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CIGARETTE FILTERS OF PAPER CONTAINING CELLULOSE ACETATE FIBERS

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FIG. 1

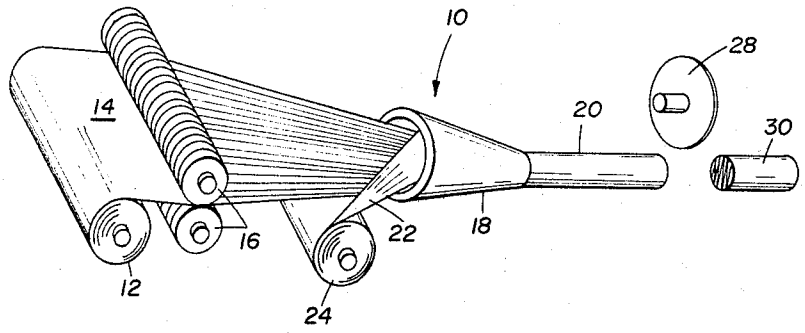
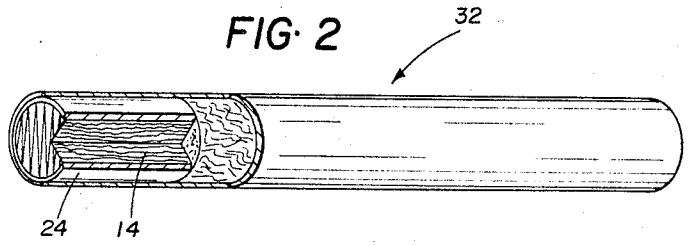


FIG. 2



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CIGARETTE FILTERS OF PAPER CONTAINING CELLULOSE ACETATE FIBERS

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ABSTRACT OF THE DISCLOSURE

A paper cigarette filter for extracting deleterious ingredients including carcinogenic substances. The paper composition of the invention has incorporated therein fibers of acyl esters of cellulose. The paper composition may be masticated to form a larger surface area to effect filtration and in addition separates individual filaments which serve as points of impingement for particulate materials in the cigarette smoke.

This invention relates to a paper cigarette filter, and to the composition of the paper used therein, for extracting from tobacco smoke deleterious ingredients including suspected carcinogenic substances. More specifically this invention relates to the addition of fibers of acyl esters of cellulose to cellulose paper and the processing of such a paper into cigarette filters having improved removal characteristics.

It is well known that tobacco smoke contains various solid and gaseous constituents which are produced by the burning of the smoking tobacco and, in the case of cigarettes, its paper or other type combustible wrapper. Certain of these constituents including phenols have long been suspected as a possible source of certain adverse physiological effects noted by some cigarette smokers. Therefore, a broad research program has been undertaken by the tobacco industry to find some suitable method or process whereby these deleterious constituents can be removed or otherwise eliminated from tobacco smoke without at the same time destroying the desirable flavor and aroma of the smoke. This research effort has led to the development of various types of tobacco smoke filters which are generally attached on the tips of the cigarettes.

Cigarette filters prepared from crepe paper in which, for example, the direction of creping runs parallel to the longitudinal axis of the filter has been among the more successful filters heretofore developed. These filters have been found to be relatively ineffective, however, in removing certain materials such as phenol from the gaseous phase of cigarette smoke. In addition, the commercial paper cigarette filters cause the cigarette smoke to taste dry and hot to many smokers. It is believed that this is due at least in part to the removal of moisture from the smoke by the paper used in the filter. Another limitation of commercial paper filters is their high pressure drop, generally in the range of about 3.0 inches of water, which is very close to the maximum that can be permitted for commercial cigarettes. Thus, the paper filters cannot be wound or otherwise compressed any tighter in the hopes that this will remove appreciably larger quantities of the undesirable tobacco smoke constituents since this would further impede the flow of smoke therethrough to such an extent that the smoking properties of the device would be greatly impaired. If, however, a lower pressure drop were required it becomes necessary to reduce the amount of paper in the filter or alter the geometry of the filter. In either event the hardness or firmness of the filter will, in all probability, be reduced. If the hardness is reduced below a certain point the filters cannot be handled on

automatic processing equipment. In addition, soft filters do not have an acceptable feel in the smoker's mouth.

