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(54) POLYESTER FIBRES

(71) 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 14650, United States of America 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:—

This invention relates to fibres made from amorphous polyesters of terephthalic acid or ester forming derivatives thereof, 1,2-propanediol, and from 0 to 40 mole % of ethylene glycol. These polyester fibres are bondable into coherent tobacco smoke filters when treated with high-boiling plasticizers. Additionally, these polyester fibres, when bonded by high-boiling solvents, are also useful as nonwoven fabrics.

There are numerous publications which disclose the lack of utility of polyesters derived from terephthalic acid and 1,2-propanediol. These polyesters are said to lack utility as fibres because of their "inherently" poor physical properties. For example, Japanese Patent 47941/73 reflects the state of the art regarding poly(1,2-propylene terephthalate), as it states:

"Propylene terephthalate resin, on the other hand, is markedly different from polyethylene terephthalate resin, even though the molecular structure would suggest that they can be treated similarly. Since propylene terephthalate resin is noncrystalline, it is not used to prepare fibres and films, let alone injection-moulded products. Thus, it has not been considered possible to mix it with glass fibres to improve its properties."

Furthermore, United States Patent Specification No. 3,321,437 discloses (in Column 1): "Polyesters obtained, for example, by using 1:2-propylene glycol . . . melt at 122° . . . and are of no value for fibres". These disclosures are illustrative of the state of the art prior to the present invention.

In accordance with the present invention, there is provided a fibre of a polyester of terephthalic acid or an ester forming derivative thereof and a glycol of from 60 mole % to 100 mole % of 1,2-propanediol and from 0 to 40 mole % of ethylene glycol, the polyester having no crystallinity, and having an inherent viscosity of at least 0.35 and a glass transition temperature of at least 80°C.

The fibres of the present invention can be used in the manufacture of tobacco smoke filters and other articles. These fibres exhibit highly advantageous properties in that (1) they are rapidly bondable with small amounts of plasticizers commonly used in the preparation of bonded cigarette filter rods, (2) they can be used to prepare cigarette filter tow with highly acceptable taste characteristics, (3) they exhibit low shrinkage at the high temperatures encountered during cigarette smoking, (4) they can be bonded with plasticizer into rigid coherent structures, (5) they can be commingled with other crystalline polymeric materials and still maintain their advantageous filter capabilities, (6) they can be drawn into very fine deniers which allow better filtration than other filter materials, such as cellulose acetate fibres, (7) the rate at which they can be bonded by plasticizer is higher than the rates for other cigarette filter tow, and (8) they can easily be made into nonwoven articles such as diaper liners which also exhibit highly advantageous physical properties.

The fibres of the present invention can be bonded with various high boiling organic ester plasticizers that are used in the production of cellulose acetate cigarette filter rods. These plasticizers can be plasticizing bonding agents such as glyceryl triacetate (triacetin) mixed with a dialkyl orthophthalate, e.g. diethyl phthalate, dibutyl phthalate and dioctyl phthalate; alkylene glycol and polyalkylene glycol esters of acetoacetic acid, e.g. glyceryl triacetoacetate, ethylene glycol diacetoacetate, propylene glycol diacetoacetate, tetraethylene glycol diacetoacetate, dipropylene glycol diacetoacetate, and glyceryl triacetoacetate; propionic acid esters of tri, tetra and pentaethylene glycol; polyethylene glycol diacetate; and plasticizer blends made from 40 to 60% by weight of glyceryl triacetate and 60 to 40% by weight of a polyethylene glycol diacetate.

The preferred plasticizers are triethylene glycol diacetate and glyceryl triacetate when the fibres are used in cigarette filters, and dimethyl phthalate, 1,3-butanediol diacetate and *p*-butyrolactone when the fibres are used in the preparation of other nonwoven articles. Mixtures of these plasticizers may be used. Bonding can take place at room temperature. The bondability of the fibres is essential in order to give the mass of fibres the rigidity needed for use in cigarette filters and other nonwoven articles.

