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(54) Titre : BRINS A PLUSIEURS FILAMENTS D'ELASTOESTER FILES PAR FUSION

(54) Title: MELT-SPUN ELASTOESTER MULTIFILAMENT YARNS

**(57) Abrégé/Abstract:**

This invention relates to a method of melt-spinning a polyether ester thermoplastic elastomer under commercially viable conditions to produce an elastoester multifilament yarn, wherein the polyether ester thermoplastic elastomer is a polytrimethylene ether ester comprising a polytrimethylene ether dicarboxylate ester soft segment and hard segment selected from a trimethylene dicarboxylate ester and/or a tetramethylene dicarboxylate ester hard segment.



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(54) Title: MELT-SPUN ELASTOESTER MULTIFILAMENT YARNS

(57) Abstract: This invention relates to a method of melt-spinning a polyether ester thermoplastic elastomer under commercially viable conditions to produce an elastoester multifilament yarn, wherein the polyether ester thermoplastic elastomer is a polytrimethylene ether ester comprising a polytrimethylene ether dicarboxylate ester soft segment and hard segment selected from a trimethylene dicarboxylate ester and/or a tetramethylene dicarboxylate ester hard segment.

TITLE

## MELT-SPUN ELASTOESTER MULTIFILAMENT YARNS

FIELD OF THE INVENTION

This invention relates to a method of melt-spinning a polyether ester thermoplastic elastomer under commercially viable conditions to produce an elastoester multifilament yarn, wherein the polyether ester thermoplastic elastomer is a polytrimethylene ether ester comprising a polytrimethylene ether dicarboxylate ester soft segment and hard segment selected from a trimethylene dicarboxylate ester and/or a tetramethylene dicarboxylate ester hard segment.

BACKGROUND OF THE INVENTION

The name "elastoester" is, in the USA, a generic description in the context of a fiber that contains at least 50% by weight of an aliphatic polyether and at least 35% by weight of a polyester. An elastoester is stretchy like a spandex, readily washable, and capable of withstanding high temperatures when wet. Elastoesters are said to retain dyes better than fabrics made of nylon and spandex, and are less likely to be discolored or adversely affected by chlorine, which is an important characteristic for garments like swimming suits.

US6562457 discloses a polyether ester thermoplastic elastomer comprising a polytrimethylene ether ester soft segment and a tetramethylene ester hard segment. US6599625 discloses a polyether ester thermoplastic elastomer comprising a polytrimethylene ether ester soft segment and trimethylene ester hard segment. US6905765 discloses a polyether ester elastomer comprising a poly(trimethylene-ethylene ether) ester soft segment and an alkylene ester hard segment.

The above publications indicate that the polyether esters disclosed therein are useful in making fibers, either continuous filaments or staple fibers, and that the fibers can be used to prepare woven, knit and non-woven fabrics. The fibers disclosed are stretchy, have good chlorine resistance, can be dyed under normal polyester dyeing conditions, and have excellent physical properties including superior strength and stretch recovery properties, particularly improved unload power and stress decay.

US6562457 and US6599625 further indicate that the fibers can be melt spun from the disclosed polymers at speeds up to about 1200 m/min, and can be drawn up to about 6x. Being able to melt spin under these conditions would be a commercial advantage over solution spinning, which is used 5 for spandex or elastane, a manufactured synthetic fiber comprising at least 85% of a segmented polyurethane.

Despite the general disclosure mentioned above, US6562457 and US6599625 only exemplify the melt-spinning of a monofilament at a speed of about 160 m/min with a draw ratio of 4x. These spinning conditions are too 10 slow for practical commercialization of the fibers. Successful commercialization generally requires spinning speeds greater than about 1200 m/min and draw ratios as high as 5x, and further requires that the fibers be in the form of multifilaments.

#### SUMMARY OF THE INVENTION

15 It has now been found that certain polyether ester thermoplastic elastomers can be melt-spun at spinning speeds of greater than about 1200 m/min, and under other commercially feasible conditions, to produce multifilaments (multifilament yarns). The present invention thus provides commercially feasible conditions for melt spinning elastoester fibers comprising poly- 20 trimethylene ether ester polymers that contain polytrimethylene ether dicarboxylate ester soft segments, and trimethylene dicarboxylate ester or tetramethylene dicarboxylate ester hard segments.

In accordance with the present invention, there is provided a process for preparing an elastoester multifilament yarn comprising the steps of:

25 (a) providing a polyether ester thermoplastic elastomer comprising from about 80 to about 40 wt% of a polytrimethylene ether dicarboxylate ester soft segment, and from about 20 to about 60 wt% of a hard segment selected from the group consisting of a trimethylene dicarboxylate ester, a tetramethylene dicarboxylate ester and mixtures thereof, based on the combined weight 30 of the hard and soft segments;

(b) melt spinning the elastomer by extruding it through one or more spinnerets to form at least 2 filaments; and

(c) processing the filaments into a multifilament yarn,

wherein the melt spinning is conducted at a spinning speed of greater than about 1200 meters/minute.

The process of the invention may further comprise the step of cutting the multifilament yarn into staple fibers.

5 The invention also relates to multifilament yarn prepared by the above process, and to fabric comprising the so-prepared multifilament yarn.

The resulting multifilament yarn is an elastoester yarn preferably with an elongation of from about 100 to about 600%, and a tenacity of from about 0.5 to about 2.5 grams/denier, and comprises filaments of from about 0.5 to 10 about 20 denier per filament (dpf).

In a preferred embodiment of the invention the multifilament yarn is spun drawn yarn and the processing comprises drawing the filaments at a draw speed, as measured at the roller at the end of the draw step, of about 1250 to about 5000 meters/minute. Preferably the processing into spun drawn multifilament comprises drawing, annealing, interlacing and winding the filaments.

In another preferred embodiment the multifilament yarn is partially oriented yarn and the spinning speed is preferably greater than about 1500 meters/minute.

20 Multifilament textured yarn may be prepared from the partially oriented yarn by a process comprising: (a) preparing a package of partially oriented multifilament yarn; (b) unwinding the yarn from the package; (c) drawing the filaments to form a drawn yarn; (d) false-twist texturing the drawn yarn to form the textured yarn; and (e) winding the yarn onto a package.

25 **DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS**

All publications, patent applications, patents and other references mentioned herein, if not otherwise indicated, are incorporated by reference herein for all purposes as if fully set forth.

30 Unless otherwise defined, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. In case of conflict, the present specification, including definitions, will control.

Except where expressly noted, trademarks are shown in upper case.

Unless stated otherwise, all percentages, parts, ratios, etc., are by weight.

When an amount, concentration, or other value or parameter is given as either a range, preferred range or a list of upper preferable values and lower preferable values, this is to be understood as specifically disclosing all ranges formed from any pair of any upper range limit or preferred value and any lower range limit or preferred value, regardless of whether ranges are separately disclosed. Where a range of numerical values is recited herein, unless otherwise stated, the range is intended to include the endpoints thereof, and all integers and fractions within the range. It is not intended that the scope of the invention be limited to the specific values recited when defining a range.

When the term "about" is used in describing a value or an end-point of a range, the disclosure should be understood to include the specific value or end-point referred to.

