. The polyethylene oxide resin particles were admixed with 30% by weight

of activated carbon particles (Columbia LC) having a particle size of 48–150 mesh until the two types of particles were uniformly admixed. The resulting mixture was then placed in an oven which was maintained at a temperaturme of 100° C. and the mixture was permitted to remain in the oven for a period of one minute in order to partiallly fuse. The mixture was then removed from the oven and permitted to cool in the air to ambient temperature after which it was packed into 3 paper filter tubes, each of which was 90 mm. long. The 3 tubes were designated as Tube No. 1, Tube No. 2 and Tube No. 3. Tube No. 1 was placed in an oven maintained at 100° C. for three minutes, Tube No. 2 was placed in an oven maintained at 100° C. for five minutes and Tube No. 3 was placed in an oven maintained at 100° C. for eight minutes. After the indicated times, each of the tubes was removed from the oven and permitted to stand at room temperature overnight in order to cool. The following day, each of the tubes was cut into five 15 mm. lengths.

Tube No. 3 was found to have more complete fusion of the polyethylene oxide resin than did Tube No. 2, or Tube No. 1, as also indicated by an examination of the fall-out of particles from each of the tubes.

Sample filter plugs, each 15 mm. long, were employed from each of the three tubes and attached to filler rods each of which contained the identical type of tobacco and also constructed in identical manner. The resulting cigarettes were evaluated for their efficiency in removing TPM (total particulate matter). This was done by placing the samples in a smoking device which provided a continuous puff. The device consisted of a vacuum pump capable of creating a vacuum of 600 cc. per minute. The first 20 mm. of each cigarette was burned continuously. The weights of the TPM collected on the cigarette filter and on the Lucite filter which was attached to the device were measured and these weights were employed to determined filter efficiency.

The average efficiency of the five plugs made from Tube No. 1 was 62.8%. The average efficiency of the five plugs made from Tube No. 2 was 58.2% and the average efficiency of the five plugs made from Tube No. 3 was 61.4%. The data are shown in Table I.

TABLE I

Samples From Tube	No. 1	No. 2	No. 3
Av. RTD, Before Smoking	2.0	1.4	1.5
Av. RTD, After Smoking	1.9	1.8	2.2
Av. Filter and Holder (mg.)	29.4	32.3	34.5
Av. Lucite Filter (mg.)	14.1	23.3	21.8
Total	43.5	55.6	56.3
Av. Filter Efficiency (percent)	62.8	58.2	61.4

A similar series of cigarettes were prepared and were tested, together with control cigarettes, employing a synthetic gas mixture to determine their efficiency for gas phase removal. The test and control plugs were evaluated as follows:

An evacuated 1-meter gas cell was connected to a tank which contained a synthetic gas-phase smoke mixture. This mixture had been formulated with compounds reported in the literature as being representative of the several hundred compounds found in the complex mixture which is known as the gas phase of smoke.

The test plug was placed in a glass tube connecting the 1-meter gas cell with the container containing the mixture. Then 35 ml.-puffs were taken with a syringe and passed into the gas cell. Two of the test filters were used in each determination to obtain the concentration of gases necessary for the analyses. The gas from the twenty puffs was analyzed on the infrared spectrophotometer. The control filters were treated in the same manner. The filter efficiencies of the test and control plugs were compared by calculating the decrease of the absorbance bands for the respective components of the smoke mixture as shown for the test and control plugs. The plugs showed reductions of over 90% in four of the seven components of the gas mixture. Data are shown in Table II.

TABLE II

	Isoprene	Acetone	Acetaldehyde	MEOH	MECl	Methyl furan	Benzene
Control Av	8.96	3.58	11.10	6.02	6.00	2.48	3.02
Percent Reduction	7	7	5	6	4	7	25
Tube No. 1	(1)	(1)	5.32	3.88	5.32	0.42	0.32
Percent Reduction	90+	90+	54	39	15	84	92
Tube No. 2	(1)	(1)	7.12	4.70	5.80	0.48	1.14
Percent Reduction	90+	90+	39	26	7	82	72
Tube No. 3	(1)	(1)	5.62	4.0	5.50	0.32	0.18
Percent Reduction	90+	90+	52	37	12	88	96

[1] Because of the excellent filtration efficiency, the absorbance bands of these constituents were too small for accurate measurements. The reduction, however, was above 90%.