The polyesters useful in making the fibres of this invention have a glass transition temperature (T_g) of at least 80°C. Since the tobacco end of a cigarette filter reaches a temperature of about 80°C. on the last puff of a cigarette, a glass transition temperature of at least 85°C. is preferred to avoid excessive shrinkage of drafted fibres having no crystallinity. Usually, the glass transition temperature of the polyester will not exceed 96°C.

The preferred polyesters used to prepare the fibres of this invention, which in turn are used to prepare cigarette filters, have glass transition temperatures greater than 85°C. and inherent viscosities of at least 0.5, and the glycol components of the polyesters are entirely 1,2-propanediol. When the fibres are used to form nonwoven webs, the fibres are preferably prepared from polyesters containing from 10 to 30 mole % of ethylene glycol in conjunction with 90 to 70 mole % of 1,2-propanediol (based on the total amount of glycol being 100 mole %). When the fibres of the present invention are utilized for nonwoven applications other than cigarette filter tow, the bonding solvent which is used can be any of a large number of organic solvents such as ethyl acetate, methyl ethyl ketone, methylene chloride, and dimethyl formamide. Preferred solvents, however, are the high boiling solvents already mentioned, because they eliminate the need for expensive solvent recovery systems.

The polyesters that are used to make the fibres of this invention are prepared from terephthalic acid or an ester forming derivative thereof and varying amounts of 1,2-propanediol and ethylene glycol as previously stated. Additionally, from 0 to 0.5 mole % of a trifunctional modifier can be used in the preparation of the polyesters. Typical examples of such modifiers include trimethylol propane, trimethylol ethane, 1,2,3-propanetriol, trimesic acid, trimellitic acid and trimellitic anhydride. It may be desirable to use a trifunctional modifier because of the instability of the secondary hydroxyl group of the 1,2-propanediol. When an appreciable amount (e.g. 30 to 40 mole %) of ethylene glycol is also present, or when the polycondensation temperature is limited to about 230 to 240°C, no trifunctional modifier is required. Tetrafunctional modifiers (e.g. pentaerythritol) or higher functional modifiers may be used, but if a modifier is to be used, the trifunctional types are preferred.

Conventional polyester catalysts can be used to prepare these polyesters. A satisfactory catalyst system contains 130 ppm zinc, which may be added as the diacetate, 28 ppm titanium, which may be added as a tetraalkoxide, and a small amount of phosphorus, which may be added as tri(2-ethylhexyl) phosphate.

The inherent viscosity of the polyesters is at least 0.35 and preferably at least 0.5, but usually it will not exceed 1.2. All inherent viscosities referred to herein were determined at 25°C in solution in a mixture of 60 parts by weight of phenol and 40 parts by weight of tetrachloroethane, at a concentration of 0.5 gram of polyester in 100 ml of the solvent mixture.

Fibres can be prepared from these polyesters by melt spinning, and can be oriented by drafting. Since the fibres are prepared from polyesters which have no crystallinity, i.e. which are amorphous, they are not heat set. These fibres can be drafted (drawn) to two to about five times their original length to give fibres as fine as 0.8 denier/filament. For filter materials, the preferred fibre size range is from

about 0.8 den/fil. to about 8 den/fil. These fibres can then be combined into groups of 5,000 to 20,000 fibres to give total deniers in the range of 20,000 to 50,000. These fibres can then be crimped (3.9 to 11.8 crimps per cm.) and the resulting crimped tows can be used to prepare tobacco smoke filters. A high-boiling plasticizer can be added to the tow in concentrations of 2 to 10% based on the weight of the tow. The fibres are drawn into a garniture and wrapped with paper. Bonding usually occurs in ten minutes or less at room temperature.

The polyester fibres of this invention have several advantages over cellulose acetate fibres for use in cigarette filters. The polyester fibres may be drafted to several times their original length, resulting in fine denier (less than 1 den/fil.) fibres. These fine denier fibres are more effective than larger fibres for filtration of smoke aerosol particles. Some of the polyester fibres can be bonded into firm rods with very small amounts (for example 4% by weight) of bonding plasticizers. Cellulose acetate fibres required approximately twice as much bonding plasticizer for an equivalent bond. The use of less plasticizer reduces the cost, and also less plasticizer will elute into the tobacco smoke from the polyester filters of this invention than will elute from cellulose acetate filters. Additionally, bonding of many of the polyester fibres of this invention is more rapid than bonding of cellulose acetate fibres.