As used herein, the terms "comprises," "comprising," "includes," "including," "has," "having" or any other variation thereof, are intended to cover a non-exclusive inclusion. For example, a process, method, article, or apparatus that comprises a list of elements is not necessarily limited to only those elements but may include other elements not expressly listed or inherent to such process, method, article, or apparatus. Further, unless expressly stated to the contrary, "or" refers to an inclusive or and not to an exclusive or. For example, a condition A or B is satisfied by any one of the following: A is true (or present) and B is false (or not present), A is false (or not present) and B is true (or present), and both A and B are true (or present).

Use of "a" or "an" are employed to describe elements and components of the invention. This is done merely for convenience and to give a general sense of the invention. This description should be read to include one or at least one and the singular also includes the plural unless it is obvious that it is meant otherwise.

The materials, methods, and examples herein are illustrative only and, except as specifically stated, are not intended to be limiting. Although meth-

ods and materials similar or equivalent to those described herein can be used in the practice or testing of the present invention, suitable methods and materials are described herein.

### Polyether Ester Thermoplastic Elastomer

5 The polyether ester thermoplastic elastomers for use in the invention preferably contain:

from about 80 to about 40 wt%, more preferably from about 75 to about 50 wt%, and still more preferably from about 70 to about 60 wt%, polytrimethylene ether ester soft segment; and

10 from about 20 to about 60 wt%, more preferably from about 25 to about 50 wt%, and still more preferably from about 30 to about 40 wt% hard segment (polytrimethylene ester and/or polytetramethylene ester),  
based on the combined weight of the hard and soft segments.

15 The polyether ester thermoplastic elastomers preferably have an inherent viscosity of at least about 0.8 dl/g, more preferably at least about 1.2 dl/g, and preferably up to about 2.0 dl/g, and more preferably up to about 1.6 dl/g.

20 "Polytrimethylene ether ester soft segment" and "soft segment" are used in connection with the present invention to refer to the reaction product of a polymeric ether glycol and a "dicarboxylic acid equivalent", via ester linkage, wherein at least about 50 wt%, more preferably at least about 85 wt%, and still more preferably from about 95 to 100 wt%, of the polymeric ether glycol used to form the soft segment is a polytrimethylene ether glycol (PO<sub>3</sub>G).

25 "Polytrimethylene ester hard segment", "polytetramethylene ester hard segment" and "hard segment" are used in connection with the present invention to refer to the reaction product of one or more of 1,3-propane diol (trimethylene glycol) or 1,4-butane diol (tetramethylene glycol), and one or more dicarboxylic acid equivalents, via ester linkage, wherein greater than about 50 mole%, more preferably at least about 75 mole%, even more preferably at least about 85 mole%, and still more preferably from about 95 to 100

mole%, of the diol used to form the hard segment is 1,3-propane diol and/or 1,4-butane diol.

By "dicarboxylic acid equivalent" is meant dicarboxylic acids and their equivalents, which are compounds that perform substantially like dicarboxylic acids in reaction with polymeric glycols and diols, as would be generally recognized by a person of ordinary skill in the relevant art. In addition to dicarboxylic acids, dicarboxylic acid equivalents for the purpose of the present invention include, for example, mono- and diesters of dicarboxylic acids, and diester-forming derivatives such as acid halides (e.g., acid chlorides) and anhydrides.

#### Polymeric Ether Glycol for Soft Segement

PO3G for the purposes of the present invention is an oligomeric and/or polymeric ether glycol in which at least 50% of the repeating units are trimethylene ether units. More preferably from about 75% to 100%, still more preferably from about 90% to 100%, and even more preferably from about 99% to 100%, of the repeating units are trimethylene ether units.

PO3G is preferably prepared by polycondensation of monomers comprising 1,3-propanediol, thus resulting in polymers or copolymers containing - (CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O)- linkage (e.g, trimethylene ether repeating units).

In addition to the trimethylene ether units, lesser amounts of other units, such as other polyalkylene ether repeating units, may be present. In the context of this disclosure, the term "polytrimethylene ether glycol" encompasses PO3G made from essentially pure 1,3-propanediol, as well as those oligomers and polymers (including those described below) containing up to 50% by weight of comonomers.

The 1,3-propanediol employed for preparing the PO3G may be obtained by any of the various well known chemical routes or by biochemical transformation routes. Preferred routes are described in, for example, US5015789, US5276201, US5284979, US5334778, US5364984, US5364987, US5633362, US5686276, US5821092, US5962745, US6140543, US6232511, US6235948, US6277289, US6297408, US6331264, US6342646, US7038092, US20040225161A1, US20040260125A1, US20040225162A1 and US20050069997A1.

Preferably, the 1,3-propanediol is obtained biochemically from a renewable source (“biologically-derived” 1,3-propanediol).

A particularly preferred source of 1,3-propanediol is via a fermentation process using a renewable biological source. As an illustrative example of a 5 starting material from a renewable source, biochemical routes to 1,3-propanediol (PDO) have been described that utilize feedstocks produced from biological and renewable resources such as corn feed stock. For example, bacterial strains able to convert glycerol into 1,3-propanediol are found in the species *Klebsiella*, *Citrobacter*, *Clostridium*, and *Lactobacillus*. The technique 10 is disclosed in several publications, including previously incorporated US5633362, US5686276 and US5821092. US5821092 discloses, *inter alia*, a process for the biological production of 1,3-propanediol from glycerol using recombinant organisms. The process incorporates *E. coli* bacteria, transformed with a heterologous pdu diol dehydratase gene, having specificity for 15 1,2-propanediol. The transformed *E. coli* is grown in the presence of glycerol as a carbon source and 1,3-propanediol is isolated from the growth media. Since both bacteria and yeasts can convert glucose (e.g., corn sugar) or other carbohydrates to glycerol, the processes disclosed in these publications provide a rapid, inexpensive and environmentally responsible source of 1,3- 20 propanediol monomer.

The biologically-derived 1,3-propanediol, such as produced by the processes described and referenced above, contains carbon from the atmospheric carbon dioxide incorporated by plants, which compose the feedstock for the production of the 1,3-propanediol. In this way, the biologically-derived 25 1,3-propanediol preferred for use in the context of the present invention contains only renewable carbon, and not fossil fuel-based or petroleum-based carbon. The PO3G and elastomers based thereon utilizing the biologically-derived 1,3-propanediol, therefore, have less impact on the environment as the 1,3-propanediol used in the compositions does not deplete diminishing 30 fossil fuels and, upon degradation, releases carbon back to the atmosphere for use by plants once again. Thus, the compositions of the present invention can be characterized as more natural and having less environmental impact than similar compositions comprising petroleum based glycols.

