

Singapore

PATENT SPECIFICATION

NO DRAWINGS

906,424



Date of Application and filing Complete Specification: Dec. 5, 1958.

No. 39235/58.

Application made in United States of America (No. 701,310) on Dec. 9, 1957.

Complete Specification Published: Sept. 19, 1962.

Index at acceptance:—Classes 130, C1E(2:3); 15(2), GAX, GB2A1, GC2A(12A10:12B4:13A:13B), GC2C(4:5:7:8:9:11); and 140, G.

International Classification:—A24c. D04j. D06n.

COMPLETE SPECIFICATION

Improvements in or relating to Tobacco Smoke Filter Elements

5 We, EASTMAN KODAK COMPANY, a Company organized under the Laws of the State of New Jersey, United States of America, of 343 State Street, Rochester, New York, United States of America, (Assignees of GEORGE PATRICK TOUEY and ROBERT CHESTER MUM-
10 POWER II), do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to tobacco smoke filtering elements, more particularly for use as cigarette filter tips.

15 It has been found that the efficiency of tobacco smoke filtering elements constructed from a plurality of filaments can be improved by applying an additive thereto. The application of an additive in the form of a powder requires the use of a dusting booth or other special equipment. Moreover, the additive must be firmly bonded to the filaments, particularly in the case of cigarette filter tips, otherwise it may become loose in the smoker's mouth.
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30 In accordance with the present invention there is provided a method of manufacturing tobacco smoke filtering elements comprising the steps of spreading out a plurality of filaments, melting a wax or waxy material having a melting point above 35° C. and spraying it on the surface of the filaments in a particle size of less than 30 microns to form nodes and ridges thereon.

35 In the more general aspects of our invention we have found that certain normally solid additive materials may be converted to a molten state prior to use as an additive. This conversion to a molten condition is preferably carried out in combination with a suitable plasticizer. Then the molten materials comprising the molten additive and plasticizer are incorporated on the filament filter. Upon deposit of the molten materials on the filaments the plasticizer is absorbed by the
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filaments. The aforesaid normally solid material upon cooling solidifies on the surface of the filaments in the shape of fine particles thereby permitting the production of nodes and ridges or the like on the surface of the filaments. Such particles solidified in the form of nodes and ridges as just mentioned are strongly bonded to the filaments so that the particles are not dislodged from the filaments during the normal handling of the filaments in the making of filters or the subsequent handling and use of the filters. This is the situation, even though substantial amounts of the particles are thus applied to the filaments. In other words, even when the filaments contain an amount of the additive greatly in excess of 10% by weight of the filament, such particles applied by the process of the present invention do not sift or powder out of the filter.
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In considering our invention in further detail the description which follows will be generally grouped under four headings, having reference to the solid type of additive which is applied to the filaments by the present invention. The four groups are fatty acid esters, fatty acids, fatty alcohols and waxes.
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75 Considering in detail first the use of high molecular weight fatty acid esters, the product we prefer would be a fatty acid ester which is solid at room temperature (up to 35° C.) but a liquid when warmed, for example, within the range 50°—100° C. The fatty acid ester preferably is one which is completely miscible with any particular plasticizer used in conjunction therewith, said miscibility being in all proportions when the fatty acid ester is in a melted state. The fatty acid ester preferably is one which is substantially insoluble in the particular plasticizer at room temperatures. The high-molecular-weight acid esters of glycerol, ethylene glycol, propylene glycol, polyethylene glycol, and polypropylene glycol, which have melting points above 35
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[Price 4s. 6d.]

and below 100° C., are suitable for this purpose. So also are the acetylated monoglycerides with melting points within this range. In the table below there are listed several of these compounds which are quite

satisfactory, being soluble in cellulose acetate plasticizers when warmed but insoluble in the plasticizers when the solutions are cooled back to room temperature:

Compound	Trade Name	Melting Point °C
Glycerol monostearate	Aldo 28 or 33; Glyco Products, Inc.	57—61
Diethylene glycol stearate	Diglycol Stearate S; Glyco Products, Inc.	54—59
Ethylene glycol monostearate	S—151; Glyco Products, Inc.	57—60
Polyethylene glycol monostearate	S—1045; Glyco Products, Inc.	50—60
Polyethylene glycol monostearate	S—1004; Glyco Products, Inc.	54—57
The triglyceride of 12-hydroxy stearic acid	Hydrogenated castor oil	84
The acetylated monoglyceride from hydrogenated lard	Myvacet (R.T.M.) 5—100; Eastman Kodak Company	42—44
Propylene glycol monostearate	Aldo 25; Glyco Products, Inc.	58—62

The preferred compounds of this group are the monostearates of glycerol and propylene glycol, the acetylated monoglyceride and the hydrogenated castor oil since these compounds are recommended for incorporation into foods and food containers.

The cellulose acetate plasticizers which can be used as hot solvents for spraying these esters onto the cellulose acetate tow are selected from those plasticizers which have been found to be satisfactory for bonding the compressed tow into a firm or rigid filter rod. Suitable plasticizers are as follows: Glycerol di- and triacetates, Glycerol di- and tripropionates, Di - (methoxyethyl) phthalates, Ethyl phthalyl methyl glycollate, Triethyl citrate and mixtures of these plasticizers.

