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PROCESS FOR PRODUCING TOBACCO SMOKE FILTER ELEMENTS FROM CELLULOSE ESTER FILAMENTS CONTAINING POLYETHYLENE GLYCOL

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This invention relates to the production of a tobacco smoke filter. More particularly, it concerns a filter comprising filaments containing a built-in polyalkylene glycol. In our companion U.S. Patent No. 2,904,050 we have disclosed the addition of waxes such as polyalkylene glycol waxes to filament filters as media adhering to the external surfaces of the filaments. In Serial Nos. 185,824 and 185,825, filed on April 9, 1962, inventor Touey has disclosed the adition of certain phenol-removing agents to web-type filters. Although such filters have proved highly satisfactory, difficulties have sometimes been encountered due to possible sloughing off of additives, uneven and non-uniform distribution thereof, softening or weakening of the filter material, channelling of tobacco smoke without impinging thereon, and the like. It is therefore apparent that the production of a filament filter containing a built-in polyalkylene glycol represents a highly desirable result.

It is accordingly one object to this invention to provide a tobacco smoke filter comprising continuous filaments containing embodied therein or built therewithin a polyalkylene glycol. Another object of this invention is to provide a filter tow having uniformly dispersed in the continuous filaments thereof a built-in polyalkylene glycol. Still another object is to provide a method for incorporating polyalkylene glycols substantially homogeneously in a continuous filament tobacco smoke filter. Other objects will appear hereinafter.

In its broader aspects our invention involves the production of a tobacco smoke filter, the continuous filaments of which contain a built-in polyalkylene glycol, by incorporation of said polyalkylene glycol uniformly throughout the tow from which the filter is formed. The polyalkylene glycol is built into the filaments of the tow and the filters produced therefrom by adding it to the spinning solution prior to extrusion of the tow forming filaments. The filaments preferred for dispersion of the polyalkylene glycols substantially evenly therethrough according to our invention are cellulose esters, particularly cellulose acetate. According to our invention the tobacco smoke filters contain filaments which consist of intimate mixtures of the principal filter-forming material, as exemplified by cellulose acetate, with a polyalkylene glycol. The filament filters of our invention preferably contain from about 1–30% by weight of a polyalkylene glycol and from 99 to 70% by weight cellulose acetate as described in our Patent 2,904,050, the optimum amount of polyalkylene glycol being 3–25% by weight and of cellulose acetate 97 to 75% by weight.

The polyalkylene glycols which we have found useful in carrying out our invention include polyethylene glycols which contain 8 to 1,000 polyoxyethylene groups in their chain and polypropylene glycols which contain 8 to 100

polyoxypropylene groups in their chain. Other materials which may be used in other embodiments include mixed polyethers containing both polyoxyethylene and polyoxypropylene groups and esters and ethers thereof, for example, the distearate ester of polyethylene glycol, the alkylphenoxy polyethoxy ethanols and the glycerol ethers of the polyalkylene glycols.

Preferred types of cellulose acetate which may be used in accordance with our invention are those containing an average of 2 to 3 acetyl groups per glucose unit. The cellulose acetate may also contain minor amounts of other acetyl groups such as propionyl or butyl.

While the tow which may be prepared according to my invention may be of a wide range of denier per filament, total denier and crimps per inch, the optimum denier per filament is less than about 9, preferably about 1.6 to 4, total denier less than about 80,000 and crimps per inch at least about 7, preferably 7–16.

In forming the tow by extrusion from a spinning solution, the solvents for the filament-forming material and polyalkylene glycol which may be used include ketones such as acetone and methyl ethyl ketone, chlorinated hydrocarbons and esters such as ethyl acetate and the like. The tow throughout the filaments of which the polyalkylene glycol is embedded may be spun, for example, from a solution under pressure forced through the small holes or orifices of a spinneret. The filaments may be continuously pulled from such a spinneret through a spinning cabinet wherein the solvent is evaporated by means of a hot gaseous medium such as air.

The filaments may then be crimped and bundled together as tow and the tow further processed and cut into filter rods. Additional firmness may be imparted to the filter as desired by treatment with a plasticizer such as glycerol triacetate, triethyl citrate, dimethoxy ethyl phthalate, or the like before being wrapped.

A further understanding of our invention may be obtained by reference to the following examples which are intended to be illustrative only. % refers to percent by weight unless otherwise indicated.

Example I

A spinning solution was prepared consisting of 25% cellulose acetate (39.8% acetyl) and 75% of acetone. The solution was divided into eight 1 kg. samples. One sample was used as a control. A polyglycol was added to each of the other samples. In each case acetone was added with the polyglycol so that the final solution contained 25% nonvolatiles (cellulose acetate+polyglycol). Each solution was heated to 50° C. and mixed for 30 min., then spun into 3.8 denier filament fibers using a cellulose acetate spinning cabinet. Nineteen thousand of the filaments were pulled together to form a tow. The tow was crimped to 16 crimps per inch. It was then compacted into the form of a card and processed into filter rods on a standard cigarette filter rod making machine. The rods which had a circumference of 26.5 mm. were cut into 15 mm. lengths and attached to a domestic brand of cigarettes. The cigarettes were smoked on an automatic smoking machine. The smoke was collected and analyzed for its phenol content according to the method described by Rayburn, Harlan, and Hanmer in Analytical Chemistry,

vol. 25, p. 1419 (1953). The results are listed in the following table.