The biologically-derived 1,3-propanediol, and PO3G and elastomers 35 based thereon, may be distinguished from similar compounds produced from

a petrochemical source or from fossil fuel carbon by dual carbon-isotopic finger printing. This method usefully distinguishes chemically-identical materials, and apportions carbon in the copolymer by source (and possibly year) of growth of the biospheric (plant) component. The isotopes,  $^{14}\text{C}$  and  $^{13}\text{C}$ , bring complementary information to this problem. The radiocarbon dating isotope ( $^{14}\text{C}$ ), with its nuclear half life of 5730 years, clearly allows one to apportion specimen carbon between fossil ("dead") and biospheric ("alive") feedstocks (Currie, L. A. "Source Apportionment of Atmospheric Particles," Characterization of Environmental Particles, J. Buffle and H.P. van Leeuwen, Eds., 1 of 10 Vol. I of the IUPAC Environmental Analytical Chemistry Series (Lewis Publishers, Inc) (1992) 3-74). The basic assumption in radiocarbon dating is that the constancy of  $^{14}\text{C}$  concentration in the atmosphere leads to the constancy of  $^{14}\text{C}$  in living organisms. When dealing with an isolated sample, the age of a sample can be deduced approximately by the relationship:

15 
$$t = (-5730/0.693)\ln(A/A_0)$$

wherein  $t$  = age, 5730 years is the half-life of radiocarbon, and  $A$  and  $A_0$  are the specific  $^{14}\text{C}$  activity of the sample and of the modern standard, respectively (Hsieh, Y., Soil Sci. Soc. Am J., 56, 460, (1992)). However, because of atmospheric nuclear testing since 1950 and the burning of fossil fuel since 20 1850,  $^{14}\text{C}$  has acquired a second, geochemical time characteristic. Its concentration in atmospheric  $\text{CO}_2$ , and hence in the living biosphere, approximately doubled at the peak of nuclear testing, in the mid-1960s. It has since 25 been gradually returning to the steady-state cosmogenic (atmospheric) baseline isotope rate ( $^{14}\text{C}/^{12}\text{C}$ ) of ca.  $1.2 \times 10^{-12}$ , with an approximate relaxation "half-life" of 7-10 years. (This latter half-life must not be taken literally; rather, 30 one must use the detailed atmospheric nuclear input/decay function to trace the variation of atmospheric and biospheric  $^{14}\text{C}$  since the onset of the nuclear age.) It is this latter biospheric  $^{14}\text{C}$  time characteristic that holds out the promise of annual dating of recent biospheric carbon.  $^{14}\text{C}$  can be measured by 35 accelerator mass spectrometry (AMS), with results given in units of "fraction of modern carbon" ( $f_M$ ).  $f_M$  is defined by National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) 4990B and 4990C, known as oxalic acids standards HOxI and HOxII, respectively. The fundamental definition relates to 0.95 times the  $^{14}\text{C}/^{12}\text{C}$  isotope ratio HOxI (referenced to AD 1950). This is roughly equivalent to decay-corrected pre-

Industrial Revolution wood. For the current living biosphere (plant material),  $f_M \approx 1.1$ .

The stable carbon isotope ratio ( $^{13}\text{C}/^{12}\text{C}$ ) provides a complementary route to source discrimination and apportionment. The  $^{13}\text{C}/^{12}\text{C}$  ratio in a given biosourced material is a consequence of the  $^{13}\text{C}/^{12}\text{C}$  ratio in atmospheric carbon dioxide at the time the carbon dioxide is fixed and also reflects the precise metabolic pathway. Regional variations also occur. Petroleum,  $\text{C}_3$  plants (the broadleaf),  $\text{C}_4$  plants (the grasses), and marine carbonates all show significant differences in  $^{13}\text{C}/^{12}\text{C}$  and the corresponding  $\delta^{13}\text{C}$  values. Furthermore, lipid matter of  $\text{C}_3$  and  $\text{C}_4$  plants analyze differently than materials derived from the carbohydrate components of the same plants as a consequence of the metabolic pathway. Within the precision of measurement,  $^{13}\text{C}$  shows large variations due to isotopic fractionation effects, the most significant of which for the instant invention is the photosynthetic mechanism. The major cause of differences in the carbon isotope ratio in plants is closely associated with differences in the pathway of photosynthetic carbon metabolism in the plants, particularly the reaction occurring during the primary carboxylation, i.e., the initial fixation of atmospheric  $\text{CO}_2$ . Two large classes of vegetation are those that incorporate the " $\text{C}_3$ " (or Calvin-Benson) photosynthetic cycle and those that incorporate the " $\text{C}_4$ " (or Hatch-Slack) photosynthetic cycle.  $\text{C}_3$  plants, such as hardwoods and conifers, are dominant in the temperate climate zones. In  $\text{C}_3$  plants, the primary  $\text{CO}_2$  fixation or carboxylation reaction involves the enzyme ribulose-1,5-diphosphate carboxylase and the first stable product is a 3-carbon compound.  $\text{C}_4$  plants, on the other hand, include such plants as tropical grasses, corn and sugar cane. In  $\text{C}_4$  plants, an additional carboxylation reaction involving another enzyme, phosphoenol-pyruvate carboxylase, is the primary carboxylation reaction. The first stable carbon compound is a 4-carbon acid, which is subsequently decarboxylated. The  $\text{CO}_2$  thus released is refixed by the  $\text{C}_3$  cycle.

Both  $\text{C}_4$  and  $\text{C}_3$  plants exhibit a range of  $^{13}\text{C}/^{12}\text{C}$  isotopic ratios, but typical values are ca. -10 to -14 per mil ( $\text{C}_4$ ) and -21 to -26 per mil ( $\text{C}_3$ ) (Weber et al., *J. Agric. Food Chem.*, 45, 2942 (1997)). Coal and petroleum fall generally in this latter range. The  $^{13}\text{C}$  measurement scale was originally defined by a zero set by pee dee belemnite (PDB) limestone, where values are given in parts per thousand deviations from this material. The " $\delta^{13}\text{C}$ " values

are in parts per thousand (per mil), abbreviated ‰, and are calculated as follows:

$$\delta^{13}\text{C} \equiv \frac{(^{13}\text{C}/^{12}\text{C})_{\text{sample}} - (^{13}\text{C}/^{12}\text{C})_{\text{standard}}}{(^{13}\text{C}/^{12}\text{C})_{\text{standard}}} \times 1000\text{‰}$$

5 Since the PDB reference material (RM) has been exhausted, a series of alternative RMs have been developed in cooperation with the IAEA, USGS, NIST, and other selected international isotope laboratories. Notations for the per mil deviations from PDB is  $\delta^{13}\text{C}$ . Measurements are made on  $\text{CO}_2$  by high precision stable ratio mass spectrometry (IRMS) on molecular ions of  
10 masses 44, 45 and 46.

Biologically-derived 1,3-propanediol, and compositions comprising biologically-derived 1,3-propanediol, therefore, may be completely distinguished from their petrochemical derived counterparts on the basis of  $^{14}\text{C}$  ( $f_M$ ) and dual carbon-isotopic fingerprinting, indicating new compositions of matter.

15 The ability to distinguish these products is beneficial in tracking these materials in commerce. For example, products comprising both "new" and "old" carbon isotope profiles may be distinguished from products made only of "old" materials. Hence, the instant materials may be followed in commerce on the basis of their unique profile and for the purposes of defining competition, for  
20 determining shelf life, and especially for assessing environmental impact.

Preferably the 1,3-propanediol used as the reactant or as a component of the reactant will have a purity of greater than about 99%, and more preferably greater than about 99.9%, by weight as determined by gas chromatographic analysis. Particularly preferred are the purified 1,3-propanediols  
25 as disclosed in previously incorporated US7038092, US20040260125A1, US20040225161A1 and US20050069997A1, as well as PO3G made therefrom as disclosed in US20050020805A1.

The purified 1,3-propanediol preferably has the following characteristics:

30 (1) an ultraviolet absorption at 220 nm of less than about 0.200, and at 250 nm of less than about 0.075, and at 275 nm of less than about 0.075; and/or

(2) a composition having L\*a\*b\* "b\*" color value of less than about 0.15 (ASTM D6290), and an absorbance at 270 nm of less than about 0.075; and/or

(3) a peroxide composition of less than about 10 ppm; and/or

5 (4) a concentration of total organic impurities (organic compounds other than 1,3-propanediol) of less than about 400 ppm, more preferably less than about 300 ppm, and still more preferably less than about 150 ppm, as measured by gas chromatography.

