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G. P. TOUEY ET AL

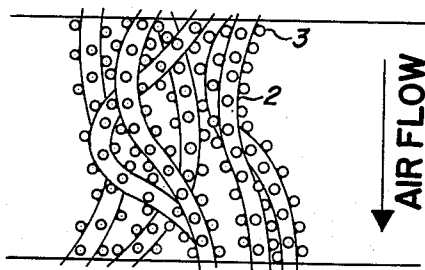
3,008,474

TOBACCO SMOKE FILTER CONTAINING A SUGAR ESTER

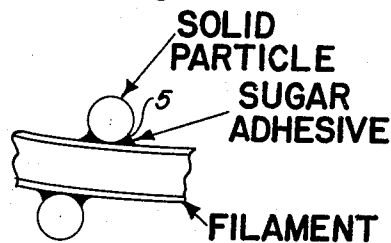
Filed April 13, 1959

*Fig. 1*

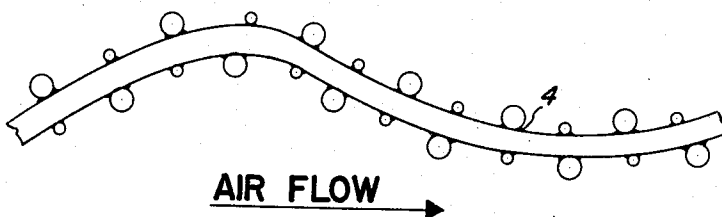
A POWDERED ADDITIVE BONDED TO CELLULOSE ACETATE FILAMENTS BY MEANS OF A LIQUID SUGAR ESTER



*Fig. 3*

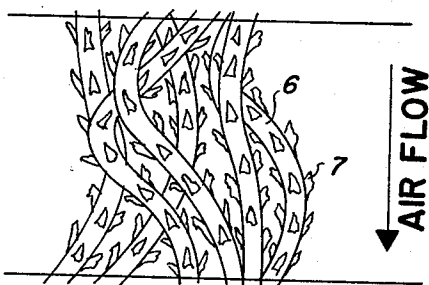


*Fig. 2*

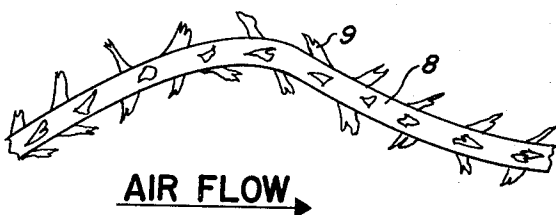


*Fig. 4*

A CRYSTALLINE SUGAR ESTER ADHERING TO CELLULOSE ACETATE FILAMENTS



*Fig. 5*



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3,008,474

## TOBACCO SMOKE FILTER CONTAINING A SUGAR ESTER

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This invention relates to improved tobacco smoke  
filters. More particularly, this invention concerns a  
filter which contains certain sugar esters in its make-up.

Tobacco smoke filters prepared from crimped, con-  
tinuous, man-made filaments have found considerable  
use in the industry. It has been proposed to improve  
such filament filters by applying additives to the surface  
of the filaments making up such filters. Although the  
incorporation of additives onto the surface of the fila-  
ments improves the filters made from such filaments,  
certain problems have arisen. That is, certain of the  
prior procedures for applying additives do not sufficiently  
bond the additive to the filaments with the result that  
the additive may sift out and its effect of improving fil-  
tration efficiency thereby becomes lost. Also, if the ad-  
ditive tends to sift out and thereby may get into the  
smoker's mouth, this may be objectionable.

In other words, since tobacco smoke filters are usu-  
ally held in the smoker's mouth, or if a holder is em-  
ployed, even then the filter is near the smoker's mouth,  
it is apparent that many materials although functioning  
as strong adhesives, because of taste, odor and for other  
reasons, are not suitable for bonding agents for such type  
of filter manufacture. Also, certain materials that have  
adhesive properties exhibit such adhesiveness at the onset  
thereby presenting problems of gumming the filter mak-  
ing equipment and plugging spray equipment.

Therefore, it is thought apparent that the development  
of a material which functions in an improved manner  
as a bonding agent, an additive and the like in the manu-  
facture of tobacco smoke filters represents a highly desir-  
able result. After extended investigation, we have  
found certain sugar esters which are readily compatible  
with the various materials employed in tobacco smoke  
filter manufacture and we believe that our use of such  
esters in the manufacture of tobacco smoke filters is  
new, unobvious and advantageous.

