May 8, 1962 G. P. TOUEY ET AL 3,033,212

TOBACCO SMOKE FILTER CONTAINING POLYOLEFIN COBWEB ADDITIVE

Filed April 17, 1959

CELLULOSE ACETATE TOW FILAMENTS WITHOUT WAX FILAMENTS

Fig. 1

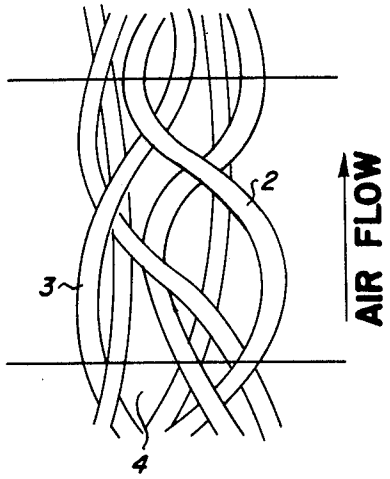
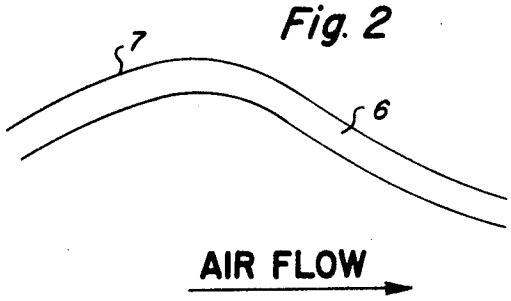


Fig. 2



CELLULOSE ACETATE TOW FILAMENTS CONTAINING COBWEB FILAMENTS OF POLYETHYLENE WAX

Fig. 3

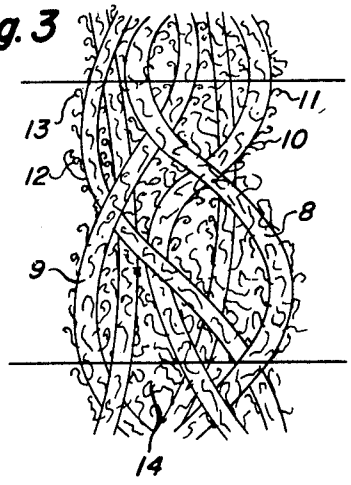
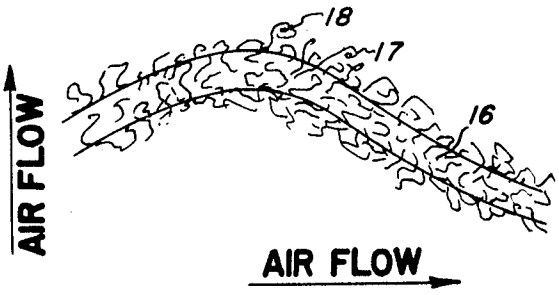


Fig. 4



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United States Patent Office

3,033,212
Patented May 8, 1962

3,033,212

TOBACCO SMOKE FILTER CONTAINING POLYOLEFIN COBWEB ADDITIVE

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Filed Apr. 17, 1959, Ser. No. 807,003
6 Claims. (Cl. 131—208)

This invention relates to a new and improved tobacco smoke filter and to methods for its preparation. In particular, this invention relates to methods of adding polyethylene and polypropylene materials in the form of fine cobweb structures to a filter material comprised essentially of crimped, continuous filaments arranged in the form of a textile tow.

This invention is a continuation-in-part of our earlier application Serial No. 480,059, now U.S. Patent 2,904,050.

