ABSTRACT OF THE DISCLOSURE

Fibrous articles (such as textile yarn, hemp rope, and tire cord) are sized with sizing compositions comprising linear, water-dissipatable polymers derived from at least one dicarboxylic acid component, at least one diol component, at least 20 mole percent of said diol component being a poly(ethylene glycol), and a diffusional monomer containing a \(-\text{SO}_3\text{M}\) group attached to an aromatic nucleus, wherein M is hydrogen or a metal ion.

This invention relates to sizing compositions and to fibrous articles sized therewith. In one of its more specific aspects, this invention relates to sizing compositions for textile yarns made from linear polymers.

When textile materials are to be used in the form of multifilament yarns for the fabrication of textile materials, it is desirable before the weaving process to treat the warp yarn with a sizing composition (sometimes referred to as an "agent") which adheres to and binds the several filaments together. This treatment strengthens the several filaments and renders them more resistant to abrasion during the subsequent weaving operations. It is especially important that the sizing composition impart abrasion resistance to the yarns during weaving because abrasion tends to sever the yarn and to produce end breaks which, of course, lower the quality of the final woven product. It is also important that the sizing composition be one which can be subsequently removed from the yarns by scouring.

Various high molecular weight materials have been suggested as sizes for yarns. Among such materials are gelatin, sodium polycrylate, polyvinyl alcohol, and the sodium salt of a 30/70 maleic anhydride-styrene copolymer. However, some of these materials are not readily compatible with or do not adhere well to textile yarns and thus do not form a protective coating or film thereon. Other materials coat the yarn but do not impart more than a slight degree of abrasion resistance. Therefore, a need exists for sizing compositions which avoid the above-mentioned disadvantages.

It is an object of this invention to provide sizing compositions, fibrous articles sized therewith, and processes for sizing said fibrous articles. Another object of this invention is to provide sizing compositions for textile yarns, especially those yarns made from linear polymers. Still another object of this invention is to provide sizing compositions which will adhere to and bind together the several filaments of textile yarns. Still another object of this invention is to provide sizing compositions which will impart abrasion resistance to textile yarns during weaving. Yet another object of this invention is to provide sizing compositions which can be removed from textile yarns by scouring. Other objects of this invention will appear herein.

These and other objects are attained through the practice of this invention, one embodiment of which comprises providing a fibrous article sized with a sizing composition comprising a linear, water-dissipatable polyester derived essentially from components (A) at least one dicarboxylic acid, (B) at least one diol, at least 20 mole percent of said diol component being a poly(ethylene glycol) having the formula 

\[\text{HOC}_2\text{H}_2\text{O}\text{OH}\]

wherein n is an integer of from two to about ten, and (C) a diffusional monomer containing a \(-\text{SO}_3\text{M}\) group attached to an aromatic nucleus, wherein M is hydrogen or a metal ion.

Another embodiment of this invention through which the above objects are attained comprises providing a process for sizing a fibrous article, wherein said process comprises applying to said fibrous article a sizing composition as described above.

The term "dissipatable" is to be understood to refer to the action of water or of an aqueous solution (preferably at least 25 percent by weight of water) on the polyester sizing composition. This term is specifically intended to cover those situations wherein the polyester sizing composition is dissolved or dispersed in water or an aqueous solution.

We have discovered that the above-described non-crystalline polymers are effective, when dissolved in water or aqueous solutions, as sizes for a variety of natural and synthetic textile yarns. Examples of such yarns include those made from polymers, such as poly(ethylene terephthalate) and poly(1,4-cyclohexylenedimethylene terephthalate), cotton, rayon, cellulose acetate, nylon, and polypropylene. Therefore, although this invention will be illustrated by references to polymers, our sizing composition may be used on these other types of textile materials with good results obtained.

The sizing compositions of this invention are particularly useful for sizing polyester yarn, which is among the most difficult of all textile yarns to size. In fact, presently no really effective sizing composition is known for continuous filament polyester fibers, and an expensive twist must be imparted to protect them during weaving. The sizing compositions of this invention make it possible to weave low or zero twist polyester fibers with substantially no defects.