According to the present invention it has been found that these aforementioned disadvantages of a tobacco smoke filter made of paper can be alleviated by the use of a filter paper having incorporated therein certain synthetic fibers. These synthetic fibers, which are selected from the acylated esters of cellulose, are mixed with the natural cellulose pulp from which the paper is to be formed to give a filter paper having unexpected tobacco smoke constituent removal capabilities.

Therefore, it is an object of this invention to produce a paper for use in tobacco smoke filters which has the capability of removing gaseous or particulate compounds from tobacco smoke.

Another object of this invention is to provide a filter paper having improved elongation and strength factors.

Yet another object of this invention is to provide a filter paper containing fibers of acyl esters of cellulose.

A further object of this invention is to provide a paper cigarette filter that can be bonded to improve the hardness thereof, and has a reduced moisture regain factor.

A still further object of this invention is to produce a paper tobacco smoke filter having improved removal capabilities for phenols, substituted phenols, xylols, cresols and other similar deleterious compounds found in the gaseous and particulate phase of tobacco smoke.

These and further objects and advantages of this invention will be more apparent upon reference to the accompanying specification, specific working examples, claims, and drawings wherein:

FIGURE 1 is a diagrammatical perspective view illustrating one type of paper filter forming machine that can be used to form the cellulose ester containing paper of this invention into tobacco smoke filters; and

FIGURE 2 is a partially sectioned perspective view of a cigarette having a filter tip formed of cellulose ester containing paper attached thereto.

As mentioned briefly hereinabove, it has been found that the filter properties of a paper tobacco smoke filter can be improved by a substantial amount through the use of a filter paper having fibers of an acyl ester of cellulose incorporated therein. These improved results, and especially the extent or degree to which the paper tobacco smoke filter is improved, was totally unexpected in view of what was previously known about synthetic fibers of this type. For example, it has been found that the addition of fibers of cellulose esters to the filter paper reduces the amount of moisture that will be absorbed by the paper. This, as will be apparent, results in the filters removing less moisture from the cigarette smoke and thus presents a cooler smoke to the cigarette smoker. Furthermore, the presence of cellulose ester fibers in the paper filter can be used to improve the firmness or hardness of the filter. This may be accomplished by applying bonding agents for the cellulose ester to the paper prior to its being formed into a filter rod. These bonding agents act to bond the cellulose ester fibers together at random points and thereby hold the filter in a fixed geometry. Thus the use of cellulose ester fibers in paper cigarette smoke filters permits the use of less material without the filter rods becoming too soft to process. This bonding may be effected by a plasticizer for the cellulose esters as can be seen in Example 3, wherein glyceryl triacetate acts to plasticize the cellulose acetate filaments present in the web and bond them together at random points.

The fibers which have been found to give especially goods results are those acyl esters of cellulose whose acyl groups contain from 1 to about 5 carbons. These esters may contain only one type of acyl group or they may contain mixtures of acyl groups such as cellulose acetate, cellulose propionate, cellulose butyrate, cellulose

acetate-propionate, and cellulose acetate-butyrate. The degree of acylation may also vary from a very low value to the maximum which would involve the replacement of all available hydroxyls in the cellulose with acyl groups. However, it is preferred that the minimum total acyl content be about 5 to 10 percent.

The cellulose ester fibers employed in this invention are generally of short lengths ranging from about ⅛ to ¾ inch. However, longer or shorter lengths may be used since the length of the fiber will be determined primarily by the ability of the equipment to handle the fibers and the physical property requirements of the paper sheets.