Among the wide variety of materials which have been recommended for tobacco smoke filters, such as cigarette filters, one type in particular is ideally suited for this purpose from a manufacturing standpoint. This type is a continuous, crimped cellulose acetate textile tow as described in the Crawford and Stevens U.S. Patents 2,794,-239 and 2,794,480. One reason for its widespread use is the ease and rapidity at which the material can be processed into rigid filter rods on commercial equipment used in the cigarette industry. Thus, for example, a crimped, continuous cellulose acetate tow of 5,000 to 30,000 filaments depending on the thickness of the spun filaments can be bloomed out, sprayed with a nonvolatile liquid plasticizer type of bonding agent, recompacted and pulled through a rod forming and paper wrapping device to produce 90 mm. filter rods at the rate of at least 750 rods per minute. Such rods become quite rigid on standing several hours, due to the plasticizer on the cellulose acetate, and therefore can be easily and rapidly cut to any desired filter length with clean, well defined cuts.

It has been proposed to improve continuous, crimped tow filters from the standpoint of its ability to remove the tiny nicotine and tar particles from cigarette smoke by applying additive to the filaments. That is, the substantial parallel relationship of many of the filaments which constitute the filtering element may permit a certain amount of the nicotine and tar particles to pass between the longitudinally aligned smooth filaments without touching them. These particles, therefore, are not trapped. One method of improving the filter is to spray the filaments with a melted wax. This method has been described in Patent No. 2,904,050. Such materials as paraffin wax, stearyl alcohol, stearic acid, glycerol monostearate and the relatively new synthetic waxes can be used for this purpose. As described in Patent No. 2,904,050, these materials are converted from a liquid to a fine spray of solid particles as they are ejected from the spray gun and come in contact with the air. These fine particles deposit on the filaments in the spread out tow making protrusions on the fibers. Such protrusions extend into the spaces between the fibers in the finished filter. These protrusions, in turn, make the path of the smoke particles through the filter more difficult and part of the nicotine and tar particles are impinged on these protrusions.

Although the addition of fine particles, as just described, improves a cellulose acetate tow type of filter, there are certain aspects which may be further improved. It may be difficult under some conditions to prevent a portion of the particles from falling out of the finished filter. This is unsatisfactory since such a condition not only decreases the effectiveness of the filter, but also presents the possibility of some of the particles entering the smoker's mouth. In addition, it may not be very desirable if a smoker can observe traces of a powder sifting out of a

filter when he taps his filter cigarette against a hard surface.

The addition of certain bonding agents to the tow prior to the application of the additive particles substantially retards this "sifting out" effect. However, the application of a bonding agent to the filter tow during the fabrication of the filter can add to operating costs. Therefore, it is believed apparent that it is highly desirable to employ an additive which will not sift out of the end of the finished filter if such an additive were capable of substantially improving the effectiveness of the filter.

After further investigation we have found a method and product which constitutes an improvement over the additive filters of our patent aforementioned as well as over other processes and products.

One object of this invention is to provide a better method for incorporating certain additive substances into a cellulose acetate tow filter to increase its effectiveness in removing nicotine and tar from tobacco smoke. Another object is to provide a method for adding a waxy composition to a cellulose acetate tow filter without producing particles which might be dislodged from the filter when it is tapped. Still another object is to provide an improved filter product resulting from our improved methods. Other objects will appear hereinafter.

In the broader aspects of our invention these objects are obtained by spraying the spread out tow with certain melted waxy combinations which produce a mass of discontinuous cobweb filaments rather than particles on the surface of the tow. These cobweb filaments, in turn, become entangled among the tow filaments disrupting the otherwise highly orientated nature of the filter. This, as in the case of additive particles, creates a more difficult path for the smoke which results in more of the nicotine and tar particles being trapped by the filter. In addition, there is the advantage that there is no additive to sift out.