This invention has for one object to provide a better  
method for bonding powdered additives to the filaments  
of a cellulose acetate tow type of cigarette filter. An-  
other object is to provide a way of producing a permanent  
bond between the additive and the filaments without re-  
sorting to the use of adhesives dissolved in volatile sol-  
vents or dissolved in water. Another object is to provide  
a non-tacky bonding agent for use in producing cellulose  
acetate filters containing powdered additives. Still an-  
other object is to provide a method of producing protrus-  
ions on the surfaces of filaments of cigarette filter ma-  
terial without having to dust or spray on a powdered  
additive. A still further object is to provide an additive  
for a textile type filter material which can be applied to  
the filaments as a liquid and after being spread on the  
filaments as a liquid it will gradually crystallize forming  
bumps or protrusions on the surfaces of the longitudi-  
nally aligned filaments after the filter is prepared. A  
further object is to disclose a textile tow type of ciga-  
rette filter containing finely divided solid particles on its  
filament surfaces, said particles showing no tendency to

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sift out of the filter when it is tapped. Other objects  
will appear hereinafter.

In the broader aspects of the present invention, we  
have found that certain materials which, for convenience  
of reference, we will generically refer to as sugar esters,  
may be used in various ways in the manufacture of  
tobacco smoke filters. These sugar ester materials which  
will be described in more detail hereinafter, not only  
function to strongly bond additives, but are compatible  
with existing materials used in filter manufacture and are  
not objectionable from the standpoint of taste and odor  
and are otherwise advantageous.

For assistance in a better understanding of this inven-  
tion, reference may be made to the attached drawing  
forming a part of the instant application.

FIG. 1 is a schematic view on an enlarged scale of a  
bundle of filaments carrying a powdered additive in ac-  
cordance with one embodiment of the present invention;

FIG. 2 likewise is a view on an enlarged scale of a  
single filament for better illustrating the bonding of the  
additive;

FIG. 3 is a detail view of a section of a single filament  
for further showing in a magnified manner the bonding  
of the additive;

FIG. 4 is a view similar to FIG. 1 but of another em-  
bodiment in accordance with the present invention  
wherein the sugar ester comprises the additive; and

FIG. 5 is a view on an exaggerated scale of a single  
filament for further illustrating the embodiment of  
FIG. 4.

Referring to FIG. 1, a continuous crimped cellulose  
acetate filament is shown at 2. Such filaments would  
carry a powdered additive 3 on the surface of the fila-  
ment and bonded thereto. The bonding of the additive  
to the filament is better shown in FIGS. 2 and 3 where  
the sugar ester bonding agent of the present invention  
is indicated at 4 and 5.

FIG. 4 illustrates an embodiment described later on  
in the instant specification wherein the sugar ester itself  
crystallizes out on the surface of the filament thereby  
comprising the additive. That is, in FIG. 4 the cellulose  
acetate filament is shown at 6 and the surface of such  
type filaments carries the sugar ester additive as illus-  
trated at 7. This is shown on a somewhat larger scale  
in FIG. 5 where the carrier filament is designated 8 and  
the sugar ester additive which crystallizes out is desig-  
nated 9.

One method of operation is accomplished by spraying  
a spread out cellulose acetate filter tow with a solution  
consisting of 10-80% plasticizer and 90-20% of a sugar  
ester of the present invention prior to the application of  
a powdered additive. The plasticizer is chosen from one  
of the organic plasticizers already mentioned in prior  
patents as being capable of producing a rigid cellulose  
acetate tow filter. The sugar ester to be dissolved in  
the plasticizer preferably is a noncrystallizing highly vis-  
cous organic solvent soluble derivative capable of pro-  
ducing a viscous tacky liquid film. Mixed aliphatic acid  
esters of glucose and sucrose of the present invention are  
typical noncrystallizing sugar esters. The following are  
further examples of such esters:

Glucose acetate propionate  
Glucose acetate isobutyrate  
Glucose propionate isobutyrate  
Sucrose acetate propionate  
Sucrose acetate isobutyrate  
Sucrose propionate isobutyrate

These mixed esters are unlike the unmixed completely esterified acetates, propionates and isobutyrate of glucose and sucrose which are well defined crystalline compounds. These esters are unique in their ability to remain as highly viscous liquids for an indefinite period of time at ordinary temperatures. Such "noncrystallizing" highly viscous esters of sucrose and glucose are described in the Touey and Davis U. S. Patent No. 2,931,802. They are extremely viscous liquids at room temperature but form non-tacky, nonvolatile and sprayable solutions when dissolved in the plasticizers used as bonding agents for cellulose acetate tow filters. This non-tacky property of such solutions can be obtained even though the sugar ester content of the combination is as high as 80%.