The denier of the fibers used in this invention may vary from about 0.25 to 25 or higher with the range of 1 to 12 denier per filament being preferred since it has been found that for a given cellulose ester fiber concentration the lower denier fibers produce filters with higher phenol removals than does the higher denier per filament fibers. In addition to affecting the removal properties of the filters the denier size of the fibers incorporated into the paper also affect the physical properties of the paper. Contrary to what might be suspected, the heavy denier per filament fibers will produce a weaker, as well as a stiffer, paper than will the low denier per filament fibers.

The amount of cellulose ester fibers that may be incorporated into the paper can vary from as low as 1 percent to about 70 percent by weight since optimum concentrations are affected by fiber denier and to some extent the "acyl" type and content. Furthermore, the optimum concentration for a given paper property will also be affected by fiber denier, length, acyl content and the like. However, it has been found that the gaseous constituent removal capabilities of the filter paper increases with increased cellulose fiber concentration until a maximum is reached after which it then levels off. Thus, the most effective and economical concentration of cellulose ester fibers for removing gaseous phase constituents will generally fall within the range of from 5 to 30 percent by weight.

In the manufacture of the filter paper the selected type and quantity of cellulose esters is added to the mixing vat containing the refined or partially refined natural cellulose or wood pulp. This mixture of wood pulp and cellulose esters is then fibrillated or mixed until a mixture having the proper consistences for forming the desired paper sheet is obtained. This can be readily accomplished since, as is well known in the paper making art, the more the mixture is fibrillated, that is, the longer it is subjected to the action of a beater, the greater the surface area of the fibrils and the tighter will be the interlock between the fibrils when they are formed into a sheet. Paper sheets which have been formed from only moderately fibrillated or unfibrillated cellulose mixtures have an open structure which is substantially absorbent and the paper is relatively easily crumpled and torn apart. In certain instances it may be possible to slurry the cellulose ester fibers with refined wood pulp and then form it into paper sheets.

Various commercially available machines can be used to form the paper sheets into suitable tobacco smoke filters. FIGURE 1 illustrates one such machine **10** for forming a cigarette filter from a reel **12** of paper containing the cellulose ester fibers. The sheet **14** from the reel **12** is fed through a set of grooved rolls **16** where it is masticated. This produces a large increase in available area for filtration and in addition separates from the paper sheet individual filaments which serve as points of impingement for particulate materials in the cigarette smoke. After mastication the paper sheet **14** is fed into a tapering cone or garniture **18** where it is condensed and formed into a cylindrical rope **20**. If desirable a wrapper paper **22** supplied from a reel **24** can be used for enclosing the compressed cellulose ester containing filter paper. The resulting rope **20** is then cut by suitable flying knife mechanism **28** into suitable length tobacco smoke filter units **30**. In the case of cigarettes the filter units **30** are 20 mm. in length and are attached to a cigarette **32** in substantially the manner shown in FIGURE 2.

A further understanding of the invention will be had from a consideration of the following examples that may be used in actual commercial practice and are set forth to illustrate certain preferred embodiments.

Example 1

A 200 gm. blend of 98 percent chemical cellulose and 2 percent ¼" long 3 denier/filament (d./f.) cellulose acetate fibers containing 39.4 percent acetyl was slurried in 10 liters of water and then beaten in a valley laboratory paper beater for 20 minutes. The beater stock was diluted and formed into 8" x 8" hand sheets weighing 0.75 gm. per sheet. Similar sheets were made from 100 percent chemical cellulose and used as a control sample.

The 100 percent pulp paper and the paper containing 2 percent cellulose acetate fibers were made into filter rods as follows. Individual sheets of each paper sample were passed between a set of grooved rolls such as illustrated at **16** in FIGURE 1 which masticated the paper. This produced a large increase in available area for filtration and in addition separated from the paper individual filaments to serve as points of impingement for particulate materials in the cigarette smoke. After mastication the paper sheets were condensed and formed into rods on a garniture similar to that shown at **18** in FIGURE 1 of a cigarette making machine.