The starting material for making PO3G will depend on the desired 10 PO3G, availability of starting materials, catalysts, equipment, etc., and comprises "1,3-propanediol reactant." By "1,3-propanediol reactant" is meant 1,3-propanediol, and oligomers and prepolymers of 1,3-propanediol preferably having a degree of polymerization of 2 to 9, and mixtures thereof. In some instances, it may be desirable to use up to 10% or more of low molecular 15 weight oligomers where they are available. Thus, preferably the starting material comprises 1,3-propanediol and the dimer and trimer thereof. A particularly preferred starting material is comprised of about 90% by weight or more 1,3-propanediol, and more preferably about 99% by weight or more 1,3-propanediol, based on the weight of the 1,3-propanediol reactant.

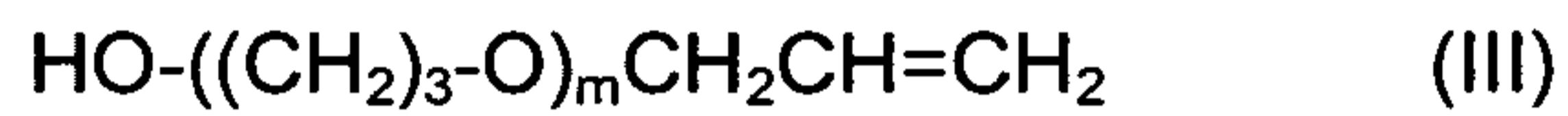
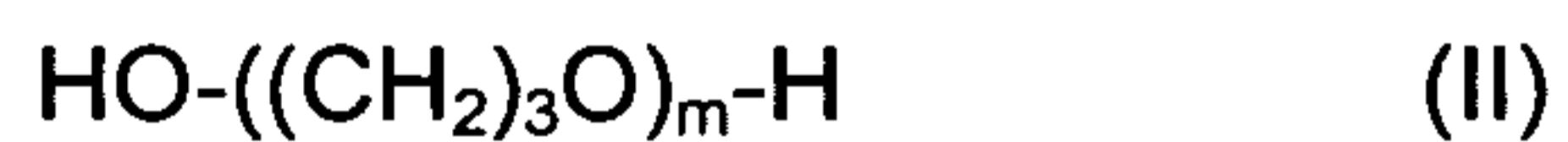
20 PO3G can be made via a number of processes known in the art, such as disclosed in US6977291 and US6720459. A preferred process is as set forth in previously incorporated US20050020805A1.

As indicated above, PO3G may contain lesser amounts of other 25 polyalkylene ether repeating units in addition to the trimethylene ether units. The monomers for use in preparing polytrimethylene ether glycol can, therefore, contain up to 50% by weight (preferably about 20 wt% or less, more preferably about 10 wt% or less, and still more preferably about 2 wt% or less), of comonomer polyols in addition to the 1,3-propanediol reactant. Suitable comonomer polyols include aliphatic diols, for example, ethylene glycol, 30 1,6-hexanediol, 1,7-heptanediol, 1,8-octanediol, 1,9-nanediol, 1,10-decanediol, 1,12-dodecanediol, 3,3,4,4,5,5-hexafluoro-1,5-pentanediol, 2,2,3,3,4,4,5,5-octafluoro-1,6-hexanediol, and 3,3,4,4,5,5,6,6,7,7,8,8,9,9,10,10-hexadecafluoro-1,12-dodecanediol; cycloaliphatic diols, for example, 1,4-cyclohexanediol,

1,4-cyclohexanedimethanol and isosorbide; and polyhydroxy compounds, for example, glycerol, trimethylolpropane and pentaerythritol. A preferred group of comonomer diols is selected from the group consisting of ethylene glycol, 2-methyl-1,3-propanediol, 2,2-dimethyl-1,3-propanediol, 2,2-diethyl-1,3-  
5 propanediol, 2-ethyl-2-(hydroxymethyl)-1,3-propanediol, C<sub>6</sub> – C<sub>10</sub> diols (such as 1,6-hexanediol, 1,8-octanediol and 1,10-decanediol) and isosorbide, and mixtures thereof. Particularly preferred diols other than 1,3-propanediol include ethylene glycol, 2-methyl-1,3-propanediol and C<sub>6</sub> – C<sub>10</sub> diols.

One preferred PO3G containing comonomers is poly(trimethylene-  
10 ethylene ether) glycol such as described in US20040030095A1. Preferred poly(trimethylene-ethylene ether) glycols are prepared by acid catalyzed polycondensation of from greater than 50 to about 99 mole% (preferably from about 60 to about 98 mole%, and more preferably from about 70 to about 98 mole%) 1,3-propanediol, and up to 50 to about 1 mole% (preferably from about 40 to about 2 mole%, and more preferably from about 30 to about 2 mole%) ethylene glycol.  
15

Preferably, the PO3G after purification has essentially no acid catalyst end groups, but may contain very low levels of unsaturated end groups, predominately allyl end groups, in the range of from about 0.003 to about 0.03  
20 meq/g. Such a PO3G can be considered to comprise (consist essentially of) the compounds having the following formulae (II) and (III):



wherein m is in a range such that the Mn (number average molecular weight)  
25 is within the range of from about 200 to about 5000, with compounds of formula (III) being present in an amount such that the allyl end groups (preferably all unsaturation ends or end groups) are present in the range of from about 0.003 to about 0.03 meq/g. The small number of allyl end groups in the  
30 PO3G are useful to control elastomer molecular weight, while not unduly restricting it, so that compositions ideally suited, for example, for fiber end-uses can be prepared.

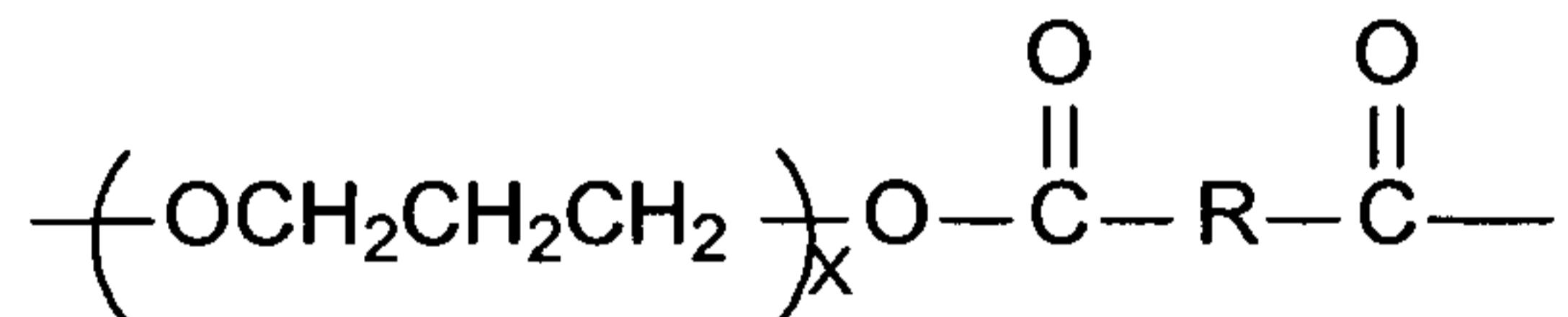
The preferred PO3G for use in the invention has an Mn of at least about 750, more preferably at least about 1000, and still more preferably at

least about 2000. The Mn is preferably less than about 5000, more preferably less than about 4000, and still more preferably less than about 3500. Blends of PO3Gs can also be used. For example, the PO3G can comprise a blend of a higher and a lower molecular weight PO3G, preferably wherein the 5 higher molecular weight PO3G has a number average molecular weight of from about 1000 to about 5000, and the lower molecular weight PO3G has a number average molecular weight of from about 200 to about 950. The Mn of the blended PO3G will preferably still be in the ranges mentioned above.