The preferred plasticizers are glycerol triacetate or a mixture of glycerol diacetate and glycerol triacetate. These plasticizers are capable of bonding the cellulose acetate filaments into a firm rod without the application of heat.

In order to obtain a sufficient amount of the high-molecular-weight fatty acid ester on the surface of the filaments to produce a firm and effective filter, it has been found that the solid ester should be dissolved in the plasticizer to an amount not less than 25% and not more

than 80% of the whole. The preferred amount of dissolved high-molecular-weight fatty acid ester is 40—70%. The amount of total additive solution (plasticizer and dissolved ester) that should be sprayed on a tow of filaments will depend on the type of continuous filaments (number and size of filaments and degree of crimps given to the filaments) employed and the ratio of plasticizer to dissolved ester in the spray solution. In order to obtain sufficient amount of the ester particle deposited on a filament bundle, it is usually desirable to add from 20 to 60% of a 50/50 plasticizer-glycerol (or glycol) ester to the filaments before they are converted to a filter plug. This will give from 10 to 30% solid additive on the filaments. In general, it has been found that at least 10% of the ester particles should be distributed on the surface of the filaments before an appreciable improvement in the filter's efficiency can be realized to the fullest extent.

The size of the ester particles which solidify on the tow is governed mainly by the openings in the spray gun. It has been found that with a conventional paint spraying gun particle sizes between 5 and 30 microns are easily obtained. However, with fine spray attachments it is possible to produce sprays

with particle sizes from 0.5 to 10 microns. As a general rule, it is more desirable to use a spraying device which maintains the particle size below 20 microns. In this way an easy drawing filter is assured since relatively large particles deposited on the filaments have a tendency to increase the pressure drop of the filter to an appreciable degree.

In the following examples and descriptions are set forth several of the preferred embodiments using the first groups of solids which are included merely for the purpose of illustration and not as a limitation thereof.

EXAMPLE I.

A 5-foot length of tow of cellulose acetate yarn, containing 10,000 filaments of 8 denier per filament and having an average of 9 crimps per inch along its entire length, was spread out to a width of 12 inches. While in this spread out condition, the tow was sprayed on both sides with a solution consisting of equal parts by weight of glycerol monostearate and glycerol triacetate maintained (within the spray gun) at a temperature of 70° C. This solution was prepared by adding the solid glycerol monostearate to the liquid glycerol triacetate and heating the mixture to 60–70° C. As the glycerol monostearate melted, it dissolved in the glycerol triacetate giving a homogeneous solution.

After the tow was so treated, the combination consisting of 64% cellulose acetate filaments 18% glycerol monostearate and 18% glycerol triacetate (plasticizer) was pulled back into a compact bundle or cord and fed into a cigarette filter plug making machine which paper wrapped it and cut it into the filter rods 90 mm. in length and 25.5 mm. in circumference. After being stored for 20 minutes at 28° C. (82.4° F.) the rods were

quite rigid. Some of the rods were opened and observed under the microscope. This revealed that substantially all of the filaments had a rough and irregular surface due to the fact that the glycerol monostearate had solidified on the tow as a fine powder which was firmly bound to it through the plasticizer. Several of the rods were cut into filter tips, 15 mm. in length. These tips were attached to 10 cigarettes (Standard Brand A) 85 mm. in length which had been shortened by 15 mm. to compensate for the length of the filter. These 10 filtered cigarettes were smoked to butt lengths of 35 mm. on a smoking machine, the smoke which passed through the cigarettes being collected and analyzed for nicotine and tar content. The results are shown in Table 1.

Control filters containing no additive and control filters containing 18% glycerol triacetate (plasticizer) alone were prepared from the same crimped tow described above. These filters were attached to the same Brand A cigarettes shortened by 15 mm. Ten of the cigarettes with the filters contained no plasticizer and ten of the cigarettes with the filters containing 18% glycerol triacetate were also smoked to butt lengths of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results are shown in Table 1.

Ten unfiltered king size cigarettes (Brand A) which were not shortened by 15 mm. were smoked to butt length of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results obtained are shown in Table 1.

TABLE I
Milligrams of Nicotine and Tar Obtained from Smoking Filtered and Unfiltered Cigarettes

	*Av. Pressure Drop. In.	Mg. Tars 10 Cigarettes	Mg. Nicotine 10 Cigarettes	% Tar Reduc- tion due to Filter	% Nicotine Reduction due to Filter
Cigarettes without filters (85 mm. Total length)	3.1	170	29	—	—
Cigarettes with acetate filters (85 mm. Total length)	3.6	150	25	11.8	13.8
Cigarettes with acetate filters containing 18% glycerol triacetate (85 mm. Total length)	3.5	153	26	10.0	10.4
Cigarettes with acetate filters containing 18% glycerol triacetate and 18% glycerol monostearate (85 mm. Total length)	3.9	110	20	35.3	31.0

* Pressure drop expressed as inches of water at an air flow rate of 17.5 ml./sec. through the cigarette.