Thus, with this new method for firmly bonding powders to the filament surfaces in a cellulose acetate tow filter, one does not need to evaporate off water or an organic solvent. The plasticizer-sugar ester solution is simply sprayed on the tow prior to the additive dusting operation. Then the tow is treated with the additive and compacted into a paper-wrapped filter in the usual manner. As the filter is stored, most of the plasticizer dissolves into the filaments in the usual manner. However, most of the sugar ester component of the spraying liquid remains on the surface of the filaments and becomes quite tacky due to the extraction of the plasticizer by the cellulose acetate filament. Thus, a tacky adhesive surface is formed on the filaments after the filter has been fabricated. This tacky surface in turn prevents the additive from sifting out of the filter after most of the plasticizer has been absorbed by the filaments.

A valuable distinction between the plasticizer-sugar ester technique of the present invention and the "adhesive in a volatile solvent" technique for producing a tacky surface for acceptance of the additive lies in the fact that the new method does not produce a tacky surface during the fabrication of the filter. The tacky surface gradually forms after the filter has been prepared and as the plasticizer gradually is absorbed into the cellulose acetate filaments. Therefore, no problems of filter fabrication due to a tacky tow are encountered. Another distinction is that there is no volatile solvent (including water) to be evaporated during the processing of the filter rod. These distinctions are quite important when cellulose acetate tow cigarette filters are prepared in the rapid economical manner described by the Crawford and Stevens Patents 2,794,239 and 2,794,480.

The amount of sugar ester which is added to the tow filaments to achieve adequate bonding of the additive to its surface will depend upon the type and amount of additive employed. Thus, with light powders such as cellulose, starch and the like, an amount between 3 and 10% based on the entire filter combination (without the paper) is sufficient. With the heavier additives such as calcium carbonate, the sugar ester concentration preferably is in the range of 8 and 15%. The particular noncrystallizing sugar ester of this invention does not seem to be critical. All are extremely tacky, highly viscous, nonodoriferous liquids at temperatures below 50° C.

The amount of plasticizer which may be used will depend on two factors: the degree of filter hardness desired and the ability of the plasticizer to reduce the viscosity and tackiness of the spraying solution. In any case, the minimum plasticizer content of the unwrapped filter usually will not be below 5% or above 25%. The preferred range of plasticizer content in the unwrapped filter is between 7 and 18%.

The following examples will further illustrate this species of the invention. In these examples reference is made to "standard cigarettes." These were domestic, unfiltered, king-size (85 mm.) cigarettes chosen from two cartons (400) of the same brand of cigarettes. Their selection was made on the basis of the average weight of the 400 cigarettes. Only those weighing within 2% of the average weight of the 400 cigarettes were used in the smoking tests.

## EXAMPLE 1-A

A crimped (12 crimps/in.), continuous, tow of cellulose acetate filaments containing 12,000 filaments of 5 denier per filament was pulled across a compressed air device which bloomed it out to a width of 12 inches. Immediately after this treatment, the spread out tow was pulled through a spraying chamber where it was sprayed on both sides with a solution consisting of 50 parts glycerol triacetate and 50 parts of sucrose acetate isobutyrate. The filaments were then led through a dusting chamber whereupon they were dusted on both sides with a finely divided activated carbon powder. From the dusting chamber the tow was pulled through a device which compacted it and paper wrapped it into the form of a continuous rod 25.5 mm. in circumference. Finally, this rod was cut into 15 mm. filter lengths.

The paper wrapper was removed from several of the filter tips for analytical purposes. Analysis of these samples showed that the filters consisted of 46% cellulose acetate filaments, 11% of glycerol triacetate, 11% sucrose acetate isobutyrate and 32% activated carbon. The sucrose ester used on this preparation contained 12.7% acetyl ( $\text{CH}_3\text{C}=\text{O}$ ) and 47.4% isobutyryl ( $\text{C}_3\text{H}_7\text{C}=\text{O}$ ).

The remainder of the filter rods were stored for a period of one week at room temperature. After the first 24 hours the rods were considered to be semifirm; however, after 48 hours they were firm due to the action of the glycerol triacetate on the cellulose acetate filaments. After a storage time of one week, it was not possible to dislodge the carbon particles from the ends of the filters by tapping them vigorously against a smooth hard surface. Normally the activated carbon particles might have sifted out of the filters within 24 hours after their preparation if certain other materials had been used as the bonding agent.