It is necessary for greater effectiveness that a textile size be substantially scoured or removed from the woven fabric so that it will not interfere with subsequent finishing and dyeing operations. In practical terms, this means that the sizing composition must be water-dissipatable (that is, either water-soluble or water-dispersible). The non-crystalline polymers that are effective as sizes in the process of this invention contain a hydrophobic moiety and a hydrophilic moiety. For example, a preferred sizing composition is prepared from isophthalic acid, the sodium salt of 5-sulfosulfonic acid, and diethylene glycol. In this composition, the isophthalic acid is hydrophilic, the sodium sulfosulfonic acid is hydrophilic, and the diethylene glycol is hydrophilic. This particular composition, when added to water, forms a dispersion which exhibits a "dispersion viscosity" higher than that of water but lower than that which might be expected if the polymers were completely dissolved. As the temperature is raised from 25° C. to about 90° C., no appreciable increase in dispersion or solution viscosity results. Thus, this particular composition acts as if it were partially soluble in water and partially insoluble, a behavior which is consistent with its hydrophobic-hydrophilic composition. In addition, the hydrophilic portion of the molecule can be increased and a completely water-soluble composition obtained. Conversely, the hydrophilic moiety of the polyester molecule can be increased and a composition which is water-dispersible but which imparts little or no additional viscosity to water can be used. It is desirable that the size composition, on evaporation of the water, (1)
adhere to the fiber being sized, (2) form a sufficiently protective film so that the fiber is protected during weaving, and (3) be removable from the fiber under ordinary conditions of scouring.

The efficiency of a material as a size can be determined by measuring its Duplan abrasion resistance—the higher the Duplan value, the better it protects the yarn. The Duplan Cohesion Tester is a machine designed to test the effectiveness of a size solution by testing the cohesion of the sized filament yarn before the warp reaches the loom. Samples of sized yarn under a constant tension are abraded by friction plates moving back and forth on the yarn at a constant rate. The average number of strokes per strand required to separate the filaments in ten strands of yarn are reported as the Duplan value. Forty tests on each sample are considered the standard test. In examining the strands at intervals for open places, an air jet is blown along each strand to assist in detecting openings.

The novel sizes of this invention may be used in the following manner: the size, in powder or pellet form, is added to water at any convenient temperature between somewhat below room temperature to about 100°C and is subjected to mild agitation. Depending upon the specific properties of the particular composition being used, a clear to cloudy, moderately viscous, stable dissipation of the size in water is obtained.

The yarns to be sized are the highly polymeric, fiber- and film-forming, linear polymers derived from at least one aliphatic, cycloaliphatic, or aromatic dicarboxylic acid and at least one aliphatic, cycloaliphatic, or aromatic diol. The preparation of these polymers and the spinning of fibers therefrom are well-known procedures and need not be detailed herein. The acids and diols described below for the sizing composition are examples of components from which these polymers can be prepared.

The dicarboxylic acid component from which the linear, water-dissipatable polyester sizing composition is prepared can be any aliphatic, cycloaliphatic, or aromatic acid. Examples of such dicarboxylic acids include oxalic; malonic; dimethylmalonic; succinic; glutaric; adipic; trimethylene dicarboxylic; pimelic; 2,2-dimethyl adipic; azelaic; sebacic; fumaric; maleic; itaconic; 1,3-cyclopentanedicarboxylic; 1,2-cyclohexanedicarboxylic; 1,3-cyclohexanedicarboxylic; 1,4-cyclohexanedicarboxylic; phthalic; terephthalic; isophthalic; 2,5-norbornanedicarboxylic; 1,4-naphthalic; diphenic; 4,4'-oxydibenzoic; dicyclo; thiodipropionic; 4,4'-sulfonyldibenzoic; and 2,2'-dicarboxylic acids. The terephthalic acid is used as the dicarboxylic acid component of the polyester, especially good results are achieved when at least five mole percent of one or the other acids listed above is used.