5 The nicotine and tar reduction values shown in Table 1 show that the filter with the liquid plasticizer (bonding agent) removed slightly less nicotine and tar than the filter without any additive. However, when the glycerol monostearate dissolved in the plasticizer was added to the tow, the efficiency of the filter was increased appreciably. This increase in filtering efficiency was obtained without a substantial increase in the pressure drop of the cigarette.

EXAMPLE II.

15 A 5-foot length of cellulose acetate yarn tow containing 14,000 filaments of 5 denier per filament and having an average of 9 crimps per inch along its entire length was spread out to a width of 12 inches. While

20 in this spread out condition the tow was sprayed on both sides with a solution consisting of 70% hydrogenated castor oil and 30% glycerol triacetate by weight. During the spraying operation, the solution was maintained at 85° C. within the spray gun. After the tow was so treated the combination consisting of 70% cellulose acetate fibres, 21% hydrogenated castor oil, and 9% glycerol triacetate was pulled back into a compact bundle or cord and fed into the cigarette filter plug making machine which paper wrapped it and cut it into filter rods 90 mm. in length and 25.6 mm. in circumference. After being stored for 20 minutes at approximately 30° C., the rods were rigid and could be readily cut into filter tip lengths of 15 mm. These

tips were attached to 10 cigarettes (Brand A) 85 mm. in length which had been shortened by 15 mm. and the cigarettes were smoked to butt lengths of 35 mm. on the automatic smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. Only 102 mg. of tar and 19 mg. of nicotine were found in the smoke from the 10 cigarettes. When these values are compared with those obtained from the unfiltered cigarette (Table 1) it is found that the filter removes 40% more tar and 34.6% more nicotine than the 15 mm. length of tobacco it replaced. The average pressure drop of the filtered cigarettes was 4.0 inches of water. Control filters containing 10%, 20% and 30% glycerol triacetate, re-

5 spectively, were prepared from the same crimped tow in a similar manner. The filters containing 10% plasticizer removed 20% more nicotine and 10% more tars than the 15 mm. tip of tobacco they replaced. The filters containing 10% plasticizer remove 18% more tars and 18% more nicotine while the filters containing 30% plasticizer removed only 15% more tars and 17% more nicotine. 10 The average pressure drops for these three sets of filtered cigarettes ranged from 3.6 to 4.1 inches.

EXAMPLE III.

15 A continuous tow of cellulose acetate containing 14,000 filaments of 5 denier per filament and having an average of 9 crimps per inch along its entire length was spread out to a width of 12 inches. While in this spread condition the tow was pulled through a spray booth wherein it was sprayed on both sides with glycerol triacetate plasticizer (unheated). After this treatment the spread out tow was then pulled through another booth wherein it was sprayed on both sides with glycerol monostearate (m.p. 57—61° C.) maintained within the spray gun at 85° C. As the liquid ester emerged from the gun it formed a mist of fine particles which were uniformly distributed over the surface of the tow.

30 After the tow was so treated the combination consisting of 67% cellulose acetate fibres, 17% glycerol monostearate and 16% glycerol triacetate plasticizer was pulled back into a compact bundle or cord and fed into the filter machine which paper wrapped it and cut it into rods 90 mm. in length and 25.6 mm. in circumference. After being stored for 20

minutes at room temperature the rigid rods were cut into tips 15 mm. in length. These tips were attached to 10 cigarettes (Brand A) which were originally 85 mm. in length but which were shortened to 70 mm. to compensate for the length of the filter. The filtered cigarettes were smoked to butt lengths of 35 mm. on the automatic smoking machine. 45 The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. Only 99 mg. of tar and 16 mg. of nicotine were found in the smoke from the 10 cigarettes. When these values were compared with those obtained from the unfiltered cigarettes (Table 1) it is found that the filter removed 42% more tar and 45% more nicotine than the 15 mm. length of tobacco it replaced. The average pressure drop of the filtered cigarette was 4.1 inches. 55

In accordance with the next description we now describe our invention as carried out with a high-molecular-weight fatty acid material which is normally solid at room temperature. 60

The high-molecular-weight fatty acids which have melting points above 35 and below 100° C. are quite suitable for this purpose. They can be represented by the empirical formulas $C_nH_{2n}O_2$ and $C_nH_{2n-2x}O_2$ wherein n is 10 to 24 and x is an integer from 1—5. 65 The $C_nH_{2n}O_2$ formula represents the saturated fatty acids and the $C_nH_{2n-2x}O_2$ represents the unsaturated fatty acids. In the tables below are listed several of these compounds which are quite satisfactory, being soluble in the cellulose acetate bonding plasticizers when warmed but insoluble in the plasticizers when the solutions are cooled. 70



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Saturated Fatty Acids ($C_nH_{2n}O_2$)

Name	Formula	Melting Point °C.
Lauric	$C_{11}H_{23}COOH$	43—44
myristic	$C_{13}H_{27}COOH$	53—45
palmitic	$C_{15}H_{31}COOH$	62—63
stearic	$C_{17}H_{35}COOH$	68—70

Unsaturated Fatty Acids ($C_nH_{2n-2x}O_2$)

Name	Formula	Melting Point (°C.)
erucic	$CH_3(CH_2)_7CH=CH(CH_2)_{11}COOH$	35
elaidic	$CH_3(CH_2)_7CH=CH(CH_2)_7COOH$	51—52
elaceostearic	$CH_3(CH_2)_3CH=CHCH=CHCH=CH(CH_2)_7COOH$	49—50
angelic	$CH_3CH=C(CH_3)COOH$	45



In addition, the fatty acids may contain substituent groups (hydroxyl, carbonyl, chloro, etc.). However, if the substituting groups on the fatty acid render the acid insoluble in the plasticizer at the desired temperature range of 35—100° C. or if they radically alter its melting point such substituted fatty acids are not satisfactory.