Ten of the 15 mm. filters were attached to the standard cigarettes which had been shortened by 15 mm. These cigarettes were smoked to 30 mm. butt lengths on an automatic smoking machine which pulled a 35 ml. puff of 2 seconds duration at the rate of one puff per minute. The main stream smoke from the ten cigarettes was analyzed for volatile aldehydes by the method described by G. P. Touey in "Analytical Chemistry," vol. 27, page 27 (1955). Another set of ten filtered cigarettes was prepared and smoked in the manner described and in this case, the main stream smoke was analyzed for tar and nicotine content. The results of both of these tests are shown in Table I.

## EXAMPLE 1-B

A crimped (12 crimps/in.) continuous tow of cellulose acetate filaments containing 16,000 filaments of 5 denier per filament was bloomed out to a width of 12 inches. The spread out tow was then sprayed on both sides with glycerol triacetate. After the spraying operation, it was processed into 15 mm. filters in the manner described above.

Within 24 hours, the filters were quite firm. Ten of these filters were attached to the standard cigarettes which had been shortened by 15 mm. and the cigarettes were automatically smoked to 30 mm. butt lengths. The main stream smoke from the ten cigarettes was analyzed for volatile aldehydes. Another set of ten filtered cigarettes was prepared and smoked in the manner described and in this case, the main stream smoke was analyzed for tar and nicotine content. The results of both of these tests are shown in Table I.

## EXAMPLE 1-C

Ten of the unshortened standard cigarettes containing no filters were smoked to 30 mm. butt lengths and the main stream smoke was analyzed for volatile aldehydes. Another 10 of the unshortened standard cigarettes were smoked in a similar manner and the main stream smoke was analyzed for tar and nicotine. The results of these tests are shown in Table I.

Table I

Cigarettes	Average Cigarette Pressure Drop	Mg. Volatile Aldehydes From 10 Cigarettes	Mg. Tar From 10 Cigarettes	Mg. Nicotine From 10 Cigarettes	Aldehydes Removed by Filter, percent	Tar Removed by Filter, percent	Nicotine Removed by Filter, percent
Example 1-C (no filter)-----	3.1	10.9	170	26	-----	-----	-----
Example 1-B (filter without activated carbon)-----	3.8	10.3	140	21.5	0.5	18	17
Example 1-A (filter containing activated carbon)-----	4.1	7.9	119	19.0	26	30	27

## EXAMPLE 2

This example illustrates the inability of plasticizer alone to bond the activated carbon to the tow in a manner as firmly as the bonding of the present invention.

A crimped (12 crimps/in.) continuous tow of cellulose acetate filaments containing 12,000 filaments of 5 denier per filament was bloomed out and sprayed on both sides with glycerol triacetate. Then it was dusted immediately and on both sides with the same brand of activated carbon powder as used for Example 1. The dusted tow was then converted into filters in the manner described in Example 1. The filters consisted of 51% cellulose acetate fibers, 23% glycerol triacetate plasticizer and 26% carbon powder, based on the total combination without the paper wrapper. Thus, in this case, the amount of plasticizer in the filters was slightly higher than the total amount of plasticizer and sugar ester in the filters of Example 1.

When first prepared, only a slight trace of carbon could be dislodged from the filters by tapping them against a hard surface. However, after a period of time a significant amount of the carbon powder could be dislodged from the filter in this manner. This condition grew more pronounced as the age of the filters increased.

## EXAMPLE 3

This example illustrates the ability of sucrose acetate propionate to strongly bond powders of starch, calcium carbonate and ground cellulose to cellulose acetate tow filaments in the form of a cigarette filter.

A 30-ft. section of a crimped (12 crimps/in.) cellulose acetate tow of 12,000 filaments of 5 denier per filament was divided into 3 equal lengths. All of the samples were then bloomed out to a width of 12 inches and sprayed on both sides with a solution consisting of 75% sucrose acetate propionate and 25% propylene glycol monoacetate. The first sample was then dusted on both sides with rice starch. The second and third samples were dusted on both sides with calcium carbonate and ground cellulose respectively. All of the samples were then processed into filters in the manner described in Example 1.

Filter	Percent Filaments	Percent Plasticizer	Percent Sucrose Acetate Propionate	Type of Powdered Additive	Percent Powdered Additive
1-----	60	7.0	12	Starch-----	21
2-----	49.7	9.3	16	CaCO <sub>3</sub> -----	25
3-----	63.6	6.4	11	Cellulose-----	19

After a period of one month's storage, there was no indication of loosely bound powders in the filters when they were tapped on a hard surface. Subsequent smoking tests indicated that the filters were capable of removing 32-40% more tars from the smoke of the standard cigarette than the 15 mm. length of tobacco they replaced.