PO3G preferred for use herein is typically a polydisperse polymer having a polydispersity (i.e. Mw/Mn) of preferably from about 1.0 to about 2.2, more preferably from about 1.2 to about 2.2, and still more preferably from about 1.5 to about 2.1. The polydispersity can be adjusted by using blends of 10 PO3G.

PO3G for use in the present invention preferably has a color value of 15 less than about 100 APHA, and more preferably less than about 50 APHA.

When a PO3G based substantially on 1,3-propanediol is used to form the soft segment, the soft segment can be represented as comprising units represented by the following structure:



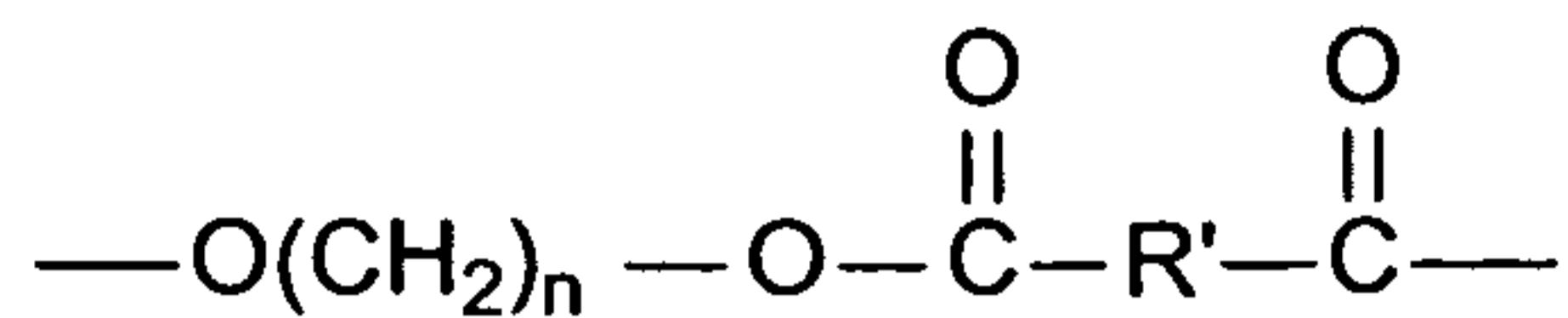
20 wherein R represents a divalent radical remaining after removal of carboxyl functionalities from a dicarboxylic acid equivalent, and x is a whole number representing the number of trimethylene ether units in the PO3G.

The polymeric ether glycol used to prepare the polytrimethylene ether ester soft segment of the polyether ester may also include up to 50 wt% of a 25 polymeric ether glycol other than PO3G. Preferred such other polymeric ether glycols include, for example, polyethylene ether glycol, polypropylene ether glycol, polytetramethylene ether glycol, polyhexamethylene ether glycol, copolymers of tetrahydrofuran and 3-alkyl tetrahydrofuran, and mixtures thereof.

Diol for Hard Segment

When trimethylene/tetramethylene glycol is used to form the hard segment, the hard segment can be represented as comprising units having the following structure:

5



wherein n is 3 (trimethylene) or 4 (tetramethylene), and R' represents a divalent radical remaining after removal of carboxyl functionalities from a dicarboxylic acid equivalent. In most cases, the dicarboxylic acid equivalents used to prepare the soft segment and the hard segment of the polyether ester elastomers used in this invention will be the same.

10

The hard segment can also be prepared with less than 50 mole % (preferably up to about 25 mole %, more preferably up to about 15 mole %), of diols other than trimethylene glycol or tetramethylene glycol, preferably having a molecular weight lower than about 400. The other diols are preferably aliphatic diols and can be acyclic or cyclic. Preferred are diols with up to about 15 carbon atoms such as ethylene, isobutylene, butylene, pentamethylene, 2,2-dimethyltrimethylene, 2-methyltrimethylene, hexamethylene and decamethylene glycols, dihydroxy cyclohexane, cyclohexane dimethanol, hydroquinone bis(2-hydroxyethyl) ether. Especially preferred are aliphatic diols containing 2-8 carbon atoms. Most preferred is ethylene glycol. Two or more other diols can be used.

15

20

Dicarboxylic Acid Equivalent

25

30

The dicarboxylic acid equivalent can be aromatic, aliphatic or cycloaliphatic. In this regard, "aromatic dicarboxylic acid equivalents" are dicarboxylic acid equivalents in which each carboxyl group is attached to a carbon atom in a benzene ring system such as those mentioned below. "Aliphatic dicarboxylic acid equivalents" are dicarboxylic acid equivalents in which each carboxyl group is attached to a fully saturated carbon atom or to a carbon atom which is part of an olefinic double bond. If the carbon atom is in a ring, the equivalent is "cycloaliphatic." The dicarboxylic acid equivalent can contain any substituent groups or combinations thereof, so long as the sub-

stituent groups do not interfere with the polymerization reaction or adversely affect the properties of the polyether ester product.

Preferred are the dicarboxylic acid equivalents selected from the group consisting of dicarboxylic acids and diesters of dicarboxylic acids.

5 More preferred are dimethyl esters of dicarboxylic acids.

Preferred are the aromatic dicarboxylic acids or diesters by themselves, or with small amounts of aliphatic or cycloaliphatic dicarboxylic acids or diesters. Especially preferred are the dimethyl esters of aromatic dicarboxylic acids.

10 Representative aromatic dicarboxylic acids useful in the present invention include terephthalic acid, isophthalic acid, bibenzoic acid, naphthalic acid, substituted dicarboxylic compounds with benzene nuclei such as bis(p-carboxyphenyl)methane, 1,5-naphthalene dicarboxylic acid, 2,6-naphthalene dicarboxylic acid, 2,7-naphthalene dicarboxylic acid, 4,4'-sulfonyl dibenzoic acid, and C1-C10 alkyl and other ring substitution derivatives such as halo, alkoxy or aryl derivatives. Hydroxy acids such as p-(hydroxyethoxy)benzoic acid can also be used providing an aromatic dicarboxylic acid is also present. Representative aliphatic and cycloaliphatic dicarboxylic acids useful in this invention are sebacic acid, 1,3- or 1,4-cyclohexane dicarboxylic acid, adipic acid, dodecanedioic acid, glutaric acid, succinic acid, oxalic acid, azelaic acid, diethylmalonic acid, fumaric acid, citraconic acid, allylmalonate acid, 4-cyclohexene-1,2-dicarboxylate acid, pimelic acid, suberic acid, 2,5-diethyladipic acid, 2-ethylsuberic acid, 2,2,3,3-tetramethyl succinic acid, cyclopentanenedicarboxylic acid, decahydro-1,5- (or 2,6-)naphthalene dicarboxylic acid, 4,4'-bicyclohexyl dicarboxylic acid, 4,4'methylenebis(cyclohexylcarboxylic acid), 3,4-furan dicarboxylate, and 1,1-cyclobutane dicarboxylate. The dicarboxylic acid equivalents in the form of diesters, acid halides and anhydrides of the aforementioned aliphatic dicarboxylic acids are also useful to provide the polyether ester of the present invention. Representative aromatic diesters include dimethyl terephthalate, bibenzoate, isophthalate, phthalate and naphthalate.