The preferred fatty acids are the straight chain naturally occurring saturated fatty acids such as myristic, palmitic and stearic acid since these acids are inexpensive, completely nontoxic, and have little or no odor.

The plasticizers which can be used as hot solvents for spraying these acid additives onto the filaments are selected from those plasticizers which have been found to be satisfactory for bonding the compressed filaments into a firm or rigid filter rod. Suitable plasticizers are as follows: glycerol di- and triacetates, glycerol di- and tripropionates, di- (methoxyethyl) phthalate, ethyl phthalyl methyl glycollate, triethyl citrate and mixtures of these plasticizers.

The preferred plasticizers are glycerol triacetate or a mixture of glycerol diacetate and glycerol triacetate. These plasticizers are capable of bonding the filament bundle into a firm rod without the application of heat.

In order to obtain a sufficient amount of the high-molecular-weight fatty acid on the surface of the filaments to product a firm and effective filter, it has been found that the solid acids should be dissolved in the plasticizer to an amount not less than 25% and not more than 80% of the whole. The preferred amount of dissolved high-molecular-weight fatty acid is 40—70%. The amount of total additive solution (plasticizer and dissolved acid) that should be sprayed on the filaments will depend on the type of filaments (number and size of filaments) and the degree of crimps given to the bundle employed and the ratio of plasticizer to dissolved acid in the spray solution. In order to obtain sufficient amount of the solid acid particles deposited on a tow, it is usually desirable to add from 20 to 60% of a 50/50 plasticizer-fatty acid solution to the filament bundle before it is converted to a filter plug. This will give from 10 to 30% solid additive on the filaments. In general, it has been found that at least 10% of the solid acid particles must be distributed on the surface of the filaments before the desired improvement in the filter's efficiency can be realized.

The size of the solid acid particles which deposit on the tow is governed mainly by the openings on the spray gun. It has been found that with a conventional paint spraying gun particle sizes between 5 and 30 microns are easily obtained. However, with fine spray attachments it is possible to produce spray with particle sizes from 0.5 to 10 microns. As

a general rule it is more desirable to use a spraying device which maintains the particle size below 20 microns. In this way an easy drawing filter is assured since relatively large particles deposited on the fibres have a tendency to increase the pressure drop of the filter to an appreciable degree.

In the following examples and descriptions are set forth several of the preferred embodiments of this species of our invention.

EXAMPLE IV.

A 5-foot length of tow of cellulose acetate yarn, containing 10,000 filaments of 8 denier per filament and having an average of 9 crimps per inch along its entire length, was spread out to a width of 12 inches. While in this spread out condition, the tow was sprayed on both sides with a solution consisting of equal parts by weight of stearic acid and glycerol triacetate maintained (within the spray gun) at a temperature of 70° C. This solution was prepared by adding the stearic acid to the liquid glycerol triacetate and heating the mixture to 60—70° C. As the acid melted it dissolved in the glycerol triacetate giving a homogeneous solution.

After the tow was so treated, the combination consisting of 62% cellulose acetate filaments, 19% stearic acid and 19% glycerol triacetate (plasticizer) was pulled back into a compact bundle or cord and fed into a cigarette filter plug-making machine which paper wrapped it and cut it into filter rods 90 mm. in length and 25.5 mm. in circumference. After being stored for 30 minutes at 27° C., the rods were quite rigid. Some of the rods were opened and observed under the microscope. This revealed that substantially all of the filaments had a rough and irregular surface due to the fact that the stearic acid had solidified on the filaments as a fine powder which was firmly bound to the filaments. Several of the rods were cut into filter tips, 15 mm. in length. These tips were attached to 10 cigarettes (Standard Brand A) 85 mm. in length which had been shortened by 15 mm. to compensate for the length of the filter. These 10 filtered cigarettes were smoked to butt lengths of 35 mm. on the smoking machine and the smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results are shown in Table 2.

Control filters containing no additive and control filters containing 19% glycerol triacetate (plasticizer) alone were prepared from the same crimped filament starting material described above. These filters were attached to the same Brand A cigarettes shortened by 15 mm. Ten of the cigarettes with the filters containing no plasticizer and ten of the cigarettes with the filters containing 19% glycerol triacetate were also smoked to butt

lengths of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results are shown in Table 2.

Ten unfiltered king size cigarettes (Brand A) which were not shortened by 15 mm. were smoked to butt lengths of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results are shown in Table 2.