To prepare nonwoven webs useful in textile applications, the polyester fibres of this invention can be converted to a web from tow, or preferably they can be converted to crimped staple lengths of 2.5 to 5 cm. The staple fibres are laid into a mat and from 2 to 10% by weight, based on the weight of the mat, of the bonding ester is sprayed or otherwise applied to the mat. The mat is drawn through rolls or otherwise subjected to pressure to give the necessary contact for bonding. Bonding usually occurs in ten minutes or less, giving a strong nonwoven fabric. There is no need for a drying step, as would be the case when conventional adhesives are used in producing nonwoven fabrics or articles.

When the polyester fibres of this invention are treated with small amounts of plasticizers, for example 3% by weight of glyceryl triacetate, the fibres are bonded instantly into coherent rods when treated with warm air, even though the glass transition temperature of the polymers is in the range of 80 to 95°C. The formation of unwrapped rods in this manner is advantageous for several reasons. The paper wrap may be eliminated, thus reducing the cost of the rods. The filter rods are firm immediately and can be processed and cut more rapidly than conventional filter rods. These unwrapped rods and rods made by conventional processes can be moulded with moderate heat into designs which are very efficient for the filtration of particulate matter from cigarette smoke. Additionally, these polyester fibres can be moulded at surprisingly low temperatures, for example 60°C., which is 60 degrees lower than the moulding temperature for cellulose acetate fibres.

The fibres of this invention can also be utilized to prepare nonwoven webs bondable by plasticizer that are useful as cigarette filter material. This can be accomplished by preparing strong nonwoven webs from mixed staple fibres of a non-crystalline polyester as used in this invention and a crystalline polyester such as poly(ethylene terephthalate). The staple fibres can be blended and converted into a loose batting on a textile carding machine. A small amount (less than 10% by weight) of plasticizer such as triethylene glycol diacetate or an aqueous plasticizer solution is applied to the batting, and the batting is subjected to pressure for 10 minutes, or is processed through rolls heated to about 60—80°C., to form a strong nonwoven web. These coherent webs of staple fibres can then be converted to rigid rods on a cigarette filter-making device with the application of heat or some additional plasticizer. The resulting filters are as efficient as cellulose acetate filters in removing tar and nicotine from tobacco smoke.

This invention will be further illustrated by the following Examples.

Example 1

A mixture of 97 g. (0.5 mole) of dimethyl terephthalate, 114 g. (1.5 moles) of 1,2-propanediol, 0.34 g. of trimethylolpropane (0.0025 mole, 0.5 mole%), 0.04 g. of zinc acetate (130 ppm Zn), 0.8 ml of titanium tetraisopropoxide catalyst solution (28 ppm Ti) and 0.02 g. of tri(2-ethylhexyl) phosphate (colour stabilizer) was placed in a 500 ml flask equipped with a stirrer and a short distillation head with inlet and outlet for nitrogen. The nitrogen outlet, which was connected to a receiver, had provision for applying vacuum. The mixture was stirred at 180—190°C. in a nitrogen atmosphere until the ester interchange was completed (3 hours). A vacuum of 0.5 mm Hg was then applied at 240°C. and stirring was continued for

3.35 hours. A clear very light yellow polymer was obtained. The polymer had an inherent viscosity of 0.57 and a Tg of 96°C. Pressed films of this polymer having a thickness of 0.127 mm were clear and tough, and could be creased without breaking.

Fibres were melt spun (250°C.) and drafted to three times their length. The fibres had the following properties: tenacity 2.3 g./den., elongation 12%, elastic modulus 68 g./den., and flow point 118°C. The fibres were used to prepare cigarette filter rods which were effectively bonded with triethylene glycol diacetate, a bonding agent used to manufacture cellulose acetate filters.

The polymer was soluble in the following solvents (10 g. polymer/90 g. solvent): methylene chloride, propylene chloride, ethylene chloride, and trichloroethylene.