## EXAMPLE 4

The work in Example 3 was repeated. However, in this example, a noncrystallizing sucrose propionate isobutyrate was dissolved in the plasticizer to produce a solution con-

sisting of 63% sucrose propionate isobutyrate and 37% propylene glycol monoacetate. This solution was then sprayed on the tow in place of the solution of Example 3.

Filter	Percent Filaments	Percent Plasticizer	Percent Sucrose Propionate Isobutyrate	Type of Powdered Additive	Percent Powdered Additive
1-----	66.1	5.9	10	Starch-----	18
2-----	53.1	8.9	15	CaCO <sub>3</sub> -----	23
3-----	65.7	5.3	9	Cellulose-----	20

After a period of one month's storage, there was no evidence of loosely bound additive in any of the filters when they were tapped on a hard surface. Subsequent smoking tests indicated that the filters of the present invention removed approximately 1/3 more of the tars from the smoke of the standard cigarette than the 15 mm. length of tobacco they replaced.

In the above description and examples we have shown the use of our sugar esters somewhat in accordance with current tobacco smoke manufacturing procedures. That is, we have shown that the sugar esters are compatible with plasticizers hence may be readily applied to improve the manufacture of additive filters wherein the filaments are sprayed with a bonding agent and then the additive applied to the sprayed filaments. In connection with such use it will be observed that the sugar esters of the present invention are not tacky as initially used and, therefore, do not hamper manufacturing operations. However, our esters become tacky and strongly bond the additive as the filter may be stored for a period.

It has further been found that certain of our sugar esters may be used to accomplish the function of the sugar esters themselves giving the additive effect by giving bumps and protrusions on the sprayed filaments. This further improvement will be further apparent from the description which is now set forth.

This feature may be accomplished by spraying the continuous and crimped tow material with a liquid consisting of a crystallizable sugar ester of the present invention dissolved in an organic solvent. At some point in the continuous operation, the cord or tow is bloomed out to a width of 6-12 inches to expose the separate filaments and while in this spread out condition the solution of the sugar ester is applied to its top and bottom sides by means of conventional spray guns. Then the sprayed tow is pulled back together and is pulled through that section of the apparatus which wraps it with a suitable paper and cuts it into filter rods having a circumference equal to that of a cigarette.

After the rods have been prepared, the particular sugar esters of this invention gradually crystallize on the filament surfaces of the filter, forming protrusions which extend into the spaces between the longitudinally aligned filaments. These protrusions function as spots for impingement of part of the smoke particles which would channel through the filter.

The sugar esters for this species of our invention are chosen from the low-molecular-weight aliphatic acid esters of glucose, sucrose, lactose or from the low-molec-

ular-weight aliphatic acid esters of the simple sugar derivatives namely sorbitol,  $\alpha$ -methyl glucoside and  $\alpha$ -ethyl glucoside.

#### GROUP I SUGAR ACETATES

Glucose penta acetate  
 Sucrose octaacetate  
 Lactose octaacetate  
 $\alpha$ -Methyl-D-glucoside tetraacetate  
 $\alpha$ -Ethyl-D-glucoside tetraacetate  
 Sorbitol hexaacetate

#### GROUP II SUGAR PROPIONATES AND ISOBUTYRATES

Glucose penta propionate  
 Glucose penta isobutyrate  
 Sucrose octapropionate  
 Sucrose octaisobutyrate  
 Lactose octaisobutyrate  
 Lactose octapropionate

All of these sugar esters are solid compounds at room temperature and all crystallize rather slowly from organic solvents even when the concentration of the sugar ester in the solvent is quite high. This unique property affords the opportunity of applying them to the tow as liquids which spread over the surfaces of the filaments before the crystals begin to form. This leads to a better distribution of the solid additive to the tow than can be obtained by dusting or spraying on a powdered additive. Also, since the solids form after the filter is made, there is no danger of the solids being displaced from the tow during its conversion to a filter tip.

The amount and type of solvent which is used to prepare the sugar ester solution will now be described. Two types of solvents may be applied. The first type is the volatile solvent. These are usually aliphatic alcohols, esters and ketones. Examples of such solvents are methyl alcohol, ethyl alcohol, isopropyl alcohol, methyl acetate, ethyl acetate, acetone and the like. In the case of the volatile solvent the concentration of the sugar ester can range from 30 to 90%. At the lower concentrations, i.e. 30-60% a good portion of the volatile solvent will evaporate during the spraying operation leaving a viscous solution containing a residual amount of solvent. This residual amount of solvent prevents the sugar from crystallizing. Then, as the filter rod is stored for several hours or days, the residual solvent evaporates and the sugar derivative crystallizes on the filament surfaces.