TABLE 2

Milligrams of Nicotine and Tar Obtained from Smoking Filtered and Unfiltered Cigarettes

	*Av. Pressure Drop, In.	Mg. Tars 10 Cigarettes	Mg. Nicotine 10 Cigarettes	% Tar Reduction due to Filter	% Nicotine Reduction due to Filter
Cigarettes without filters (85 mm. total length)	3.1	170	29	—	—
Cigarettes with acetate filters (85 mm. total length)	3.6	150	25	11.8	13.8
Cigarettes with acetate filters containing 19% glycerol triacetate (85 mm. total length)	3.5	156	26	8.3	10.4
Cigarettes with acetate filters containing 19% glycerol triacetate and 19% stearic acid (85 mm. total length)	3.9	106	19	37.5	34.5

* Pressure drop expressed as inches of water at an air flow rate of 17.5 ml./sec. through the cigarette.

The nicotine and tar reduction values shown in Table 2 show that the filter with the liquid plasticizer (bonding agent) removed slightly less nicotine and tar than the filter without any additive. However, when the stearic acid dissolved in the plasticizer was added to the tow, the efficiency of the filter was increased appreciably. This increase in filtering efficiency was obtained without a substantial increase in the pressure drop of the cigarette.

EXAMPLE V.

A 5-foot length of cellulose acetate yarn containing 14,000 filaments of 5 denier per filament and having an average of 9 crimps per inch along its entire length was spread out to a width of 12 inches. While in this spread out condition the yarn was sprayed on both sides with a solution consisting of 70% palmitic acid and 30% glycerol triacetate by weight. During the spraying operation, the solution was maintained at 80° C. within the spray gun. After the yarn was so treated the combination consisting of 70% cellulose acetate, 21% palmitic acid, and 9% glycerol triacetate was pulled back into a compact bundle or cord and fed into the cigarette filter plug-making machine which paper wrapped it and cut it into filter rods 90 mm. in length and 25.6 mm. in circumference. After being stored for 20 minutes at approximately 30°

C., the rods were rigid and could be readily cut into filter tip lengths of 15 mm. These tips were attached to 10 cigarettes (Brand A) 85 mm. in length which had been shortened by 15 mm. and the cigarettes were smoked to butt lengths of 35 mm. on the automatic smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. Only 99 mg. of tar and 20 mg. of nicotine were found in the smoke from 10 cigarettes. When these values are compared with those obtained from the unfiltered cigarette (Table 2) it is found that the filter removes 42% more tar and 38% more nicotine than the 15 mm. length of tobacco it replaced. The average pressure drop of the filtered cigarettes was 4.0 inches of water.

Control filters containing 10%, 20%, and 30% glycerol triacetate, respectively, were prepared from the same crimped yarn in a similar manner. The filters containing 10% plasticizer removed 20% more nicotine and 19% more tar than the 15 mm. tip of tobacco they replaced. The filters containing 20% plasticizer removed 18% more tars and 18% more nicotine while the filters containing 30% plasticizers removed only 15% more tars and 17% more nicotine. The average pressure drops for these three sets of filtered cigarettes ranged from 3.6 to 4.1 inches.

EXAMPLE VI.

A continuous tow of cellulose acetate containing 14,000 filaments of 5 denier per filament and having an average of 9 crimps per inch along its entire length was spread out to a width of 12 inches. While in this spread condition the tow was pulled through a spray booth wherein it was sprayed on both sides with glycerol triacetate plasticizer (unheated). After this treatment the spread out filaments were then pulled through another booth wherein it was sprayed on both sides with stearic acid (m.p. 69° C.) maintained within the spray gun at a temperature of 80° C. As the liquid acid emerged from the gun it formed a mist of fine particles which were uniformly distributed over the surface of the tow.

After the tow was so treated the combination consisting of 69% cellulose acetate filaments, 18% stearic acid, and 13% glycerol triacetate plasticizer was pulled back into a compact bundle or cord and fed into the filter machine which paper wrapped it and cut it into rods 90 mm. in length and 25.6 mm. in circumference. After being stored for 20 minutes at room temperature the rigid rods were cut into tips 15 mm. in length. These tips were attached to 10 cigarettes (Brand A) which were originally 85 mm. in length but which were shortened to 70 mm. to compensate for the length of the filter. The filtered cigarettes were smoked to butt lengths 35

mm. on the automatic smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. Only 103 mg. of tar and 19 mg. of nicotine were found in the smoke from the 10 cigarettes. When these values are compared with those obtained from the unfiltered cigarettes (Table 2) it is found that the filter removed 39% more tar and 35% more nicotine than the 15 mm. length of tobacco it replaced. The average pressure drop of the filtered cigarettes was 3.9 inches.

In accordance with the next description we now describe our invention as carried out with a high-molecular-weight fatty alcohol.

In order to product this desired effect in this invention, the dissolved alcohol should be a solid at room temperature (up to 35° C.) but a liquid when warmed (e.g. from 50—100° C.). It preferably is completely miscible with the plasticizer in all proportions when it is in the melted state and is substantially insoluble in the plasticizer at room temperature, preferably below 40° C.

Although any alcohol having a melting point above 35 and below 100° C. is in many instances satisfactory for the operation of this invention it is preferred to use those alcohols which are known to be nontoxic and which can be obtained from natural sources. The fatty alcohols which can be derived from their corresponding naturally occurring esters are ideally suited for this purpose. Stearyl alcohol (octadecanol) and cetyl alcohol (hexadecanol) are examples of such alcohols. Cetyl alcohol, $\text{CH}_3(\text{CH}_2)_{15}\text{OH}$ melts at 49—50° C. and stearyl alcohol $\text{CH}_3(\text{CH}_2)_{17}\text{OH}$ melts at 58—60° C. Both alcohols are soluble in the heated plasticizers or bonding agents for synthetic filaments but substantially insoluble in the plasticizers below 40° C.