Similar results can be obtained when the trimethylolpropane is replaced with 0.5 mole % of other trifunctional modifiers, such as glycerol, trimesic acid or trimellitic anhydride.

Example 2

This example illustrates that a high molecular weight poly(1,2-propylene terephthalate) can be obtained when no trimethylolpropane is used, if the reaction is carried out at 240°C.

Example 1 was repeated, except that no trimethylolpropane was added to the reaction mixture and a vacuum (0.5 mm Hg) was applied at 240°C. for 3.75 hours. A clear very light yellow polymer having an inherent viscosity of 0.46 and a Tg of 96°C. was obtained. A film 0.127 mm thick pressed at 240°C. was clear, colourless and flexible. Fibres spun at 240°C. and drafted to 3.6 times their length in water at 68°C. had a tenacity of 2.0 g./den., an elongation of 36%, and an elastic modulus of 68 g./den.

Example 3

A crimped tow composed of poly(1,2-propylene terephthalate) (prepared as described in Example 2) consisting of about 14,000 drafted fibres of 3.0 den./fil. and containing 6.3 crimps per cm. was bloomed out (i.e. spread out) with air and then sprayed with 4% by weight of triethylene glycol diacetate. The tow was drawn into a cylindrical device having the same inside diameter as a cigarette (7.85 mm), where it was wrapped with filter rod paper and then cut into lengths of 100 mm. In about 15 minutes these rods were quite rigid because of the curing action of the plasticizer. Filters 20 mm in length were cut from the cured rod and attached to 65 mm cigarette columns. The cigarettes were automatically smoked to 27 mm butt lengths and total particulate matter (TPM) in the smoke was determined. By smoking control cigarettes without filters, the amount of total particulate matter removed by the filters was determined. The efficiency of the filters for removing nicotine was determined by gas chromatography. Results of the tests are shown in Table I.

TABLE I

Cigarettes	TPM in Mg. per Cigarette	Nicotine in Mg. per Cigarette	Percent TPM Removal	Percent Nicotine Removal
No filter	34.0	2.30	0	0
Filter	22.4	1.53	34	33.5

Example 4

Example 3 was repeated except that 4% by weight of glyceryl triacetate was applied to the tow as the bonding agent. The fibres were bonded into coherent firm rods in 20 minutes. Similar efficiency was observed.

Example 5

A crimped tow composed to about 6,000 poly(1,2-propylene terephthalate) fibres of 3.0 den./fil. and about 6,000 poly(ethylene terephthalate) fibres of 3.0 den./fil. commingled together was sprayed with 6% (by weight) of triethylene glycol diacetate. (The poly(1,2-propylene terephthalate) was prepared as described in Example 2). The tow was converted to paper wrapped filter rods by a filter-making device. In approximately 20 minutes, a firmly bonded rod was formed.

The filter rods were tested for efficiency in the following manner: A 20 mm segment, having a pressure drop of 5.3 cm. of water, was attached to a 65 mm cigarette column. The cigarette was smoked to a 27 mm butt length and total

particulate matter (TPM) delivery was determined. By smoking control cigarettes without filters, the amount of total particulate matter removed by the filters was determined. The efficiency of the filters for removing nicotine was determined by gas chromatography. Results of the tests are shown in Table II.

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TABLE II

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	Cigarettes	TPM in Mg. per Cigarette	Nicotine in Mg. per Cigarette	Percent TPM Removal	Percent Nicotine Removal
10	No filter	33.0	2.25	0	0
	Filter	21.8	1.48	34.0	34.0

Example 6

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Example 3 was repeated except that 5% by weight of a 1:1 blend of glyceryl triacetate and triethylene glycol diacetate was sprayed on the tow as the bonding agent. The tow was converted to filters and bonding occurred in approximately 20 minutes. Similar efficiency was observed. When dimethyl phthalate and *p*-butyrolactone are used as bonding agents, similar results can be obtained.