The second type of solvent is the non-volatile solvent. This type is designed specifically for the cellulose acetate type of textile tow filter described in the Crawford and Stevens patents. It is selected from those type of organic plasticizers which are solvents for the sugar ester and which are also bonding or hardening agents for cellulose acetate tow filters. Examples of such solvents are glycerol di- and triacetate, alkylene glycol acetates, triethyl citrate, di[methoxy ethyl]phthalate, ethyl phthalyl methyl glycolate, acetyl triethyl citrate, glycerol di- and tripropionate and mixtures of such products with high boiling alcohols and glycols. When the non-volatile solvent is used the amount of solvent is more critical. Thus, the solvent concentration preferably is no more than 40%. Expressed in another way, the sugar ester concentration in the solution is high—at least 60%. Preferably the sugar ester concentration should be between 70 and 90%. This is because most of the sugar esters listed above will not crystallize completely out of solution or a non-volatile solvent unless the concentration of dissolved sugar ester is high. Also, if the concentration of non-volatile solvent is high (say more than 40%) an excessive amount thereof would be added to the cellulose acetate tow in order to obtain the desired amount of sugar ester on the tow. This might lead to too much bonding of the filaments into a more or less solid rod.

The amount of sugar ester as just described which is applied to the textile tow to produce a more effective

filter can vary between 5 and 50%. In the case of a fine denier (0.6 to 3 denier per filament) filter only 5-20% of the sugar ester is required. This is because in such a filter the filaments are closer together, therefore, less tar and nicotine can channel through it without touching the filaments. In the case of the filters prepared from the heavier filaments (4-16 denier per filament) then more protrusions into the spaces between the filaments may be desired.

When the cellulose acetate plasticizer type of solvent is used in conjunction with a cellulose acetate textile tow the advantage of using a one spray operation is realized. Previously on incorporating a solid additive into a cellulose acetate tow filter by the dusting technique the tow was sprayed with the bonding agent and then dusted. By this new method of incorporating the solid additive only one operation, spraying operation, is required. This is because the sugar ester and the bonding agent are applied from the one solution.

Also, when the cellulose acetate plasticizer type of solvent is used for the sugar ester, it is desirable to maintain the temperature of the spray solution between 35 and 100° C. This will prevent part of the sugar ester from crystallizing out of solution and clogging the spray gun as it is applied to the opened textile tow.

Aside from being dissolved in a solvent it is also possible to spray the sugar esters in their melted state onto the surface of the textile tow filaments. However, melts of the undiluted sugar esters usually have a viscous, tacky characteristic which may cause the tow to adhere to the filter rod making equipment. Therefore, the preferred method is to use a small amount of diluent.

The following examples will further illustrate this species of the invention. In these examples we have limited the sugar derivatives to a few representative examples of the Groups I and II listed above. In the smoking tests in the examples the same brand of king-size cigarette was used. It was a brand representing a typical domestic type of cigarette tobacco as already discussed above.

#### EXAMPLE 5

This example concerns filters prepared from polypropylene tow containing a sugar ester representing Group I. A continuous crimped (18 crimps/in.) tow of polypropylene fibers containing 12,000 filaments of 5 denier per filament was pulled over a series of air jets which bloomed it out to a width of 12 inches. While in this spread out condition, it was sprayed on both sides with a solution consisting of 50 parts sorbitol hexa acetate and 50 parts ethyl alcohol. After being sprayed, the tow was pulled back together in the form of a rope or cord and led through a device which paper (cigarette paper) wrapped it into the form of a continuous rod with a circumference equal to that of a standard cigarette. The rod was cut into 15 mm. lengths and these were stored at room temperature for 48-hours in an open container. During this time several of the 15 mm. filters were opened and observed under the microscope. After a period of 6 hours, the sugar ester was still visible on the surface of the filaments in the filters as a liquid. However, after 12 hours, crystals of the sorbitol hexaisobutyrate began to form on the filaments and after 24 hours all of the sugar ester had crystallized on the filaments. After 48 hours only a trace amount of ethyl alcohol remained in the filters. Analysis of some of the tips after 48 hours showed that they consisted of 79% polypropylene and 21% sorbitol hexa acetate. These values are based on the total weight of the filter without the paper wrapper. Ten of the tips were placed on 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 one puff per minute. The smoke which passed through

the filtered cigarettes was collected and analyzed for its nicotine and tar content.