It should be understood that the use of the corresponding acid anhydrides, esters, and acid chlorides of these acids is included in the term "dicarboxylic acid." The esters are preferred, examples of which include dimethyl 1,4-cyclohexanedicarboxylate; dimethyl 2,6-naphthalenedicarboxylate; dibutyl 4,4'-sulfonyldibenzoate; dimethyl isophthalate; dimethyl terephthalate; and diphenyl terephthalate. Copolymers may be prepared from two or more of the above dicarboxylic acids or derivatives thereof.

At least about 20 mole percent of the diol component used in preparing the polyester sizing composition is a poly(ethylene glycol) having the formula

\[ \text{HOC(OCH}_2\text{CH}_2\text{O)}_n\text{OH} \]

wherein \( n \) is an integer of from two to about ten. Examples of suitable poly(ethylene glycols) include diethylene, triethylene, tetraethylene, pentaethylene, hexaethylene, heptaeethylene, octaethylene, nonaethylene, and decaethylene glycols, and mixtures thereof. Preferably the poly(ethylene glycol) employed in the present invention is diethylene glycol, triethylene glycol, or mixtures thereof. The remaining portion of the diol component is at least one aliphatic, cycloaliphatic, or aromatic diol. Examples of these diols include ethylene glycol; propylene glycol; 1,3-propanediol; 2,4-dimethyl-2-ethylhexane-1,3-diol; 2,2-dimethyl-1,3-propanediol; 2-ethyl-2-buty1-1,3-propanediol; 2-ethyl-2-isobutyl-1,3-propanediol; 1,4-butanediol; 1,3-butanediol; 1,5-pentanediol; 1,6-hexanediol; 2,2,4-trimethyl-1,6-hexanediol; 1,2-cyclohexanediol; 1,3-cyclohexanediol; 1,4-cyclohexanediol; 2,2,4,4-tetramethyl-1,3-cyclobutandiol; and p-xylene diol. Copolymers may be prepared from two or more of the above diols.

A third component used to prepare the polyester sizing composition is a difunctional monomer containing a functional group of an aromatic nucleus, wherein M is hydrogen or a metal ion. This difunctional monomer component may be either a dicarboxylic acid (or derivative thereof) containing a —SO\(_3\)M group or a diol containing a —SO\(_3\)M group. The metal ion of the sulfonate salt group may be Na\(^+\), Li\(^+\), K\(^+\), Mg\(^{2+}\), Ca\(^{2+}\), Cu\(^{2+}\), Fe\(^{3+}\), or Fe\(^{3++}\). It is possible to prepare the polyester using, for example, a sodium sulfonate salt and later by ion-exchange replace this ion with a different ion (for example, calcium) and thus alter the characteristics of the polyester.

The —SO\(_3\)M group is attached to an aromatic nucleus, examples of which include benzene, naphthalene, anthracene, diphenyl, oxydiphenyl, sulfonyldiphenyl, and methylenediphenyl.

Especially good results are obtained when the difunctional monomer is the sodium salt of a sulfophtalic acid, sulfoterephthalic acid, sulfophthalic acid, or 4-sulfophthalic acid, 7,12-dicarboxylic acid (or derivatives of such acids). A highly preferred such monomer is 5-sodiumsulfophthalic acid or a derivative thereof such as 5-sodiumsulfodimethyl isophthalate. Another preferred difunctional monomer is 5-sodiumsulfophthalic acid. Monomers containing a —SO\(_3\)M group are described in Kibler et al. U.S. Ser. No. 695,539, filed Jan. 3, 1968 now abandoned in favor of a continuation-in-part.

Other effective difunctional monomers containing a —SO\(_3\)M group attached to an aromatic nucleus include metal salts of aromatic sulfonic acids (or esters thereof).