In order to produce a spray of discontinuous waxy cobweb filaments rather than tiny particles, we have found that the melt can be made up of either 100% polyethylene or polypropylene wax or a compatible mixture of polyethylene or polypropylene wax and another waxy compound. In either case the average molecular weight of the waxy melt should be within the range of 500 and 10,000 and preferably between the range of 1,000 and 5,000. Also, the temperature of the melt in the spray gun should be within the range of 90 and 200° C. Should the molecular weight of the pure polyolefin or the blend of polyolefin and another wax be lower than that specified or should the temperature be higher than that specified, the product will be ejected from the spray gun in the form of tiny particles rather than fine cobweb filaments. Should the temperature be lower than that specified, the wax would not be a free flowing liquid. One advantage of using blends of polyethylene or polypropylene and other waxy materials is that one can use a relatively high molecular weight polyolefin i.e., about 6,000, and by dilution with a low molecular weight wax (in comparison with the polyolefin) arrive at an average molecular weight within the limits of 500 and 10,000.

It is important to the operation of this invention that the average molecular weight of the polyolefin wax or the wax blend containing the polyolefin should be within the limits specified. As previously mentioned, when the average molecular weight is below 500 there is a tendency of the melt to form particles as it is sprayed rather than the desired cobweb structure. When the molecular weight of the melt is above 10,000, the melt is quite viscous at the specified temperature range of 100 to 200° C. and cannot be conveniently sprayed by conventional methods.

In order to render this aspect definite, we have selected the Harris method for determining the molecular weight of our polyolefin or polyolefin modified waxes. This

method is discussed in the Journal of Polymer Science, vol. 8, pp. 352–364 (1952). However, if it is desirable to use the so-called oxidized or emulsifiable polyolefins in place of the pure polyolefin waxes, then we prefer to use the Erchak (U.S. Patent 2,504,400) method for the molecular weight determination. The waxes to be used as diluents for a high molecular weight polyethylene or polypropylene may be chosen from the list of waxes disclosed in U.S. Patent No. 2,904,050. Paraffin wax and stearyl alcohol are examples of such diluents.

Any number of hot spray guns are suitable for spraying the hot wax melts of the present invention onto the cellulose acetate tow to produce the cobweb filament structure. A Binks spray gun operating at a temperature of 100 to 200° C. is suitable. If desirable, steam pressure in place of air pressure may be used to eject the melt from the gun in the form of the fine (cobweb) filament structure. The proper opening for the nozzle of any spray gun may be obtained simply by trying various openings.

On advantage of applying a spray of waxy cobweb filaments rather than minute particles is the fact that the filaments become entangled with each other and with the tow filaments in such a manner that it is practically impossible for them to be dislodged from the finished filter. Thus, the efficiency of a cellulose acetate tow filter is increased to the levels disclosed in our Patent No. 2,904,-050 by using certain polylefin wax melts which at the same time do not introduce any problem of the wax being loosely bound to the filaments.

Adding a molten wax or wax mixture to produce cobweb filaments on the tow filaments has other advantages. For example, no volatile solvent is required. This is a marked improvement over those methods for producing a mat of fine fibers by spraying various polymers dissolved in a volatile solvent. Such methods delay the rate of filter manufacture since the filter material preferably is free of volatile solvents before it is attached to cigarettes.

The amount of waxy cobweb filaments which are added to the cellulose acetate tow to produce a more effective filter depends to some extent on the type of tow employed as the carrier for the filaments. Thus with a tow consisting of filaments of 8 denier per filament and larger, it has been found that more additive may be used than is the case when a tow consisting of, say, 2 denier per filament. Also, the degree of crimps in the tow is another factor to be considered. In any event the maximum amount of cobweb filaments required is 50% and the minimum amount is 5%, based on the entire weight of the filter without its wrapper. The most desirable range is 10 to 35% cobweb filaments.

In addition to polyethylene and polypropylene we have found that several modifications of these compounds can be used alone or blended with other waxy materials. Thus as already mentioned the oxidized or emulsifiable polyethylene and polypropylene can be used. Since these can be extremely low molecular weight polymers, they can be used as blending agents with the higher molecular weight polyolefin compounds to produce an average molecular weight without the range of 500 to 10,000.

A cellulose acetate tow is the desired type of carrier or base for the operation of this invention. This is because it can be "cured" (bonded) with plasticizers. However, we have found that textile tows of other compositions may be employed with other means of producing a firm rod.