30 Of the above, preferred are terephthalic, bibenzoic, isophthalic and naphthalic acid; dimethyl terephthalate, bibenzoate, isophthalate, naphthalate and phthalate; and mixtures thereof. Particularly preferred dicarboxylic acid equivalents are the equivalents of phenylene dicarboxylic acids especially

35

those selected from the group consisting of terephthalic and isophthalic acid and their diesters, especially the dimethyl esters, dimethyl terephthalate and dimethyl isophthalate. In addition, two or more dicarboxylic acids equivalents can be used. For instance, terephthalic acid and/or dimethyl terephthalate 5 can be used with small amounts of the other dicarboxylic acid equivalents.

In a preferred embodiment, at least about 70 mole% (more preferably at least about 80 mole%, still more preferably at least about 90 mole%, and still more preferably from about 95 to 100 mole%) of the dicarboxylic acid equivalent is terephthalic acid and/or dimethyl terephthalate.

10

### Preparation of Polyether Ester Elastomers

The polyether esters for use in the spinning processes of the invention are preferably prepared by providing and reacting (a) polytrimethylene ether glycol, (b) trimethylene glycol or tetramethylene glycol or mixtures thereof, and (c) dicarboxylic acid, ester, acid chloride or acid anhydride. The other 15 glycols, diols, etc., as described above are can also be provided and reacted. Procedures for preparation of polyether ester elastomers comprising polytrimethylene ether ester soft segment and trimethylene ester hard segment are disclosed in detail in US6599625. Procedures for preparation of polyether ester elastomers comprising polytrimethylene ether ester soft segment and 20 tetramethylene ester hard segment are disclosed in detail in US6562457.

### Additives

The polyether ester thermoplastic elastomer may be in the form of a composition comprising, in addition to the elastomer, one or more additives including, but not limited to, those selected from the group consisting of delusterants, nucleating agents, heat stabilizers, viscosity boosters, optical brighteners, pigments and antioxidants. Titanium dioxide or other pigments can, for 25 example, be added to the polymers or in fiber manufacture.

The polyether ester elastomers can also be made acid-dyeable using the additives described in US2003-0083441A1 and WO01/034693. The additives of WO01/034693 comprise a secondary amine or secondary amine salt 30 in an amount effective to promote acid-dyeability of the acid dyeable and acid dyed compositions. Preferably, the secondary amine unit is present in the polymer composition in an amount of at least about 0.5 mole %, more pref-

erably at least about 1 mole %. The secondary amine unit is present in the polymer composition in an amount preferably of about 15 mole % or less, more preferably about 10 mole % or less, and still more preferably about 5 mole % or less, based on the weight of the composition. The additives of 5 US2003-0083441A1 are polymeric additives based on a tertiary amine. The polymeric additive is prepared from (i) triamine containing secondary amine or secondary amine salt unit(s) and (ii) one or more other monomer and/or polymer units. One preferred polymeric additive comprises polyamide selected from the group consisting of poly-imino-bisalkylene-terephthalamide, - 10 isophthalamide and -1,6 naphthalamide, and salts thereof.

The polyether esters useful in this invention can also be made cationically dyeable using dyeability modifiers such as those described in US6312805.

#### Filament and Yarn Preparation

15 For spinning, the polymer is heated to a temperature above its melting point and then extruded through a spinneret, preferably a multi-hole spinneret, at a temperature of about 175 to about 295°C, preferably at least about 200°C and up to about 275°C, most preferably up to about 270°C. Higher temperatures are useful for low residence times.

20 Fibers may be drawn or undrawn. When they are drawn, the draw ratio is at least 1.01, preferably up to about 5, more preferably up to about 4 and most preferably up to about 3.

In a preferred embodiment of the invention, the process is advantageously utilized to prepare spun drawn yarn, also known as "fully drawn yarn".  
25 The preferred steps of manufacturing spun drawn yarns including spinning, drawing, optionally annealing, optionally interlacing, and winding the filaments, are similar to those used for preparing poly(ethylene terephthalate) yarns. Preferably the spun drawn yarns produced by the process of the invention are multifilament yarns.

30 An advantage of this invention is that spun drawn yarns can be prepared using higher draw ratios than disclosed in US6562457 and US6599625 for the same polyether esters. This can be accomplished by using a lower spin speed than normal, and then drawing at previously used speeds. When

carrying out this process, there are fewer breaks than previously encountered.

Draw speeds (as measured at the roller at the end of the draw step) are higher than about 1200 m/m, and are preferably at least about 3000 m/m, 5 more preferably at least about 3200 m/m, and preferably up to about 8000 m/m, more preferably up to about 7000 m/m. Another advantage of this invention is that spun drawn yarns can be spun on equipment previously used to spin spun drawn yarns of poly(ethylene terephthalate).

Spun drawn yarns are usually wound on a package, and can be used 10 to make fabrics or further processed into other types of yarn, such as textured yarn.

Although the processes of the present invention are exemplified below for the preparation of spun drawn yarns, the invention is also useful for preparing partially oriented yarns.

15 The basic steps of manufacturing partially oriented yarns including spinning, interlacing and winding poly(trimethylene terephthalate) filaments are described in US6287688, US6333106 and US2001-030378A1. This invention can be practiced using those steps or other steps conventionally used for making partially oriented polyester yarns.

20 The partially oriented yarns are multifilament yarns. The yarns (also known as "bundles") preferably comprise at least about 2, and more preferably at least about 25 filaments. The yarns typically have a total denier of from about 1 to about 500, preferably at least about 20, more preferably at least about 50, and even more preferably from about 50 to about 300.

25 Filaments are preferably at least about 0.5 denier per filament (dpf), more preferably at least about 1 dpf, and up to about 20 or more dpf, more preferably up to about 7 dpf. Typical filaments are about 3 to 7 dpf, and fine filaments are about 0.5 to about 2.5 dpf.

30 Spin speeds are greater than about 1200 and can be up to about 5000 or more meters/minute ("m/m"). They are preferably at least about 1500 m/m, and more preferably at least about 3000 m/m. Spin speed is generally defined as maximum process speed and, in the case of the present invention, is considered draw roll speed. Thus, an advantage of this invention is that the

process can be carried out at higher speeds than those disclosed in US6562457 and US6599625 for the same polyether esters.

An advantage of this invention is that partially oriented yarns of poly-ether ester can be spun on equipment previously used to spin partially oriented yarns of poly(ethylene terephthalate), so spin speeds are preferably up to about 4000 m/m, more preferably up to about 3500 m/m.

Partially oriented yarns are usually wound on a package, and can be used to make fabrics or further processed into other types of yarn, such as textured yarn. They can also be stored in a can prior to preparing fabrics or further processing, or can be used directly without forming a package or other storage.

Textured yarns can be prepared from partially oriented yarns or spun drawn yarns. The main difference is that the partially oriented yarns usually require drawing whereas the spun drawn yarns are already drawn.