Filters (15 mm.) were prepared from a continuous and crimped (18 crimps/in.) tow of polypropylene fibers containing 16,000 filaments of 5 denier per filament. The method of preparing the filters was identical to that described above with the exception that no additive was sprayed on the tow. These filters were also attached to 10 of the shortened king-size cigarettes and the cigarettes were smoked to butt lengths of 30 mm. The smoke which passed through these filtered cigarettes was collected and analyzed for nicotine and tar content.

As a control ten of the unshortened king-size cigarettes were smoked on the automatic machine to butt lengths of 30 mm. The smoke which passed through these unfiltered cigarettes was analyzed for nicotine and tar content.

Filter (15 mm.)	Average (10 cigarettes) pressure drop	Mg. tar from 10 cigarettes	Mg. nicotine from 10 cigarettes
No filter (control).....	3.1	170	28
Polypropylene tow without additive.....	4.0	135	23
Polypropylene tow with sorbitol hexa acetate crystals.....	4.1	110	19

These results show that the polypropylene filter removed 20% more tar and 18% more nicotine than the tobacco it replaced while the polypropylene-sorbitol hexa acetate filter removed 35% more tar and 32% more nicotine than the tobacco it replaced. The results also show that this improvement in filter efficiency cannot be achieved simply by including more fibers in the non-additive type of filter to raise its pressure drop to that of the filter containing the additive.

### EXAMPLE 6

This example concerns filters prepared from viscose tow containing a sugar ester representing Group II. A continuous and crimped (15 crimps/in.) tow of regenerated cellulose filaments (viscose) containing 12,500 filaments of 5 denier per filament was pulled over a series of air jets which bloomed it out to a width of 12 inches. While on this spread out condition it was sprayed on both sides with a solution consisting of 60 parts sucrose octapropionate in 40 parts acetone. After being sprayed the tow was pulled back together and led through a device which paper wrapped it into a continuous rod with the circumference the size of a cigarette. The rod was cut into 15 mm. lengths and these were stored at room temperature for 48 hours in an open container. Microscopic observation of some of the opened filter tips after this time showed that all of the sucrose octapropionate had crystallized on the filaments in the form of needles which protruded out into the spaces between the filaments. Analysis of some of the tips after 48 hours showed that they consisted of 75% regenerated cellulose filaments, 25% sucrose octapropionate and a trace of acetone. These values are based on the total weight of the filter without the paper wrapper. Ten of these tips were placed on king-size 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 filtered cigarettes was collected and analyzed for nicotine and tar content.

Filters (15 mm.) were prepared from a continuous and crimped (15 crimps/in.) tow of regenerated cellulose filaments containing 16,350 filaments of 5 denier per filament. The method for preparing these filters was identical to that described above with the exception that no additive was sprayed on the tow. These filters were also attached to 10 of the shortened king-size cigarettes and the cigarettes were smoked on the automatic machine

to butt lengths of 30 mm. The smoke which passed through these filter cigarettes was collected and analyzed for nicotine and tar content.

Filter (15 mm.)	Average (10 cigarettes) pressure drop	Mg. tar from 10 cigarettes	Mg. nicotine from 10 cigarettes
Regenerated cellulose tow without additive.....	3.8	138	23
Regenerated cellulose tow with sucrose octapropionate crystals..	4.0	113	18

When compared with the results obtained from the unfiltered cigarettes (Example 5) these values show that the regenerated cellulose filter removed 19% more tar and 18% more nicotine than the tobacco it replaced. On the same basis, the regenerated cellulose containing the sucrose octapropionate crystals removed 34% more tar and 36% more nicotine than the tobacco it replaced. The results also show that simply adding more regenerated cellulose filaments to make the filter without the additive equal in pressure drop to that of the regenerated cellulose-sucrose octapropionate filter did not make it as effective as the latter filter.

### EXAMPLE 7

This example concerns filters prepared from cellulose acetate tow containing a sugar ester representing Group II. A continuous and crimped (16 crimps per inch) tow of cellulose acetate filaments (40% acetyl) containing 15,000 filaments of 2 denier per filament was pulled over a series of air jets which bloomed it out to a width of 12-inches. While in this spread out condition, it was sprayed on both sides with a solution consisting of 75 parts sucrose octaisobutyrate and 25 parts glycerol triacetate. After being sprayed, the tow was pulled back together and led through a device which paper wrapped it into a continuous rod with the circumference the size of a cigarette. The rod was cut into 15 mm. lengths and these were stored at room temperature for a total of 5 days. Microscopic observation of some of the opened filter tips after one day of storage revealed that only a few crystals of sucrose octaisobutyrate had formed on the filaments. However, after 4 days the surfaces of the filaments were covered with tiny protrusions of the crystals. Analyses of the tips showed that they consisted of 70% cellulose acetate filaments, 22.5% sucrose octaisobutyrate and 7.5% glycerol triacetate. These values are based on the total weight of the filter without the paper wrapper.