For a further understanding of certain aspects of our invention reference may be made to the attached drawing forming a part of the instant application.

In this drawing FIG. 1 is a diagrammatic illustration on a considerably enlarged scale of a bundle of cellulose acetate filaments which have not been treated by the present invention;

FIG. 2 is a diagrammatic illustration on an enlarged scale of a single cellulose acetate filament which has not been treated;

FIG. 3 is a diagrammatic illustration on a considerably enlarged scale of a bundle of filaments similar to the bundle of FIG. 1 but in which the filaments have been treated in accordance with the present invention to provide cobweb filaments intermingled with the cellulose acetate filaments; and

FIG. 4 is similar to FIG. 2 but shows a single filament which has the cobweb filaments in association therewith.

Referring to FIG. 1, 2 and 3 illustrate particular crimped cellulose acetate filaments. It may be observed at point 4 that there are channels or openings between the various filaments through which the smoke may pass without encountering filter action. FIG. 2, which shows the single filament 6, indicates at 7 the relatively smooth surface of the filaments.

Referring now to FIG. 3, it will be observed that the several cellulose acetate filaments exemplified by 8 and 9 have a multitude of cobweb filaments 10, 11, 12 and 13 attached to the surface. As will be noted at 14, the channels or other openings between the cellulose acetate foundation filaments are thereby filled to some extent with the cobweb filaments of the present invention. Referring to FIG. 4, the effect of the cobweb filaments 17 and 18 on the single filament 16 are further illustrated by this showing.

For a further understanding of our invention, reference will now be made to the following examples for illustrating certain preferred embodiments. In these examples the same brand of king-size cigarette was used throughout the smoking tests. It was a brand representing a typical domestic type of cigarette tobacco. Only cigarettes with a weight range between ±2% of the average weight of 400 cigarettes were chosen to represent the brand. These are designated as "standard cigarettes" in the examples.

EXAMPLE 1A. CIGARETTES CONTAINING A FILTER OF CELLULOSE ACETATE AND POLYETHYLENE WAX

A 5 denier per filament cellulose acetate yarn was converted to a crimped (12 crimps per inch) tow containing 14,000 filaments. The tow was pulled over a series of air jets which bloomed it out to a width of 12 inches and while in this spread out condition it was sprayed on both sides with glycerol triacetate plasticizer. After this treatment the tow was pulled through a spray booth where it was sprayed on both sides with a web-like formation of fine filaments of polyethylene wax. The polyethylene had an average molecular weight of 2,700 as measured by the Harris method (J. Polymer Science, 8, 353–364 (1952)). The cobweb filaments were formed by heating the polyethylene (150° C.) in the spray gun pots until the polymer melted to a sprayable liquid which was forced through the nozzle of the guns by means of air pressure.

The tow was then pulled back together into its original compact form and led through a device which paper wrapped it producing a rod 10 feet in length and with a circumference equal to that of the standard cigarette. The rod was cut into 15 mm. filter tip lengths which gradually became quite firm due to the action of the plasticizer on the cellulose acetate fibers. Analysis of several of the filters from which the paper wrapper was removed showed that they consisted of 70% cellulose acetate filaments, 20% polyethylene cobweb filaments and 10% glycerol triacetate plasticizer. The polyethylene filaments could not be dislodged from the filters by viborously tapping them against a hard surface. Microscopic examination of several of the opened tips showed that the polyethylene filaments were entangled among the orientated acetate filaments in a fairly uniform manner. Ten of the filter tips were attached to the standard king-size cigarettes which had been shortened by 15 mm.

to compensate for the length of the filter. These cigarettes were smoked to butt lengths of 30 mm. on an automatic smoking machine which produced a 35 ml. puff of 2 seconds duration at the rate of 1 puff per minute. The smoke which passed through the filters was collected and analyzed for its nicotine and tar content. The results of this test are listed in Table I.