These monomers have the general formula

\[ \text{ROOC}_2\text{O-SO}_3\text{M} \]

wherein X is a trivalent aromatic hydrocarbon radical, Y is a divalent aromatic hydrocarbon radical, R is hydrogen or an alkyl group of one to four carbon atoms, M is hydrogen, Na\(^+\), Li\(^+\), K\(^+\), Mg\(^{2+}\), Ca\(^{2+}\), Cu\(^{2+}\), Fe\(^{3+}\), or Fe\(^{3++}\), and \( x \) is 1, 2, or 3. These monomers are described, including methods for their preparation, in Lappin et al., U.S. Ser. No. 695,349 filed Jan. 3, 1968 now U.S. Pat. No. 3,529,947, patented on Sept. 15, 1970. Examples of preferred monomers here are 4-sodiumsulfophenyl-3,5-dicarboxybenzenesulfonate, 4-lithiosulfophenyl-3,5-dicarboxybenzenesulfonate; and 6-sodiumsulf-2-naphthalene-3,5-dicarboxybenzenesulfonate.

Other effective difunctional monomers containing a —SO\(_3\)M group attached to an aromatic nucleus include metal salts of sulfodiphenyl ether dicarboxylic acids (or esters thereof). These monomers have the general formula

\[ \text{ROOC}_2\text{O-SO}_3\text{M} \]

wherein R is hydrogen, an alkyl group of one to eight carbon atoms, or phenyl, and M is hydrogen, Na\(^+\), Li\(^+\), Mg\(^{2+}\), Ca\(^{2+}\), Cu\(^{2+}\), Fe\(^{3+}\), or Fe\(^{3++}\), and \( x \) is 1, 2, or 3. These monomers are described, including methods for their preparation, in Lappin et al. U.S. Ser. No. 671,565, filed Sept. 29, 1967 now abandoned and substituted by streamlined continuation Ser. No. 835,295 which was published Nov. 18, 1969 as a Defensive Publication, 868, O.G. 730. Examples of preferred monomers here are dimethyl
5 positions of a polymer containing the —SO₃M groups are an acid or derivative thereof (such as its ester), the polyester should contain at least about eight mole percent of the monomer based on total acid content, with more than ten mole percent giving particularly advantageous results. When the difunctional monomer is a diol, the polyester should also contain at least about eight mole percent of the monomer based on total diol content, with more than ten mole percent giving particularly advantageous results. Greater dissipatability is achieved when the difunctional monomer constitutes from about 12 mole percent to about 45 mole percent of the total content of acid or diol components of the polyester.

To obtain the polyester sizing compositions of this invention, the difunctional monomer containing the —SO₃M group may be added directly to the esterification reaction mixture from which the polyester will be made. Thus, these monomers can be used as a component in the original polyester reaction mixture. Other various processes which may be employed in preparing these sizing compositions are well known in the art and are illustrated in such patents as U.S. 2,465,319; 3,018,272; 2,901,466; and 3,075,952. These patents illustrate ester interchange and polymerization processes.

Both the fiber-forming polyesters to be sized and the polyester sizing compositions will have an inherent viscosity (I.V.) of at least 0.3, as measured at 25°C. using 0.25 gram of polymer per 100 ml of a solvent composed of 60 percent phenol and 40 percent tetrachloroethane.

Various additives may be incorporated into the sizing compositions to achieve specific results. Examples of such additives include size (to prevent the powder or pellets from adhering together), titanium dioxide, dyes, other pigments, and stabilizers.

The following examples are included for a better understanding of this invention.

**EXAMPLE 1**

A mixture of 48.5 grams (0.25 mole) of dimethyl isophthalate, 24.2 grams (0.125 mole) of dimethyl terephthalate, 15 grams (0.075 mole) of hexahydroxyisophthalic acid, 14.8 grams (0.05 mole) of dimethyl 5-sodiumsulfophenylisophthalate, 68.9 grams (0.65 mole) of diethylene glycol, and 0.8 ml. of a 21 percent catalyst solution of titanium isopropoxide is stirred and heated at 200°C and a vacuum of 0.3 mm. is applied. Heating and stirring is continued for one hour under these conditions. After cooling the polymer obtained has an I.V. of 0.53 and is tough and rubbery. It is dissipated in hot water to the extent of about 20 weight percent to give a clear, slightly viscous solution. After storage of the solution for three months at room temperature, the water is evaporated from a portion. The residual polymer has an I.V. of 0.54. No measurable hydrolysis has occurred.