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Example 7

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A tow of undrafted fibres was prepared from a co-polyester having an inherent viscosity of 0.54 and a Tg of 94°C. composed of 100 mole % of terephthalic acid, 28 mole % of ethylene glycol, and 72 mole % of 1,2-propanediol. The tow, consisting of about 13,000 fibres of 3.0 den./fil. containing 5.5 crimps per cm, was sprayed with 4% by weight triethylene glycol diacetate. The treated tow was converted to paper wrapped filter rods by a filter-making device. Filter tips, 20 mm in length, were cut from the rods and attached to 65 mm cigarette columns. The cigarettes were smoked to 27 mm lengths and total particulate matter (TPM) delivery was determined. By smoking control cigarettes without filters, the amount of total particulate matter removed by the filters was determined. The efficiency of the filters for removing nicotine was determined by gas chromatography. Results of the tests are shown in Table III.

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TABLE III

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	Cigarette	TPM in Mg. per Cigarette	Nicotine in Mg. per Cigarette	Percent TPM Removal	Percent Nicotine Removal
35	No filter	34.0	2.35	0	0
	Filter	21.4	1.49	37.0	36.5

Example 8

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Example 6 was repeated except that 4% by weight of glyceryl triacetate was used to bond the tow instead of triethylene glycol diacetate. The filters reached a satisfactory firmness in 20 minutes, and similar filtration efficiency was observed. When dimethyl phthalate and *p*-butyrolactone are used as bonding agents similar results can be obtained.

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Example 9

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A crimped tow consisting of about 15,000 fibres of 3.0 den./fil. and containing 6.3 crimps per cm was prepared from bicomponent fibres. The fibres had an inner core of poly(ethylene terephthalate) and an outer shell of poly(1,2-propylene terephthalate). (The poly(1,2-propylene terephthalate) was prepared as described in Example 2). The tow was bloomed (spread apart) and sprayed with 5% by weight of triethylene glycol diacetate. It was then converted to paper wrapped filter rods by a filter making device. The filter rods were firmly bonded in about 20 minutes.

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Example 10

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A crimped tow consisting of about 15,000 fibres of 3.0 den./fil. and containing 6.3 crimps per cm was prepared from poly(1,2-propylene terephthalate) obtained as described in Example 2. The tow was bloomed and sprayed with 4% by weight of 1,3-butanediol diacetate. It was then converted to filter rods by a filter-making device. The fibres were firmly bonded in about 15 minutes.