EXAMPLE 1B CIGARETTES CONTAINING A FILTER OF CELLULOSE ACETATE AND POLYPROPYLENE WAX

Another rod was prepared from the same tow of Example 1A and in the same manner as that described above. However, in this case polypropylene filaments were used as the waxy additive. These filaments were formed by spraying a hot melt (160° C.) of polypropylene with an average molecular weight of 3,800 against the spread out tow after the plasticizer has been applied. After the rod had become firm it was cut into 15 mm. filter tips. The tips (paper removed) consisted of 74% cellulose acetate filaments, 18% polypropylene filaments and 8% glycerol triacetate plasticizer. The polypropylene filaments were firmly entangled among the longitudinally aligned cellulose acetate filaments in a fairly uniform manner. Ten of the filter tips were attached to the standard king-size cigarettes which had been shortened by 15 mm. and the cigarettes were smoked to butt lengths of 30 mm. by the automatic smoking machine. The smoke which passed through the filters was analyzed for its nicotine and tar content. The results of this test are listed in Table I.

EXAMPLE 1C. CIGARETTES CONTAINING CELLULOSE ACETATE FILTER WITH A WAX BLEND OF POLYPROPYLENE AND STEARYL ALCOHOL

A third filter rod was prepared from the same tow of Example 1A and in the same manner as that described above. In this case the waxy cobweb filaments were a blend of 80 parts polypropylene (average molecular weight of 12,000) and 20 parts stearyl alcohol. The cobweb filaments were formed by spraying the melted blend (135° C.) against the tow after the plasticizer has been applied. After the rod had become firm, it was cut into 15 mm. filter tips which consisted of 63% cellulose acetate filaments, 26% cobweb filaments and 11% glycerol triacetate plasticizer based on the total weight of the filter without the paper wrapper. The waxy cobweb filaments were firmly entangled among the longitudinally aligned cellulose acetate filaments in a fairly uniform manner. Ten of the filters were attached to the standard king-size cigarettes which had been shortened by 15 mm. and the cigarettes were smoked to butt lengths of 30 mm. by the automatic smoking machine. The smoke which passed through the filters was analyzer for its nicotine and tar content. The results of this test are listed in Table I.

EXAMPLE 1D. CIGARETTES CONTAINING CELLULOSE ACETATE FILTERS WITHOUT COBWEB ADDITIVE

A 5 denier per filament cellulose acetate yarn was converted to a crimped (18 crimps per inch) tow containing 18,000 filaments. The tow was bloomed out to a width of 12 inches and sprayed on both sides with glycerol triacetate plasticizer. It was then pulled back into its original compact form and led through the filter rod-making apparatus which produced a 10-feet rod with a circumference equal to that of the standard cigarette. The rod which becomes quite firm after about 3 hours consisted of 88% cellulose acetate filaments and 12% plasticizer based on the total weight of the filaments and plasticizer. Part of the rod was cut into 15 mm. filter tips and ten of these tips were attached to the standard cigarettes which had been shortened by 15 mm. These cigarettes were smoked to butt lengths of 30 mm. on the automatic smoking machine. The smoke which passed through the filters was collected and analyzed for its nicotine and tar content. The results of this test are listed in Table I.

EXAMPLE 1E. CIGARETTES CONTAINING NO FILTERS (CONTROLS)

As controls, ten of the unshortened standard king-size cigarettes were smoked on the automatic machine to butt lengths of 30 mm. The smoke which passed through the butt end of these cigarettes was analyzed for nicotine and tar content. The results of this test are listed in Table I.