The resulting rods were cut into 20 mm. long filter tips and evaluated for pressure drop, tar removal and phenol removal using the following test procedures.

The pressure drop of the filter tips was measured on a pressure drop apparatus which pulled 1050 cc./min. of air through the filter and measuring the pressure drop across the filter in inches of water. The tar removal was determined by a procedure that consists essentially of smoking two (2) cigarettes and collecting the smoke they produced. The tar trapped by the filter was extracted quantitatively and the amount determined by photofluorometric method. The tar present in the trapped smoke was also determined by the same photofluorometric method. The percent tar removal was then determined by the following equation.

$$\text{Percent removal} = \frac{\text{weight tar on filter} \times 100}{\text{weight tar on filter} + \text{weight tar in smoke}}$$

The percent phenol removal was determined by a test in which twenty (20) standard cigarettes equipped with the filter in question were smoked and the phenol in the smoke trapped on a Soka-Floc filter. The phenol from the cigarette filters and the Soka-Floc filter were quantitatively extracted and the amount present determined by gas chromatography using o-chlorophenol as an internal standard.

The 20 mm. filter made from the 100 percent pulp had a pressure drop of 3.9 inches of water with tar and phenol removals of 57 percent and 38 percent respectively. The filter made from the paper containing 2 percent acetate fibers had a pressure drop of 4.3 inches of water with tar and phenol removals of 61 percent and 88 percent respectively.

From the above it will be noted that the paper filter containing the acetate fibers had essentially the same tar removal as the 100 percent pulp control. It will also be noted that the phenol removal of the pure pulp filter was only 38 percent as compared to 88 percent for the filter containing the acetate fibers.

Example 2

A sample of paper containing 40 percent by weight of ¼" long 8 d./f. cellulose acetate fibers having 39.4 percent acetyl was prepared in the manner discussed in Example 1. A 100 percent wood pulp control was also prepared in the same manner. An 8" x 8" sheet of each paper sample weighed 0.75 gm. These two paper samples were made into filters and tested as in Example 1. The

sample containing the cellulose acetate fibers had a pressure drop of 2.9 inches water with tar and phenol removals of 35 percent and 76 percent respectively. The pressure drop, tar and phenol removals of the 100 percent wood pulp sample were 3.1 inches, 44 percent and 40 percent, respectively.

Example 3

A sample of paper containing 60 percent 5/16" long 3 d./f. cellulose acetate fibers with 39.4 percent acetyl was prepared and made into filter rods as described in Example 1 except that the paper was sprayed with 8 percent glyceryl triacetate before being formed into rods. A 100 percent wood pulp control was also prepared and made into rods in the same manner. Both filters were of the same weight. These filter rods were then evaluated for hardness. The sample containing the acetate fibers had a hardness value of 9.1 while the 100 percent wood pulp sample had a hardness value of 15.6. The hardness value is the amount of filter deformation which occurs when the filter is subjected to a given load with the lower values representing a firmer and thus more desirable filter element. This test shows the filter containing the acetate fibers and the glyceryl triacetate are much harder than the 100 percent wood pulp rods even though both weighed the same and were of the same dimensions.

Example 4

A sample of paper weighing 1.25 grams per 8" x 8" sheet was made from a blend of 40 percent refined wood pulp and 60 percent 5/16" long 3 d./f. cellulose acetate having an acetyl value of 39.4. The acetate fibers and wood pulp were mixed together and beaten for 30 minutes in a valley laboratory paper beater.

The beater stock was diluted and formed into 8" x 8" sheets on a Noble and Wood hand sheet machine. A control was made in the same manner except that 100 percent refined wood pulp was used. Both the sheets containing acetate fibers and those from the 100 percent wood pulp were passed between a set of grooved rolls and masticated to increase the area available for filtration. These sheets were then checked for moisture regain by conditioning for 120 hours at 70° F. and 65 percent relative humidity. The moisture regain of the control was 8.20 percent while that for the paper containing the acetate fibers was 7.00 percent which represents a reduction of 15 percent in moisture regain. This reduction would be even greater at higher relative humidities due to differences in the moisture regain of cellulose acetate and cellulose fibers.