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Example 11

A crimped tow consisting of about 14,000 fibres of 3.0 den./fil. and containing

- 7.9 crimps per cm was made from a polyester having an inherent viscosity of 0.49 and a Tg of 96°C., composed of 100 mole % terephthalic acid, 30 mole % of ethylene glycol, and 70 mole % of 1,2-propanediol. The fibres comprising the tow were drafted to 3.5 times their original length after spinning. To prevent shrinkage of the tow when exposed to heat above about 95°C., which is the glass transition temperature of the fibres, the tow was preshrunk at a temperature of 80°C. for 10 minutes. The tow shrank to approximately 80% of its original length. When used as a cigarette filter, no additional shrinkage occurred during the final puffs of smoke.
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- Example 12
- 10 A crimped tow of about 12,000 filaments of 3.0 den./fil. spun from poly(ethylene terephthalate) was bloomed and sprayed with 4% by weight of triethylene glycol diacetate. The treated tow was positioned over a bloomed tow of about 12,000 filaments, 3 den./fil., of poly(1,2-propylene terephthalate) prepared as described in Example 2. The two tows were pressed together. A bond was formed
- 15 between fibres as the bonding solvent partially dissolved the poly(propylene terephthalate), causing the connecting fibres to adhere together at contact points. The excess bonding agent diffused into the fibres, leaving a dry nontacky surface. The nonwoven fabric is useful for clothing insulation, blanket batting and other uses where an insulation material is required.
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- Example 13
- 20 Example 12 was repeated except that two layers of bloomed poly(ethylene terephthalate) tow (12,000 filaments, 3.0 den./fil.) were sprayed with 4% by weight of triethylene glycol diacetate and placed on each side of a bloomed poly(1,2-propylene terephthalate) tow (12,000 filaments, 3.0 den./fil.), forming a three-layer sandwich-type nonwoven fabric. The bonded fabric is useful in the manufacture of clothing, bed coverings, and in other applications.
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- Example 14
- 30 Two sections of bloomed crimped tow of poly(ethylene terephthalate) of about 12,000 filaments (3.0 den./fil.) were sprayed with 4% by weight of triethylene glycol diacetate. A section of tow of about 12,000 filaments (3.0 den./fil.) made from a copolymer having an inherent viscosity of 0.54 and a Tg of 94°C., composed of 100 mole % of terephthalic acid, 28 mole % of ethylene glycol and 72 mole % of 1,2-propanediol was sandwiched between the two treated sections of poly(ethylene terephthalate) tow and pressed firmly together. After 5 minutes, a nonwoven fabric was formed by the action of the bonding solvent on the copolymer causing its filaments to fuse with the other fibres at random points of contact.
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- Example 15
- 40 Example 12 was repeated except that a staple of 5 cm. fibres of 3 den./fil. containing 50% poly(ethylene terephthalate) and 50% poly(propylene terephthalate) was spread out to form a batting 40.64 cm long and 30.48 cm wide. The batting was sprayed with 4% by weight of triethylene glycol diacetate and pressed firmly together for 10 minutes between two flat metal plates. When the plates were removed, the batting was bonded into a nonwoven fabric.
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- Example 16
- 45 Example 14 was repeated except that the staple was composed of 50% of a staple fibre of poly(1,4 - cyclo - hexylenedimethylene terephthalate) and a copolymer staple spun from 100 mole % of terephthalic acid, 28 mole % of ethylene glycol and 72 mole % of 1,2-propanediol. The blend was sprayed with 6% by weight of a bonding blend of 50% triethylene glycol diacetate and 50% glyceryl triacetate. When the batting was pressed firmly together for 10 minutes, a bonded nonwoven fabric was formed.
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- Example 17
- 55 Example 11 was repeated except that dimethyl phthalate was used as the bonding agent.
- Example 18
- 55 Example 11 was repeated except that 6% by weight of glyceryl triacetate was used to bond the fibres together.
- Example 19
- 60 A tow was prepared from a polyester of terephthalic acid (100 mole %), 1,2-propanediol (72 mole %) and ethylene glycol (28 mole %), having an inherent
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viscosity of 0.54 and a Tg of 94°C. The tow, containing approximately 12,800 filaments of 3.6 den./fil., was bloomed and sprayed with 4% by weight of glyceryl triacetate. The treated tow was then drawn through a tubular apparatus (approximately 150 mm in length and 8 mm in diameter) into which air at 85°C. was flowing at the rate of about 23 litres per minute. Residence time in the tube was about one second. The compacted tow emerged from the apparatus as a bonded rod.

Sections of the rod, 20 mm in length, were attached to 64 mm cigarette columns and tested by standard methods used to test cigarette filters for efficiency in removing total particulate matter and nicotine from the smoke. The filters were comparable with commercial cellulose acetate filters of similar fibre size and pressure drop in efficiency for removal of these smoke components. The data are shown below:

Filter	% Plasticizer	Pressure Drop ⁽¹⁾ cm Water	% Total Particulate Matter Removed	% Nicotine Removed
Polyester	3.1	7.62	47.5	47.5
Polyester	6.0	3.56	34.0	35.0
Cellulose Acetate	8.0	7.62	47.0	44.0
Cellulose Acetate	8.0	4.57	36.0	34.0

⁽¹⁾Measured at an air flow of 17.5 ml./sec.

The above experiment was repeated except that cellulose acetate tow was substituted for the polyester tow. The cellulose acetate tow was sprayed with 8% by weight of glyceryl triacetate and drawn through the air curing tube containing air at 95°C. The cellulose acetate tow was not bonded into a rod as was the polyester tow.

Example 20

These examples were carried out using the procedure of Example 18, using the same tow, but with the curing temperature and amount of bonding plasticizer varied. The results are shown in Table IV.