US6287688, US6333106 and US2001-030378A1 all describe the basic steps of manufacturing textured yarns from partially oriented yarns. This invention can be practiced using those steps or other steps conventionally used for making partially oriented polyester yarns. The basic steps include unwinding the yarns from a package, drawing, twisting, heat-setting, untwisting, and winding onto a package. Texturing imparts crimp by twisting, heat setting, and untwisting by the process commonly known as false twist texturing. The false-twist texturing is carefully controlled to avoid excessive yarn and filament breakage.

A preferred process for friction false-twisting, as described in US6287688, US6333106 and US2001-030378A1, comprises heating the partially oriented yarn to a temperature between 140°C and 220°C, twisting the yarn using a twist insertion device such that in the region between the twist insertion device and the entrance of the heater, the yarn has a twist angle of about 46° to 52°, and winding the yarn on a winder.

When prepared from spun drawn yarn, the process is the same except that drawing is reduced to a very low level (e.g., draw ratio can be as low as 1.01).

These multifilament yarns (also known as "bundles") comprise the same number of filaments as the partially oriented yarns and spun drawn yarns from which they are made. Thus, they preferably comprise at least about 2 and even more preferably at least about 25 filaments. The yarns 5 typically have a total denier of from about 1 to about 500, preferably at least about 20, preferably at least about 50, and more preferably from about 50 to about 300. Filaments are preferably at least about 0.1 dpf, more preferably at least about 0.5 dpf, more preferably at least about 0.8 dpf, and up to about 10 or more dpf, more preferably up to about 5 dpf, and most preferably up to 10 about 3 dpf.

Staple fibers and products can be prepared from the polyether esters of the invention. One preferred process comprises: (a) providing poly-trimethylene ether ester comprising from about 80 to about 40 wt% poly-trimethylene ether dicarboxylate ester soft segment and from about 20 to 15 about 60 wt% hard segment selected from the group consisting trimethylene dicarboxylate ester and tetramethylene dicarboxylate ester (b) melt spinning the polytrimethylene ether ester at a temperature of about 245 to about 285°C into filaments, (c) quenching the filaments, (d) drawing the quenched filaments, (e) crimping the drawn filaments using a mechanical crimper at a 20 crimp level of about 8 to about 30 crimps per inch (about 3 to about 12 crimps/cm), (f) relaxing the crimped filaments at a temperature of about 50 to about 120°C, and (g) cutting the relaxed filaments into staple fibers, preferably having a length of about 0.2 to about 6 inches (about 0.5 to about 15 cm). In one preferred embodiment of this process, the drawn filaments are annealed at about 85 to about 115°C before crimping. Preferably, annealing is 25 carried out under tension using heated rollers. In another preferred embodiment, the drawn filaments are not annealed before crimping.

Staple fibers are useful in preparing textile yarns and textile or non-woven fabrics, and can also be used for fiberfill applications and making carpets. 30

The spinning processes of this invention can also be used to improve the spin feed in preparation of bulked continuous filament ("BCF") yarns. BCF yarns and their manufacture are described, for example, in US5645782, US6109015, US6113825, US6576340, US6723799, US6740276 and 35 US2003-0175522 A1. BCF yarns are used to prepare all types of carpets, as

well as textiles. The compositions of this invention can be used to improve the spin speed of their preparation.

Preferred steps involved in preparing bulked continuous filaments include spinning (e.g., extruding, cooling and coating (spin finish) the filaments), 5 single stage or multistage drawing (preferably with heated rolls, heated pin or hot fluid assist (e.g., steam or air)) at about 80 to about 200°C and at a draw ratio of about 1.1 to about 5, preferably at least about 1.5 and preferably up to about 4.5, annealing at a temperature of about 120 to about 200°C, bulking, entangling (which can be carried out in one step with bulking or in a subsequent 10 separate step) optionally relaxing, and winding the filaments on a package for subsequent use.

Bulked continuous filament yarns can be made into carpets using well known techniques. Typically, a number of yarns are cable twisted together and heat set in a device such as an autoclave, and then tufted into a primary 15 backing. Latex adhesive and a secondary backing are then applied.

The individual filaments can be round or have other shapes, such as octalobal, delta, sunburst (also known as sol), scalloped oval, trilobal, tetra-channel (also known as quatra-channel), scalloped ribbon, ribbon, starburst, etc. They can be solid, hollow or multi-hollow.

20 While it is possible to prepare filaments in a yarn bundle with differing cross section geometries using a single spinneret, the invention is preferably practiced by spinning one type of filament cross section from a spinneret.

### EXAMPLES

25 The following examples are presented for the purpose of illustrating the invention, and are not intended to be limiting. All parts, percentages, etc., are by weight unless otherwise indicated.

#### Inherent Viscosity

Inherent Viscosity (IV) measurements were made following ASTM Method 2857-70. The polymer samples were dried at 70°C for 3 hours before 30 weighing. Samples were run at 30°C using 0.5% solutions in m-cresol. To improve efficiency, accuracy, and precision an AUTOVISC® Automatic Measuring System (Cannon Instrument Company, State College, Pa., U.S.A.)

automated viscosity measuring system was used. A high density infrared fiber optic detection system was used in place of a human operator, and an air bath was used in place of the oil or water bath normally used to provide constant temperature. The AUTOVISC® exceeds the accuracy specifications of 5 ASTM D-445, "Standard Test Method For Kinematic Viscosity of Transparent and Opaque Liquids".

#### Number Average Molecular Weight

The number average molecular weights were calculated according to 10 ASTM D 5296-97 using a poly(ethylene terephthalate) of  $M_w \sim 44,000$  calibration standard and hexafluoroisopropanol solvent.

#### % Hard Segment

Percent hard segment was determined as described in US6562457.

#### Fiber Tenacity and Elongation

Tenacity at break,  $T$ , in grams per denier (gpd) and percent elongation 15 at break,  $E$ , were measured on an INSTRON.RTM. Tester equipped with a Series 2712 (002) Pneumatic Action Grips equipped with acrylic contact faces. The test was repeated three times. The average of the results is reported.

The average denier for the fibers used in the tenacity and elongation 20 measurements is reported as "Denier 1".

#### Fiber Unload Power, Stress Decay and Percent Set

The average denier for the fibers used in measuring unload power, stress decay and percent set is reported as "Denier 2".

Unload power was measured in  $dN/texeffx1000$ . One filament, a 2-25 inch (5 cm) gauge length, was used for each determination. Separate measurements were made using zero-to-300% elongation cycles. Unload power (i.e., the stress at a particular elongation) was measured after the samples had been cycled five times at a constant elongation rate of 100% per minute and then held at 300% extension for half a minute after the fifth extension. 30 While unloading from this last extension, the stress, or unload power, was measured at various elongations. Unload powers are reported herein as the

effective unload power using the general form "UPx/y" where x is the percent elongation to which the fiber was cycled five times and y is the percent elongation at which the stress, or unload power, was measured.

5 Stress Decay was measured as the percent loss of stress on a fiber over a 30 second period with the sample held at 300% extension at the end of the fifth load cycle.

$$S = ((F-C) * 100) / F$$

Wherein  $S$ =Stress Decay, %

$F$ =Stress at full extension

10  $C$ =Stress after 30 seconds

The percent set was measured from the stress/strain curve recorded on chart paper.