In addition, the fatty alcohols may contain substituent groupings (hydroxy, carbonyl, chloro, etc.). However, the substituent group should not alter its melting point or its solubility characteristics to such an extent that these properties would bar its use within the scope of this invention. In other words, the substituted alcohol would be a solid at least up to 35° C. and preferably up to 40° C. or above and preferably be insoluble in the plasticizer at temperatures below 40° C.

The cellulose acetate plasticizers which can be used as hot solvents for spraying the alcohols on the cellulose acetate tow are selected from those plasticizers which have been found to be satisfactory for bonding the compressed tow into a firm or rigid filter rod, and are as already described above.

In order to obtain a sufficient amount of the high-molecular-weight alcohol on the surface of the filaments to produce a firm and effective filter, it has been found that the alcohol should be dissolved in the plasticizer to an

amount not less than 25% and not more than 80% of the whole. The preferred amount of dissolved high-molecular-weight alcohol is 40—70%. The amount of total additive solution (plasticizer and dissolved alcohol) that should be sprayed on the filaments will depend on the type of filaments (number and size of filaments and degree of crimps given to the filaments) employed and the ratio of plasticizer to alcohol in the spray solution. In order to obtain sufficient amount of the alcohol particles deposited on a bundle of filaments, it is usually necessary to add from 20 to 60% of a 50/50 plasticizer-alcohol mixture to the filaments before it is converted to a filter plug. This will give from 10 to 30% solid additive on the filament bundle. In general, it has been found that at least 10% of the solid alcohol particles must be distributed on the surface of the filaments before an appreciable improvement in the filter's efficiency can be realized.

The size of the alcohol particles which deposit on the filaments is governed mainly by the openings on the spray gun. It has been found that with a conventional paint spraying gun, particle sizes between 5 and 30 microns may be obtained. However, with fine spray attachments it is possible to produce sprays with particle sizes from 0.5 to 10 microns. As a general rule, it is more desirable to use a spraying device which maintains the particle size below 20 microns. In this way an easy drawing filter is assured.

In the next examples and descriptions are set forth several of the preferred embodiments of this invention pertaining to the use of alcohols of the class described above.

EXAMPLE VII.

A five-foot length of cellulose acetate continuous filament yarn, containing 10,000 filaments of 8 denier per filament and having an average of 9 crimps per inch along its entire length, was spread out to a width of 12 inches. While in this spread out condition, the tow was sprayed on both sides with a solution consisting of equal parts by weight of stearyl alcohol and glycerol triacetate maintained (within the spray gun) at a temperature of 70° C. This solution was prepared by adding the stearyl alcohol to the liquid glycerol tri-

acetate and heating the mixture to 60—70° C. As the alcohol melted it dissolved in the glycerol triacetate giving a homogeneous solution. After the continuous filament yarn was so treated, the combination consisting of 68% cellulose acetate, 16% stearyl alcohol and 16% glycerol triacetate (plasticizer) was pulled back into a compact bundle or cord and fed into a cigarette filter plug-making machine which paper wrapped it and cut it into filter rods 90 mm. in length and 25.5 mm. in circumference. After being stored for 30 minutes at 27° C., the rods were quite rigid. Some of the rods were opened and observed under the microscope. This revealed that substantially all of the fibres had a rough and irregular surface due to the fact that the stearyl alcohol had solidified on the filaments as a fine powder, which powder was firmly bound to it through the plasticizer. Several of the rods were cut into filter tips, 15 mm. in length. These tips were attached to 10 cigarettes (Standard Brand A), 85 mm. in length which had been shortened by 15 mm. to compensate for the length of the filter. These 10 filtered cigarettes were smoked to butt lengths of 35 mm. on the automatic smoking machine and the smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results are shown in Table 3.

Control filters containing no additive and control filters containing 19% glycerol triacetate (plasticizer) alone were prepared from the same crimped filaments described above. These filters were attached to the same Brand A cigarettes shortened by 15 mm. Ten of the cigarettes with the filters containing no plasticizer and ten of the cigarettes with the filters containing 19% glycerol triacetate plasticizer were also smoked to butt lengths of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results are shown in Table 3.

Ten unfiltered king size cigarettes (Brand A) which were not shortened by 15 mm. were smoked to butt lengths of 35 mm. by means of the smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. The results obtained are shown in Table 3.

TABLE 3

Milligrams of Nicotine and Tar obtained from Smoking Filtered and Unfiltered Cigarettes

	*Av. Pressure Drop In..	Mg. Tars 10 Cigarettes	Mg. Nicotine 10 Cigarettes	% Tar Reduc- tion due to Filter	% Nicotine Reduction due to Filter
Cigarettes without filters (85 mm. total length)	3.1	170	29	—	—
Cigarettes with acetate filters (85 mm. total length)	3.6	150	25	11.8	13.8
Cigarettes with acetate filters containing 19% glycerol triacetate (85 mm. total length)	3.5	156	26	8.3	10.4
Cigarettes with acetate filters containing 16% glycerol triacetate and 16% stearyl alcohol (85 mm. total length)	4.0	112	20	34.0	31.0

* Pressure drop expressed as inches of water at an air flow rate of 17.5 ml./sec. through the cigarette.