TABLE IV
Copolyester Tow¹ Bonded with Glyceryl Triacetate
and Hot Air

Example	% Glyceryl triacetate	Curing Air Temperature, °C.	Bonded or Not Bonded
a	0	85	Unbonded
b	2	85	Slightly Bonded
c	8	85	Bonded
d	3	75	Bonded
e	8	75	Bonded
f	4	60	Bonded
g	8	50	Slightly Bonded

¹Copolyester tow (3.6 denier/filament, 45,000 total denier, round cross-section) composed of 100 mole % of terephthalic acid, 28 mole % of ethylene glycol and 72 mole % of 1,2-propanediol, having an inherent viscosity of 0.54 and a Tg of 94°C.

Example 21

The procedure of Example 18 was repeated except that a polyester tow made from 100 mole % of terephthalic acid and 100 mole % of 1,2-propanediol was used. The polyester had an inherent viscosity of 0.46 and a Tg of 96°C. The tow contained 12,000 filaments of 3.3 denier/filament. The amount of bonding plasticizer and temperature of the curing air was varied. The results are shown in Table V.

TABLE V
Polyester Tow¹ Bonded with Glyceryl Triacetate
and Hot Air

	Example Number	% Glyceryl triacetate	Curing Air Temperature, °C.	Bonded or Unbonded	
5	a	0	85	Unbonded	5
	b	2	85	Slightly Bonded	
10	c	8	85	Bonded	10
	d	3	75	Bonded	
	e	8	75	Bonded	
	f	3	60	Bonded	
	g	8	60	Bonded	
15	h	8	50	Slightly Bonded	15

¹Polyester tow (3.3 denier/filament 40,000 total denier, round cross-section) composed of 100 mole % of terephthalic acid and 100 mole % of 1,2-propanediol.

20 Example 22
Example 18 was repeated except that 4% by weight of triethylene glycol diacetate was applied as the bonding agent instead of glyceryl triacetate. A firm rod was formed as the compacted tow was drawn through the curing tube injected with air at 85°C. 20

25 Example 23
Example 18 was repeated except that after application of 4% by weight of glyceryl triacetate, the tow was drawn through the curing tube into which steam was injected (approximately 8.2 litres per minute). The compacted tow instantly bonded into a firm rod. Extreme care was taken to keep the actual fibre temperature below 90°C. This was done by regulating the steam flow rate with respect to the linear tow speed. When the fibre temperature was allowed to reach 95°C. the drafted copolyester fibres shrank considerably. This phenomenon is in direct contrast to the behaviour of cellulose acetate fibres. 25

35 Example 24
Example 18 was repeated and firmly bonded filter rods were obtained. The rods were cut to 100 mm lengths and immediately placed in a metal mould at 85°C. which was designed to produce a moulded slanted groove radial flow filter. The polyester rod was moulded into a firm structure having the shape of the mould. 35
The above experiment was repeated except that a cellulose acetate rod bonded with 8% by weight of glyceryl triacetate was placed in a slanted groove filter mould at 85°C. The cellulose acetate could not be moulded at this temperature. 40

45 Example 25
A tow was prepared from a polyester of terephthalic acid (100 mole %), 1,2-propanediol (72 mole %) and ethylene glycol (28 mole %). The polyester had an inherent viscosity of 0.54 and a Tg of 94°C. The tow, containing about 12,800 filaments of 3.6 denier/filament, was converted to rods wrapped with paper at room temperature on a conventional commercial filter rod maker. Four weeks after the filter rods were made, the paper was removed. The rods were placed in a mould designed to produce a moulded slanted groove filter. The mould was heated to 60°C. The filter was moulded to the desired configuration in less than one second. 45