Example 5

A sample of paper containing 10 percent 1/4" long 5 d./f. cellulose propionate and 90 percent wood pulp was made in the above described manner along with a control sample of 100 percent wood pulp. The cellulose propionate contained 46 percent propionyl and 3.5 percent acetyl. Both types of paper were then fabricated into filter rods and tested as in Example 1.

The filter rods containing the cellulose propionate fibers had a phenol removal of 75 percent while the phenol removal of the 100 percent wood pulp control was 41 percent.

Example 6

An 8" x 8" sample sheet of paper, which contained 15 percent of 1/4" long 3 d./f. cellulose acetate-butyrate fibers (37 percent butyryl and 13 percent acetyl) and 85 percent refined wood pulp, was made in the manner described in Example 1. A similar 8" x 8" sheet of pure pulp was also made for comparison purposes. Both sheets of paper weighed 1 gram and were passed between grooved rolls to masticate the paper and then made into 100 mm. long x 5 cm. circumference rods using the garniture of a cigarette machine. One-half of an 8" x 8" sheet was used for each 100 mm. rod. The rods were checked for pressure drop and phenol removal by the test described in Example 1.

The filter rod containing the acetate- butyrate fibers had a pressure drop of 2.9 inches water while that for the 100 percent wood pulp control was 3.1 inches of water. The phenol removal of a 20 mm. tip was 83 percent for the acetate-butyrate sample and 42 percent for the 100 percent wood pulp sample.

Example 7

A sample of paper containing 84 percent of chemical cellulose and 16 percent by weight of 1/4" long 3 d./f. cellulose acetate fibers having 39.4 percent acetyl was prepared in the manner described in Example 1. A 100 percent wood pulp control was prepared in the same manner and from the same chemical cellulose. The sample containing the cellulose acetate fibers weighed 4.75 pounds per 500 17" x 22" sheets while the control weighed 5.80 pounds per 500 17" x 22" sheets.

The samples were conditioned for 24 hours at 70° F. and 65 percent relative humidity and then one inch wide x 3" long cut strips were broken on an Instron tensile tester. Seven (7) breaks were made on each sample. The sample containing the acetate fibers had a breaking strength and elongation at break of 4.6 grams and 4.0 percent respectively. The breaking strength and elongation of the control were 0.54 gms. and 2.2 percent respectively.

From the foregoing it is readily apparent that a paper tobacco smoke filter made with the cellulose ester containing paper of this invention has greatly improved removal capabilities for those tobacco smoke constitutes of the phenols, substituted phenols, xylols, cresols or like gaseous and particulate class of matter. Furthermore, paper filters made from this novel filter paper has improved strength and elongation properties. The presence of the cellulose esters in the filter paper also permits the filter to be bonded with a plasticizer type hardening agent which greatly improves the firmness of the filter. The low moisture regain factor of the filter paper is also of great importance to the tobacco smoke filter industry.

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

What is claimed and desired to be secured by the United States Letters Patent is:

1. A tobacco smoke filter having improved gaseous removal and hardness characteristics and including a masticated, plasticized paper medium compressed into a substantially cylindrical form the improvmeent comprising the incorporation within the paper medium of from between 5 and 30 percent by weight of cellulose acyl ester fibrous material in which the acyl group contains from 1–5 carbons and the fiber size is from about 1 to 12 denier per filament.

2. An improved paper tobacco smoke filter according to claim 1 wherein the minimum degree of acylation of the acyl ester of cellulose is about 5 to 10 percent.

3. An improved paper tobacco smoke filter according to claim 2 wherein the cellulose ester fibers in said cellulose ester fibrous material is between 1/8 and 3/4 inch in length.

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