The nicotine and tar reduction values shown in Table 3 show that the filter with the liquid plasticizer (bonding agent) removed slightly less nicotine and tar than the filter without any additive. However, when the stearyl alcohol dissolved in the plasticizer was added to the tow, the efficiency of the filter was increased appreciably. This increase in filtering efficiency was obtained without a substantial increase in the pressure drop of the cigarettes.

EXAMPLE VIII.

A five-foot length of cellulose acetate continuous filament yarn containing 14,000 filaments of 5 denier per filament and having an average of 9 crimps per inch along its entire

length was spread out to a width of 12 inches. While in this spread out condition, the yarn was sprayed on both sides with a solution consisting of 70% cetyl alcohol and 30% glycerol triacetate by weight. During the spraying operation, the solution was maintained at 80° C. within the spray gun. After the yarn was so treated, the combination consisting of 75% cellulose acetate, 17.5% cetyl alcohol and 7.5% glycerol triacetate was pulled back into a compact bundle or cord and fed into the cigarette filter plug making machine which paper wrapped it and cut it into filter rods 90 mm. in length and 25.6 mm. in circumference. After being stored for 20 minutes at approximately 30° C., the rods were rigid

and could be readily cut into filter tip lengths of 15 mm. These tips were attached to 10 cigarettes (Brand A) 85 mm. in length which had been shortened by 15 mm. and the cigarettes were smoked to butt lengths of 35 mm. on the automatic smoking machine. The smoke which passed through the cigarettes was collected and analyzed for nicotine and tar content. Only 106 mg. of tar and 20 mg. of nicotine were found in the smoke from the 10 cigarettes. When these values are compared with those obtained from the unfiltered cigarettes (Table 3) it is found that the filter reduces 38% more tar and 31% more nicotine than the 15 mm. length of tobacco it replaced. The average pressure drop of the filtered cig-



arettes was 4.1 inches of water.

Control filters containing 10%, 20% and 30% glycerol triacetate, respectively, were prepared from the same crimped yarn in a similar manner. The filters containing 10% plasticizer removed 20% more nicotine and 19% more tars than the 15 mm. tip of tobacco they replaced. The filters containing 20% plasticizer removed 18% more tars and 18% more nicotine while the filters containing 30% plasticizer removed only 15% more tars and 17% more nicotine. The average pressure drops for these sets of filtered cigarettes ranged from 3.6 to 4.1 inches.

While in the above examples we have shown various combinations of solid materials with various plasticizers, which combinations may be applied to filaments, and the resultant filaments carrying particles of solids are formed into improved filters, there are other combinations which also give good results. For example, we have found a spray composition which may be made up of about 55% triacetin plasticizer and 45% of the molten solids. The molten solids are comprised of 80% Myverol (Registered Trade Mark) and 20% Myvacet (Registered Trade Mark). Myverol is a distilled monoester, such as glycerol monostearate, produced by Distillation Products Industries, Division of Eastman Kodak Company, Rochester, New York. Myvacet is a hydrogenated, acetylated monoglyceride, likewise produced by the same company. By using the combination of Myverol and Myvacet, the former appears to function to some extent as a binder for more strongly bonding the Myverol to the filaments.

While the combinations of plasticizer and waxy type solid additives described above are preferred under a number of circumstances, in the broader aspects of our invention it is possible to use the more conventional and certain lower cost waxy materials. Thus certain vegetable, petroleum, insect, animal, mineral and synthetic waxes may be employed. More specifically, some of these are as follows:

a. *Carnauba Wax*—This is a well-known vegetable wax and consists chiefly (about 75%) of myricyl cerotate, a small quantity of free cerotic acid, and myricyl alcohol.

b. *Paraffin*—This is a petroleum wax obtained from the high boiling fractions of petroleum. It is a mixture of a great number of saturated hydrocarbons. Its physical properties are: melting range 43—66° C.; specific gravity, 0.88—0.92 at ordinary temperatures; acid number, 0; saponification number, 0; soluble in benzene ether, chloroform and turpentine.

c. *Beeswax*—This is a well-known insect wax. It is composed of myricyl palmitate, cerotic and homologous acids, and small amounts of hydrocarbons, cholesterol esters,

ceryl alcohols, pollen and resins.

d. *Spermaceti*—This is a well-known animal wax obtained from the sperm whale. It is composed of cetyl palmitate, cetyl alcohol and numerous esters of high-molecular weight fatty acids.

e. *Montan*—This is a mineral wax obtained by solvent extraction of woody coals or lignite. It is a monohydric ester of a high-molecular-weight acid. The physical properties are: melting point, 72—92° C.; specific gravity, 1.0; acid number, 15—85; saponification number, 58—89; soluble in benzene, chloroform, naphtha, and isopropyl ether.