50 Example 26
Poly(1,2-propylene terephthalate) having an inherent viscosity of 0.43 and a Tg of 96°C., prepared as in Example 1, was dried overnight in a vacuum oven at 85 to 90°C. Melt-blown webs of the polymer were prepared as follows. The dried polymer was melt extruded at 265°C. in a 1.27 cm diameter screw extruder through a melt-blown spinneret. Major components in the spinneret were a nozzle which provided a passage for the melt and a housing which formed a pressure equalization chamber for compressed air, which flowed into the chamber at a pressure of 4.22 kg/sq.cm gauge. Air was heated externally to 300°C. and fed to the spinneret at a rate of 56.6 litres (converted to standard conditions) per minute. For minimizing 50

heat loss the spinneret was heated electrically and was maintained at 266°C. As the air left the spinneret assembly, it attenuated the melt into fine fibres and deposited them on to a 16 cm diameter screen drum rotating at a surface speed of 144 cm per second and at the same time reciprocating with a 15.2 cm stroke along its shaft at 41 cycles per minute. The distance from the spinneret to the point of impingement on the drum was 10.2 cm. The fibres were collected for 15 minutes and the resulting web, which was approximately 12 cm wide, was removed by cutting across the width of the web. The length of the web was 50 cm.

The web, sprayed with approximately 4% by weight of glyceryl triacetate, was formed into a firm cigarette filter 20 mm long containing 47 mg of the web. Smoking tests gave 6.35—6.86 cm. water pressure drop and 49.4% removal of total particulate matter. To obtain these levels of pressure drop and total particulate matter removal with a conventional cellulose acetate tow would normally require about 120 mg of the tow.

WHAT WE CLAIM IS:—

1. A fibre of a polyester of terephthalic acid or an ester forming derivative thereof and a glycol of from 60 mole % to 100 mole % of 1,2-propanediol and from 0 to 40 mole % of ethylene glycol, the polyester having no crystallinity, and having an inherent viscosity of at least 0.35 and a glass transition temperature of at least 80°C.

2. A fibre as claimed in Claim 1, wherein the polyester has an inherent viscosity of at least 0.5 and a glass transition temperature of at least 85°C.

3. A fibre as claimed in Claim 1, wherein the ethylene glycol content of the polyester is from 10 to 30 mole % and the inherent viscosity is at least 0.5.

4. A fibre as claimed in Claim 1 and substantially as hereinbefore described.

5. A fibrous article prepared by bonding together with at least one plasticizer a plurality of fibres as claimed in Claim 1.

6. A fibrous article as claimed in Claim 5, wherein the or each plasticizer is selected from triethylene glycol diacetate, glyceryl triacetate, dimethyl phthalate, 1,3-butanediol diacetate, and *p*-butyrolactone.

7. A fibrous article as claimed in Claim 5, in the form of a nonwoven web.

8. A fibrous article as claimed in Claim 5 and substantially as hereinbefore described.

9. A tobacco smoke filter tow comprising a plurality of fibres as claimed in Claim 1.

10. A tobacco smoke filter tow comprising a plurality of fibres as claimed in Claim 2.

11. A tobacco smoke filter tow as claimed in Claim 9 and substantially as hereinbefore described.

12. A tobacco smoke filter rod prepared from a tobacco smoke filter tow as claimed in Claim 9.

13. A tobacco smoke filter rod as claimed in Claim 12, wherein the tow has been bonded with a plasticizer selected from triethylene glycol diacetate and glycerol triacetate or mixtures thereof.

14. A tobacco smoke filter rod comprising a tobacco smoke filter tow as claimed in Claim 9, having no wrapping and having been bonded with steam.

15. A tobacco smoke filter rod comprising a tobacco smoke filter tow as claimed in Claim 10, having no wrapping and having been bonded with steam.

16. A tobacco smoke filter rod as claimed in Claim 12 and substantially as hereinbefore described.

17. A moulded tobacco smoke filter rod comprising a rod as claimed in Claim 14.

18. A moulded tobacco smoke filter rod comprising a rod as claimed in Claim 15.

19. A moulded tobacco smoke filter rod as claimed in Claim 17 and substantially as hereinbefore described.

20. A tobacco smoke filter rod comprising a tobacco smoke filter tow as claimed in Claim 9, having no wrapping and having been bonded with hot air.

21. A tobacco smoke filter rod comprising a tobacco smoke filter tow as claimed in Claim 10, having no wrapping and having been bonded with hot air.

22. A tobacco smoke filter rod as claimed in Claim 20 and substantially as hereinbefore described.

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