Run	Composition of Fiber	Phenol Found in Smoke From 100 Filtered Cigarettes, mg.
1	100% Cellulose acetate	10
2	95% Cellulose acetate+5% Polyethylene Glycol (M.W. 4,000).	4
3	90% Cellulose acetate+10% Polyethylene Glycol (M.W. 4,000).	4
4	85% Cellulose acetate+15% Polyethylene Glycol (M.W. 4,000).	3
5	75% Cellulose acetate+25% Polyethylene Glycol (M.W. 4,000).	2
6	90% Cellulose acetate+10% Polyethylene Glycol (M.W. 4,000).	3
7	90% Cellulose acetate+10% Polypropylene Glycol (M.W. 2,000).	5
8	90% Cellulose acetate+10% Condensate of Ethylene Oxide with Polyoxypropylene.	5
9	Unfiltered cigarette	22

Example II

A spinning solution was prepared consisting of 25% cellulose triacetate and 75% solvent. The solvent consisted of 90% methylene chloride and 10% methanol. The spinning solution was divided into 2 portions. Poly (ethylene glycol) (M.W. 600) was added to one portion. Each solution was spun into fibers using a cellulose acetate spinning cabinet. The fibers were converted into cigarette filters and attached to cigarettes as in Example I. The cigarettes were smoked on an automatic smoking machine and the smoke was collected and analyzed for its phenol content. The smoke obtained from 100 cigarettes with the cellulose triacetate filter contained 9 mg. of phenol. The smoke obtained from 100 cigarettes with the filter made from the cellulose triacetate-poly(ethylene glycol) fibers contained only 4 mg. of phenol.

From the foregoing description it will be apparent that by our invention we have provided a polyalkylene glycol-containing tow product which differs from a yarn product in as many ways as and more than yarn differs from tow. For instance, in the case of yarn the individual filaments from a single spinning cabinet shortly after being formed are wound up as yarn on bobbins or the like and thus have imparted thereto at least some twist. On the other hand, in the case of tow, as in this invention, thousands of filaments from several spinning cabinets are combined as crimped tow, the crimping being performed shortly after spinning. Yarn might be described as harsh or stringy in nature, a desirable feature for garment manufacturing purposes, whereas tow, for example, for use in cigarette filters, is comparatively smooth, porous, or cottony in appearance.

Although the invention has been described in considerable detail with particular reference to certain preferred embodiments thereof, 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. In a process for production of tobacco smoke filter elements from cellulose ester continuous filaments, the step which comprises adding to the spinning solution with thorough mixing prior to extrusion of filaments therefrom sufficient polyethylene glycol of a molecular weight of 200–4000 to build in said filaments 5 to 20% of same by weight of the cellulose acetate of the filter elements prepared from said extruded filaments and to impart to said filter elements a phenol removal capability such that they remove substantially twice as much phenol from the smoke of 100 cigarettes smoked containing same as do cellulose acetate filter elements which contain no additive.

2. A process for the production of tobacco smoke filter elements which comprises adding to a spinning solution consisting of about 20–30% by weight of a cellulose acetate containing an average of 2–3 acetyl groups per glucose unit polyethylene glycol having a molecular weight of 200–4000 in an amount of about 1–30% by weight of said spinning solution, heating said solution to temperature of about 40–60° C. and mixing same for about 15 minutes to an hour, spinning said solution through a plurality of orifices in at least one spinneret into an evaporative atmosphere in at least one spinning cabinet, forming a plurality of filaments of about 1.6 to 7 denier per filament containing said polyethylene glycol uniformly dispersed throughout all parts of said filaments, pulling said filaments together to form a tow, crimping said tow to about 7–20 crimps per inch, compacting said tow into the form of a card, processing same into cigarette filter rods of about 2.5 mm. in circumference, and cutting said rods into cigarette filter lengths, thereby building said polyethyleneglycol substantially uniformly into the filaments of said filter rods in an amount of from 5–20% by weight of the cellulose acetate in said filter rods and increasing the ability of said rods to remove phenol from the smoke of cigarettes to which attached when smoked to the extent that substantially half as much phenol is left in the smoke from 100 cigarettes smoked containing same as for the same type cigarette tipped with filters of cellulose acetate not containing such additive.

References Cited by the Examiner

UNITED STATES PATENTS

2,075,890	4/1937	Dreyfus	106—187
2,412,947	12/1946	Brant	106—187
2,780,228	2/1957	Touey	131—208
2,794,239	6/1957	Crawford et al.	161—173
2,801,638	8/1957	Schur et al.	131—208
2,834,704	5/1958	Lawrence	156—180
2,881,085	4/1959	Endicott et al.	106—191
2,953,837	9/1960	Crawford et al.	161—173
2,953,838	9/1960	Crawford et al.	161—173
2,956,329	10/1960	Touey	161—173
3,021,242	2/1962	Touey	156—180
3,080,611	3/1963	Jarrett et al.	264—207
3,141,778	7/1964	Thompson et al.	106—189

OTHER REFERENCES

"Carbowax Compounds and Polyethylene Glycols," publication of Union Carbide, New York, 1946, pp. 4, 6 and 7.

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