f. *Acrawax* (Registered Trade Mark)—These waxes are produced and sold by Glyco Products Company in three grades: Acrawax, Acrawax B, and Acrawax C. They are complex nitrogen derivatives of high-molecular-weight fatty acids. The physical properties of these three waxes range as follows: melting point, 95—139° C.; specific gravity, 0.98—1.05; soluble in hot toluene, turpentine and mineral oil.

g. *Castorwax*—This is a hydrogenated castor oil produced and sold by Baker Castor Oil Company. The physical properties are: melting point, 86—88° C.; saponification number, 175—185; acid number, 2; insoluble in most solvents.

h. *Polyolefin Waxes*—These are high-molecular-weight polymers of ethylene or propylene being produced by a number of chemical companies. Their melting points are frequently about 105—110° C. They are soluble in hot benzene and toluene but insoluble in many organic solvents in the cold.

These waxy solids (a—h) may be melted with plasticizer as described above and the melt sprayed as droplets onto the filaments, or the wax may be melted separately and sprayed onto the filaments, the plasticizer being omitted entirely. In some instances, as above described, the plasticizer may be applied separately either before or after the wax droplets are applied to the filaments.

The denier of the filaments may be from 6 to 16, and in certain instances a denier of 3 or below may be employed. The total denier is between 20,000 to 100,000 with 40,000 to 50,000 being particularly useful.

In carrying out our process we prefer to use apparatus that is suitably jacketed or insulated for heat supply and retention. That is, the spray booth wherein the spread-out filaments are sprayed with the molten solid composition would preferably have all of the booth surfaces heated above the melting point of the spray composition. There would, of course, be heated melt pots for the additive material. The conduits, sprays and pumps handling the hot solution are suitably jacketed to retain the heat. By such construction any spray droplets not attaching to the

70

75

80

85

90

95

100

105

110

115

120

125

130

50,000

jacketed



spread-out filaments will not solidify onto the apparatus but are still maintained in a fluid condition. Such fluid may be withdrawn from the lower part of the spray booth and recycled.

As the filaments which have been sprayed leave the spray booth suitable cooling devices may be mounted adjacent the exit so that the sprayed filaments may be cooled thereby causing the sprayed on particles to solidify in the form of nodes or ridges on the surface of the filaments. The various rolls and the like parts which are positioned adjacent the exit for pulling the sprayed filaments through the spray booth are fitted with suitable blades and a collection means so that any solid which deposits on such rolls may be scraped off into the collection means and returned to the melt pots.

It is believed apparent from the foregoing description that we have provided relatively simple and economical procedures for incorporating substantial amounts of additive on to the filament filters. Not only is it possible to satisfactorily apply somewhat larger amounts of additive to the surface of the filaments than heretofore possible by some of the prior art procedures but by the present invention it is possible to cause such additives to be much more firmly attached to the filaments. Since the present invention does not involve the use of dust, there is not only a possible saving in equipment costs but any danger due to the possibility of electrical discharge setting off a dust explosion is avoided.

WHAT WE CLAIM IS:—

1. A method of manufacturing tobacco smoke filtering elements comprising the steps of spreading out a plurality of filaments, melting a wax or waxy material having a melting point above 35° C. and spraying it on the surface of the filaments in a particle size of less than 30 microns to form nodes and ridges thereon.

2. Method according to claim 1, comprising the additional step of applying to the filaments a plasticizer therefor prior to, or simultaneously with, the molten material.

3. Method according to claim 2, wherein the filaments comprise a tow of at least 10,000 continuous cellulose acetate filaments, the plasticizer is a non-toxic plasticizer for the

cellulose acetate and the waxy material is a high-molecular-weight compound which is incorporated in the plasticizer and comprises an ester, acid or alcohol which is normally solid at room temperature but which is miscible with the plasticizer at a temperature above 50° C., whereafter the sprayed filaments are compacted into a bundle, which is wrapped and cut into lengths, the lengths being stored at a temperature greater than 20° C. for at least 20 minutes whereby the plasticizer action on the filaments causes said lengths to assume rigidity.

4. Method according to claim 3, wherein the plasticizer comprises triacetin and has incorporated therein the waxy material in the form of a mixture miscible with said plasticizer, the mixture comprising glycerol monostearate and hydrogenated acetylated monoglyceride.

5. Method according to claim 3 or 4, wherein the plasticizer is from the group consisting of glycerol di- and tri-acetates, glycerol di- and tripropionates, di-(methoxyethyl) phthalate, ethyl phthalyl methyl glycollate, triethyl citrate and mixtures of these plasticizers.

6. Method according to any of the preceding claims 3—5 wherein the plasticizer and high-molecular-weight materials are in about equal proportions.

7. Method according to claim 3, wherein the plasticizer is a glycerol derivative and the compound is a stearic derivative.

8. Method according to any of the preceding claims, wherein the application of the molten materials to the filaments is carried out in a heated environment whereby molten material not depositing on and adhering to the surface of the filaments may be kept in a fluid condition, withdrawn and recycled.

9. Methods of manufacturing tobacco smoke filtering elements substantially as hereinbefore described.

10. Tobacco smoke filtering elements whenever made by the method according to any of the preceding claims.

11. Tobacco smoke filtering elements constructed substantially as hereinbefore described.

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