Ten of the rigid tips which had been aged at room temperature for 5 days were placed on the king-size cigarettes which had been shortened by 15 mm. These cigarettes were smoked to butt lengths of 30 mm. on the automatic smoking machine and the smoke which passed through the filter cigarettes was collected and analyzed for its nicotine and tar content.

Filters (15 mm.) were prepared from a continuous crimped (16 crimps/in.) of tow of cellulose acetate (40% acetyl) filaments containing 17,500 filaments of 2 denier per filament. The method for preparing the filters was identical to that described above with the exception that the tow was sprayed with glycerol triacetate alone. The finished filters contained 90% cellulose acetate and 10% glycerol triacetate based on the total weight of the combination without the paper wrapper. After 4 days storage at room temperature, the rigid filters were attached to 10 of the shortened king-size cigarettes and the cigarettes were smoked on the automatic smoking machine to butt lengths of 30 mm. The smoke which passed through these filtered cigarettes was collected and analyzed for nicotine and tar content.

Filter (15 mm.)	Average (10 cigarettes) pressure drop	Mg. tar from 10 cigarettes	Mg. nicotine from 10 cigarettes
Cellulose acetate tow with glycerol triacetate.....	4.2	122	20
Cellulose acetate tow with glycerol triacetate and sucrose octaisobutyrate.....	4.3	95	15

When compared with the results obtained from the unfiltered cigarettes (Example 5) these values show that the cellulose acetate filter removed 28% more tar and 29% more nicotine than the tobacco it replaced. On the same basis, the cellulose acetate containing the sucrose octaisobutyrate crystals removed 44% more tar and 46% more nicotine than the tobacco it replaced. The results also show that adding more filaments to the filter without the sugar ester did not improve its filtering efficiency to the point where it was equal to the filter containing the sugar ester, even though the pressure drops of the two filters were about equal.

In the above description and examples we have in certain instances referred to wrapping the filter rod with a paper wrapper and the paper wrapper which has heretofore been used in the manufacture of tobacco smoke filters may likewise be used in the present invention. However, it has been found that certain of the filters of the present invention may be further improved if they are wrapped in a wrapping paper having 1.4-1.7 mils thickness. Or the filter may be wrapped in plastic sheeting such as the well-known cellophane sheeting of 0.8-1.0 mils thickness.

In further detail, when making filters where the filaments have been sprayed with solutions of the sugar ester and plasticizer, a superior filter may be produced if the filaments are wrapped with the thicker gauge paper or plastic sheeting just mentioned. That is, any tendency of the sucrose octaisobutyrate or sucrose octaisobutyrate triacetate or other plasticizer mixture to soften or moisten paper wrapper of less than 1.4 mils thickness is obviated by the use of the heavier paper or paper-plastic sheeting compositions.

In addition, when utilizing polyolefin filaments, or

particularly highly crimped filaments, the heavier paper or plastic sheeting has been found to better contain the filaments until internal bonding has taken place and also to provide a firmer, stiffer filter.

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 essentially comprised of a bundle made up to a substantial extent of longitudinally extending synthetic filaments of a denier per filament of less than 16, said filaments carrying on the surface of the filaments a content of a sugar ester in an amount greater than 3% by weight of the filter and a wrapper enclosing the periphery of the filter.

2. A filter in accordance with claim 1 wherein the sugar ester is from the group consisting of mixed aliphatic acid esters of glucose and sucrose.

3. A filter in accordance with claim 1 wherein the filaments also carry a content of plasticizer and a powdered additive.

4. A filter in accordance with claim 1 wherein the filaments are comprised of crimped continuous longitudinally extending cellulose acetate filaments.

5. A filter in accordance with claim 1 wherein the wrapper is comprised of paper suitable for wrapping filters and of a thickness greater than 1.4 mils.

#### References Cited in the file of this patent

##### FOREIGN PATENTS

1,051,907	Nelson	Feb. 4, 1913
2,174,541	Walthausen	Oct. 3, 1939
2,215,620	Skumburdis	Sept. 24, 1940
2,664,374	Slayter et al.	Dec. 29, 1953
2,694,661	Meyer	Nov. 16, 1954
2,763,267	Muller	Sept. 18, 1956
2,766,146	Ashburn	Oct. 9, 1956
2,780,228	Touey	Feb. 5, 1957
2,881,770	Touey	Apr. 14, 1959