In addition to the above experiments, a further batch of cigarettes employing the present invention were compared with samples of a conventional cigarette (Parliament brand cigarettes) by smoking to determine existing smoke temperature. The smoking was done on a constant volume smoking machine through a Lucite Cambridge filter holder which had been modified to receive a thermocouple wire across the cigarette opening. A dental dam was placed on the Lucite filter and a cigarette, previously marked in 5 mm. increments, was inserted into the dam and gently pushed against the thermocouple wire. The cigarette was then smoked to a 25 mm. butt length. The puffs were recorded on a Leeds-Northrup recorder and the recorded millivolt deflections were converted to degrees centigrade. The thermocouples had been calibrated previously by immersion in boiling water and in an ice bath to obtain a plot to be used for conversion of the millivolt readings. Temperature values that fell between the 27–33 mm. butt length were used so that an average temperature at the 30 mm. butt length could be obtained.

The average of the maximum exit smoke temperatures were 27.2° C. for the test cigarette and 55.2° C. for the Parliament control.

Example 2

50 grams of a polyethylene resin having a molecular weight of 4,000,000 (Polyox WSR 301) having a mesh size of 35–48 mesh were thoroughly admixed with 22 grams of activated carbon (coconut carbon) having a mesh size of 48–150 mesh. After the two types of particles were thoroughly admixed, the mixture was packed into 8 mm. filter tubes and placed in an oven maintained at 100° C. After 40 minutes, the packed filter tubes were removed from the oven, cooled to room temperature and cut into 15 mm. filter plugs. Each of the 15 mm. filter plugs was then placed in a 23 mm. tube, whereby an 8 mm. recess was provided in each tube. The RTD of each plug was determined to be in the range of 3.4 to 4.0 inches of water. These test filters were then submitted to a panel of judges for evaluation of the flavor which was detectable by drawing on the filters when no tobacco was attached thereto, i.e., by unlit draw. The test filters were judged by the panel to have a very low taste level and the tastes which were detected were determined by the panel to be at a level which was not undesirable.

Example 3

Filters similar to those prepared in Examples 1 and 2

were prepared in accordance with the following specifications:

A polyethylene resin having a molecular weight of about 4,000,000 (Polyox WSR 301) was employed as particles having a size of 35–48 mesh. The polyethylene oxide resin particles were admixed with 30% by weight of activated carbon particles (Columbia LC) having a particle size of 48–150 mesh until the two types of particles were uniformly admixed. The resulting mixture was then placed in a tube and put into an oven which was maintained at a temperature of 100° C. and the mixture was permitted to remain in the oven for a period of 40 minutes, removed from the oven, cooled to room temperature and cut into 15 mm. filter plugs. The RTD of each plug was determined to be in the range of 3.7 to 4.0 inches of water. The average weight of each plug was 310.3 mg. and there was approximately 93 mg. of carbon per plug.

The filters were attached to Parliament filler rods. The overall RTD (cigarette and filter) was 4.4–4.7 inches of water. A Parliament filler rod was prepared for use as a control.

The test cigarettes and the controls were smoked to determine the gas phase efficiency of the filter on whole smoke. The test and control cigarettes were smoked on a standard smoking machine with a standard procedure in which 50 mm. of each cigarette was smoked, taking each minute a 35 cc. puff of 2-second duration. The residual smoke was collected in liquid nitrogen and any particulate matter was filtered out. Then the sample was expanded into a mass spectrometer. By using a low-voltage ionization technique, the percent reduction of the major gas-phase components of the smoke were determined. The Polyox carbon filter plugs showed significant reductions in 17 of the 20 components in the gas phase. These results are given in Table III.