Table I

SMOKING TEST RESULTS FOR EXAMPLE 1

Type of Filter	Average Pressure Drop [1] of the 15 mm. Filter (in.)	Tar From the 10 Cigarettes (mg.)	Nicotine from the 10 Cigarettes (mg.)
Tobacco (Example 1E)	0.7	176	29
Cellulose acetate without wax additive (Example 1D)	1.7	140	24
Cellulose acetate with wax blend of polypropylene and stearyl alcohol (Example 1C)	2.0	102	18
Cellulose acetate with polypropylene wax cobweb (Example 1B)	1.9	114	19
Cellulose acetate with polyethylene wax cobweb (Example 1A)	1.8	118	20

[1] Pressure drop expressed as inches of water at an air flow rate through the cigarette of 17.5 ml./sec. The value shown is the average pressure drop of the filters used in the smoking test.

The results in Table I show that the cellulose acetate filter removed 20% more tar and 17% more nicotine than the 15 mm. of tobacco it replaced. The cellulose acetate-polyethylene filter removed 33% more tar and 31% more nicotine than the tobacco it replaced. The cellulose acetate-polypropylene filter removed 35% more nicotine and tar than the tobacco it replaced. The cellulose acetate-polyethylene-stearyl alcohol filter removed 42% more tar and 38% more nicotine than the tobacco it replaced.

The results also show that this improvement in filter effectiveness is obtained with a negligible increase in the pressure drop of the filter due to the addition of the cobweb filament structure.

The results further show that this degree of improvement in a cellulose acetate tow type of filter may not be achieved simply by increasing the number of filaments in the tow. The filter without a cobweb wax additive (Example 1D) had about the same pressure drop as those with the cobweb additives. This was due to the extra number (4,000) of cellulose acetate fibers in the filter.

EXAMPLE 2A. CIGARETTES CONTAINING A FILTER OF CELLULOSE ACETATE AND OXIDIZED POLYETHYLENE WAX

An 8 denier per filament cellulose acetate yarn was converted to a crimped (10 crimps per inch) tow containing 8,130 filaments. The tow was bloomed out to a width of 12 inches and while in this spread out condition it was sprayed on both sides with propylene glycol monoacetate (plasticizer). The tow was then pulled through a spray booth where it was sprayed on both sides with a web-like formation of fine filaments produced from an oxidized polyethylene having an average molecular weight of 2,500 as determined by the Erchak procedure (U.S. 2,504,400). The web-like filaments were deposited on the tow by ejecting the melt (130° C.) from the spray gun with 30 pounds pressure steam. After this treatment the tow was pulled back to its original compact form and led through the paper wrapping and rod forming apparatus. The resulting 8 feet rod, which became quite firm after 5 hours, consisted of 60% cellulose acetate filaments, 30% wax filaments and 10% plasticizer based on the combined weight of these three components. The circumference of the rod was the same as the circumference of the standard cigarettes. Part

of the rod was then cut into 15 mm. filter tips. It was not possible to jar any of the waxy cobweb filaments from these filter tips by tapping them vigorously against a hard solid surface. Microscopic observation of some of the opened tips revealed a fairly uniform blend of longitudinally aligned cellulose acetate filaments and fine waxy filaments with the waxy filaments firmly entangled in the cellulose acetate filaments.

Ten of the filters were attached to the standard king-size cigarettes which had been shortened by 15 mm. and the cigarettes were smoked to 30 mm. butt lengths by the automatic smoking machine. The collected smoke was analyzed for its nicotine and tar content. The results of this test are listed in Table II.

EXAMPLE 2B. CIGARETTES CONTAINING A FILTER OF CELLULOSE ACETATE WITHOUT A WAX ADDITIVE

An 8 denier per filament cellulose acetate yarn was converted to a crimped (10 crimps per inch) tow containing 10,000 filaments. The tow was bloomed out to a width of 12 inches and while on this spread out condition it was sprayed on both sides with propylene glycol monoacetate. It was then pulled back to its original compact form and fed into the rod forming apparatus which produced a 10 foot rod with a circumference equal to that of the standard cigarette. After the rod had become firm (5 hours) part of it was cut into 15 mm. filter tips. These tips consisted of 89% cellulose acetate filaments and 11% plasticizer, based on the combined weight of these two components. Ten of the filters were attached to the standard king-size cigarettes which had been shortened by 15 mm. and the cigarettes were smoked to 30 mm. butt lengths by the smoking machine. The collected smoke was analyzed for its nicotine and tar content.