#### Example 1

15 This example describes preparation of a polyether ester elastomer having a polytrimethylene ether ester soft segment and a tetramethylene ester hard segments, as described in US6562457. The polymer was designed to have a 72/28 weight ratio of soft segments to hard segments. The polymer was prepared using a batch process from dimethyl terephthalate, 1,4-butanediol and polytrimethylene ether glycol.

20 An autoclave reactor equipped with an agitator, vacuum jets and a distillation still was charged with 8.81 kg of dimethyl terephthalate, 8.94 kg of 1,4-butanediol, 20.2 kg of polytrimethylene ether glycol of number average molecular weight 2019, 22 g of tetraisopropyl titanate polymerization catalyst, and 37.5 g of ETHANOX 330 antioxidant (Albemarle Corporation). The temperature of the reactor was gradually raised to 210°C, and approximately 2.6 kg of methanol were recovered by distillation from the reactor. The reaction was continued further at 250°C under reduced pressure for an additional 1.5 hours to increase molecular weight. The resulting polymer was extruded from the reactor and converted into pellets, which were dried at 80-90°C under reduced pressure overnight before further use. The properties of the polymer were as follows:

	Inherent Viscosity:	1.403 dL/g
	Mn:	31400
	Mw:	61200
	Tg of Soft Segment:	-67°C
5	Tm of Hard Segment:	170°C
	% Hard Segment:	28%

The following fiber spinning examples were carried out with the polymer of Example 1.

Spinning Procedure

10        Polymer of Example 1 was extruded through a sand filter spin pack and a multi hole spinneret (0.3 mm diameter and 0.56 mm capillary depth holes, number of holes as indicated below in the Table 1) maintained at 273°C. The filamentary streams leaving the spinneret were quenched with air at 21°C, and converged to a bundle. Forwarding (feed) rolls with a surface speed described in the table below delivered the yarn bundle in sequence to a set of draw rolls, to an interlace jet, to a set of letdown rolls and to a windup at speeds described in the Table 1 below. The spinning conditions for the yarns are described in Table 1. The properties of the resultant yarns are described in Table 2.

15       

20        Comparative Examples 1 and 2 present properties of two different commercially available spandex yarns for comparison with the data of Examples 2-9.

Table 1

		<u>Spinning Conditions</u>					
EX	No. of Filaments	Grams/min	Feed Roll Speed (mpm)	Draw Roll Speed (mpm)	Draw Ratio	Letdown Roll Speed (mpm)	Windup Speed (mpm)
2	3	7.95	760	2660	3.5	2515	2500
3	3	7.95	760	2660	3.5	2515	2580
4	3	7.95	760	2660	3.5	2515	2590
5	3	7.95	760	2660	3.5	2515	2590
6	3	7.95	2000	2660	1.33	2515	2610
7	6	10.5	760	2660	3.5	2515	2590
8	6	10.5	760	2660	3.5	2515	2590
9	6	10.5	760	2660	3.5	2515	2590

Table 2  
Yarn Properties

EX	Denier 1	Tenacity (g/den)	% Elong.	Denier 2	% Stretch for 5 Cycle	Tenacity (g/den)	% Elong.	% Decay	% Set	Unloading (g/den)
2	53.5	1.00	303	49.1	300	0.83	320	18.8	52.2	18.9
3	51.6	1.11	256	51.8	300	1.04	255	16.0	21.1	21.4
4	53.0	1.07	255	52.0	200	1.05	260	16.0	21.4	21.4
5	48.1	1.13	256	50.0	200	0.96	235	16.0	20.8	20.7
6	41.1	1.07	275	43.0	200	1.03	291	14.1	20.9	14.9
7	72.5	1.08	260	78.0	200	0.96	264	15.5	18.5	29.4
8	68.3	1.19	253	70.9	200	1.14	272	16.1	19.2	30.4
9	80.3	1.02	245	80.0	200	1.13	278	16.7	19.7	33.9
C1	71.2	0.92	490	70.8	300	1.02	514	27.6	16.9	9.7
C2	41.9	1.30	487	41.3	300	1.32	495	29.0	13.4	7.3

The data in Table 1 show clearly that the polyether ester elastomers used could be spun in accordance with the invention into yarns at commercially feasible speeds that are considerably faster than those reported in US6562457 and US6599625. Furthermore, the comparative data demonstrate the yarns obtained in the process of the invention had generalized elastomer properties.

CLAIMS

What is claimed is:

1. A process for preparing an elastoester multifilament yarn comprising the steps of:

5 (a) providing a polyether ester thermoplastic elastomer comprising from about 80 to about 40 wt% of a polytrimethylene ether dicarboxylate ester soft segment, and from about 20 to about 60 wt% of a hard segment selected from the group consisting of a trimethylene dicarboxylate ester, a tetramethylene dicarboxylate ester and mixtures thereof, based on the combined weight  
10 of the hard and soft segments;

(b) melt spinning the elastomer by extruding it through one or more spinnerets to form at least 2 filaments; and

(c) processing the filaments into a multifilament yarn,

wherein the melt spinning is conducted at a spinning speed of greater than

15 about 1200 meters/minute.

2. The process of claim 1, wherein the dicarboxylate comprises terephthalate.

3. The process of claim 1, wherein the polytrimethylene ether ester comprises about 80 to about 40 wt% polytrimethylene ether terephthalate soft segment and about 20 to about 60 wt% trimethylene terephthalate hard segment.  
20

4. The process of claim 1, wherein the polytrimethylene ether ester comprises about 80 to about 40 wt% polytrimethylene ether terephthalate soft segment and about 20 to about 60 wt% tetramethylene terephthalate hard segment.  
25

5. The process of claim 1, wherein the multifilament yarn comprises filaments having a denier per filament of about 0.5 to about 20.

6. The process of claim 5, wherein the process further comprises interlacing and winding the filaments.

7. The process of claim 1, wherein the multifilament yarn is spun drawn yarn and the processing comprises drawing the filaments at a draw speed, as measured at the roller at the end of the draw step, of about 1250 to about 5000 meters/minute.
- 5 8. The process of claim 7, wherein the processing into spun drawn multi-filament comprises drawing, annealing, interlacing and winding the filaments.
9. The process of claim 1, wherein the multifilament yarn is partially oriented yarn.
10. The process of claim 1, wherein the process further comprises cutting the multifilament yarn into staple fibers.
11. The process of claim 1, wherein the polyether ester thermoplastic elastomer comprises at least one additive selected from the group consisting of delusterants, nucleating agents, heat stabilizers, viscosity boosters, optical brighteners, pigments and antioxidants.
- 15 12. The process of claim 1, wherein the multifilament yarn is an elastomer with an elongation of from about 100 to about 600%.
13. The process of claim 1, wherein the multifilament yarn is an elastomer with a tenacity of from about 0.5 to about 2.5 grams/denier.
14. A process for preparing multifilament textured yarn comprising filaments of polytrimethylene ether ester comprising from about 80 to about 40 wt% polytrimethylene ether dicarboxylate ester soft segment and from about 20 to about 60 wt% hard segment selected from the group consisting trimethylene dicarboxylate ester and tetramethylene dicarboxylate ester, the process comprising: (a) preparing a package of partially oriented multifilament yarn by the process of claim 9; (b) unwinding the yarn from the package; (c) drawing the filaments to form a drawn yarn; (d) false-twist texturing the drawn yarn to form the textured yarn; and (e) winding the yarn onto a package.
- 20 25 15. Multifilament yarn prepared by the process of claim 1.
16. Fabric comprising the multifilament yarn of claim 15.