TABLE III

Components	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20
									Control (Parliament without filter)											
Average of 3 samples	45	138	245	133	81	185	300	726	30	603	218	57	320	1,138	206	184	12	212	579	31
									Polyox With Carbon											
Average of 3 samples		2						3	18		13		255	242	86	20	12	28	803	47
Average Reduction, percent	100	99	100	100	100	100	100	100	40	100	94	100	20	79	58	89		88	+28	+34

As used herein, all parts and percentages are by weight, unless otherwise specified.

As used herein, the terms "polyethylene oxide" is interchageable with and has the same meaning as the term "poly(ethylene oxide)." The term "polyalkylene oxide," similarly, is interchangeable with the term "poly(alkylene oxide)."

We claim:

1. A filter for tobacco smoke comprising activated carbon particles and fused polyalkylene oxide particles, said filter containing from about 20 parts to about 100 parts by weight of said carbon particles per 100 parts of said polyalkylene oxide particles, at least 90% by weight of said polyalkylene oxide particles having a mesh size of from −10 to +60, the remainder of said polyalkylene oxide particles having a mesh size of from about −4 to about +100, the majority of said carbon particles having a mesh size of from −10 to +60, the remainder of said carbon particles having a mesh size of from about −10 to about +150, said activated carbon particles and polyalkylene oxide particles being fused together at a plurality of points to from a coherent mass having passages therethrough.

2. A smoking article comprising tobacco and a filter, said filter comprising activated carbon particles and fused polyalkylene oxide particles, said filter containing from about 20 to 100 parts by weight of said carbon particles per 100 patrs of said polyalkylene oxide particles, at least 90% by weight of said polyalkylene oxide particles having a mesh size of from −10 to +60, the remainder of said polyalkylene oxide particles having a mesh size of from about −4 to about +100, the majority of said carbon particles having a mesh size of from −10 to +60, the remainder of said carbon particles having a mesh size of from about −10 to about +150, said activated carbon particles and polyalkylene oxide particles being fused together at a plurality of points to from a coherene mass having passages therethrough.

3. A filter for tobacco smoke comprising activated carbon particles and fused polyethylene oxide particles, said filter containing from about 20 to 100 parts by weight of said carbon particles per 100 parts of said polyethylene oxide particles, at least 90% by weight of said polyethylene oxide particles having a mesh size of from −10 to +60, the remainder of said polyethylene oxide particles having a mesh size of from about −4 to about +100, the majority of said carbon particles having a mesh size from −10 to +60, the remainder of said carbon particles having a mesh size of from about −10 to about +150, said acitvated carbon particles and polyethylene oxide particles being fused together at a plurality of points to from a coherent mass having passages therethrough.

4. A smoking article comprising tobacco and a filter, said filter comprising activated carbon particles and fused polyethylene oxide particles, said filter containing from about 20 to 100 parts by weight of said carbon particles per 100 parts of said polyethylene oxide particles, at least 90% by weight of said polyethylene oxide particles having a mesh size of from −10 to +60, the remainder of said polyethylene oxide particles having a mesh size of from about −4 to about +100, the majority of said carbon particles having a mesh size of from −10 to +60, the remainder of said carbon particles having a mesh size of from about −10 to about +150, said activated carbon particles and polyethylene oxide particles being fused together at a plurality of points to from a coherent mass having passages therethrough.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,409,020 November 5, 1968

Claud Eric Westbrook, Jr., et al.

It is certified that error appears in the above identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 10, before "incorporating" cancel "the"; line 16, "each" should read -- even --; line 29, "no" should read -- not --. Column 3, line 12, "or" should read -- of --. Column 4, line 50, "65" should read -- 6 --. Column 5, line 66, "-1" should read -- -10 --. Column 6, line 72, after "by" insert -- polymerizing an alkylene oxide in the presence of --. Column 7, line 18, "a" should read -- the --. Column 9, line 7, "partiallly" should read -- partially --. Column 12, line 7, "patrs" should read -- parts --; line 16, "coherene" should read -- coherent --.

Signed and sealed this 31st day of March 1970.

(SEAL)

Attest:

Edward M. Fletcher, Jr.

Attesting Officer

WILLIAM E. SCHUYLER, JR.

Commissioner of Patents