Table II

SMOKING RESULTS

Type of Filter	Average Pressure Drop of the 15 mm. Filter (in.)	Tar from the 10 Cigarettes (mg.)	Nicotine from the 10 Cigarettes (mg.)
Cellulose acetate without wax (Example 2B)	1.4	155	25.5
Cellulose acetate with wax filaments	1.7	123	20

The results in Table II, when compared with those obtained from the unfiltered cigarettes (Tables I and II), show that the filter without the wax additive removed 12% nicotine and tar while the one with the wax additive removed 30% tar and 31% nicotine. These values represent the percentage increase in removal over that obtained for an equivalent length of tobacco filter. Again the results show that this increase in filter effectiveness cannot be achieved by simply adding more cellulose acetate filaments to increase the pressure drop of the filter.

Any suitable plasticizer may be used in addition to those shown. Other plasticizers are shown in the aforementioned Crawford and Stevens patents.

It is believed apparent from the foregoing description that we have provided a novel and relatively simple method for producing a tobacco smoke filter containing an additive, which filter is free of any tendency for the additive to shift out. Such filter with the cobweb filament additive not only is distinctive as compared with prior art additive filters, but as shown from the several tests set forth above, is efficient in its filtering capabilities.

The base or foundation filaments are preferably continuous, crimped filaments of a denier per filament of less than 16 and in some instances between about .3–4 denier per filament. The total denier of the tow is preferably between 20,000 and 120,000. The crimp is greater than 7 crimps per inch and preferably may be 12, 15 or even up to 25 crimps per inch.

The cobweb filaments resulting from spraying the waxy materials of the proper molecular weight and under the proper conditions are relatively fine. However, their exact size is not a limitation on the present application. As already pointed out above, due to the opened up or banded base filaments, the cobweb filaments become sufficiently intermingled and interdispersed in the base filaments so that they will not sift out or otherwise become loosened from the filter during its normal use.

Although the filters described above in the several examples are firm and the filter rods may be cut into the desired length with sharp, clean cuts, we have further found that in some instances a somewhat improved filter element may be obtained if a slightly heavier paper wrapper is used. That is, the paper wrapper frequently used in wrapping the filters is of a thickness less than 1.4 mils. We have found that in filters containing polyolefin filaments it may be advantageous in some instances to use a slightly heavier wrapper such as a paper of 1.4–1.6 mils thickness.

The invention has been described in detail with particular reference to preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention as described hereinabove and as defined in the appended claims.

We claim:

1. A tobacco smoke filter element comprised of a base of crimped, continuous filaments extending generally longitudinally of the filter element, a content of plasticizer for the aforesaid filaments associated therewith, the filter element being particularly characterized in that it also contains from 5–50% based on the weight of the filter element, of a polyolefin additive composition in the form of cobweb filaments intimately intermingled with the base whereby it is rendered difficult to dislodge the additive from the finished filter element.

2. A filter element in accordance with claim 1 wherein the crimped, continuous filaments are of cellulose acetate and the plasticizer is a plasticizer for cellulose acetate.

3. A filter element in accordance with claim 1 wherein the additive composition includes a content of stearyl alcohol.

4. A filter element in accordance with claim 1 wherein the additive composition includes a content of a paraffin wax.

5. A filter element in accordance with claim 1 wherein the additive composition includes a content of glycerol monostearate.

6. As a new article of manufacture for use in the manufacture of tobacco smoke filter elements a base of longitudinally extending crimped, continuous cellulose acetate filaments comprised of at least 10,000 filaments of less than 16 denier per filament, said cellulose acetate base filaments having 5–50% by weight of the base filaments of polyolefin cobweb type filaments intimately intermingled with the base filaments whereby it is rendered difficult to dislodge the cobweb filaments from the base filaments.

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