**EXAMPLE 2**

The following table shows the properties of a number of polyester sizing compositions made from difunctional monomers containing —SO₃M groups. All are made by the general procedure of Example 1.

| Polymer | Dicarboxylic acid | Mole Percent | Sulfonate | Mole Percent | Diol | Mole Percent | I.V.
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<td>A</td>
<td>Isophthalic</td>
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<td>0.54</td>
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<td>B</td>
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<td>10</td>
<td>10</td>
<td>DEG</td>
<td>100</td>
<td>0.53</td>
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<tr>
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<td>Hexahydroxyterephthalic</td>
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<td>25</td>
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<td>0.53</td>
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<td>F</td>
<td>Hexahydroxyterephthalic</td>
<td>15</td>
<td>15</td>
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<td>100</td>
<td>0.53</td>
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<td>H</td>
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<tr>
<td>I</td>
<td>Terephthalate</td>
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¹ SLP=4-sodiumsulfophenyl terephthalic acid.
² DEG=diethylene glycol.
³ EG=ethylene glycol.
⁴ +sodiumsulfophenyl 3,5-dicarboxybenzene sulfonate,
EXAMPLE 3

A mixture of 15 parts of the polyester sizing composition (I.V. of 0.5) derived from 90 mole percent isophthalic acid, 10 mole percent 5-sodiumsulfonlosiphthalic acid, and diethylene glycol and 85 parts of water gives a dispersion that is slightly hazy in appearance and that has a Brookfield viscosity of 15. A poly(ethylene terephthalate) yarn containing 40 filaments is passed through the aqueous dispersion of the sizing composition and dried. A 4.5 percent pickup of the size results. Pickup is a measurement of the weight percent of the size relative to the weight of the sized yarn. The DuPon abrasion resistance of the sized yarn is 275, showing that this polyester is an excellent size for polyester yarn.

EXAMPLE 4

The conditions of Example 1 are repeated except that 7.5 parts of the polyester sizing composition are suspended in 92.5 parts of water. A 2.7 percent pickup of size results. The DuPon abrasion resistance of this sized yarn is 244, indicating that this polyester is an excellent size for polyester yarns even at low "pickup."

EXAMPLE 5

The conditions of Example 1 are repeated except that 12.5 parts of gelatin are dissolved in 87.5 parts of water. The size pickup is not determined, but the supposedly sized polyester yarn has a DuPon abrasion resistance of only 13. This shows that gelatin is not a satisfactory size for polyester fibers.

It will be noted that in the preceding examples, size/water ratios (weight/weight) of 15/85 and of 7.5/92.5 are used. However, the size/water ratio may vary from 1/99 to 50/50.

When the size is a relatively hydrophobic, it does not impart much viscosity to the aqueous size composition, and very high "solids" size compositions can be prepared. In addition, in the case of relatively hydrophobic polyester sizes, very high molecular weight products can be made without the formation of a size composition that is too viscous to use. This can be a real advantage when it is desirable to use, for example, a high I.V. size material for high toughness. The extent to which the yarn being sized "pick up" size will be affected by the solution or suspension viscosity of the size composition, with more viscous compositions in general leading to higher "pick-ups." It is within the scope of this invention to add various thickening agents and the like to the size composition to increase its viscosity.

Other polyester yarns and other polyester sizing compositions (such as those from the table) can be substituted in the procedure of Example 1 and good results obtained. Although described above with particular reference to textile yarns, this invention encompasses fibrous articles in general, other examples of which are tire cord (such as rayon, nylon, or polyester tire cord) and hemp rope. In sizing these other fibrous articles, this invention does not necessarily require the removal of the sizing composition.

The invention has been described in detail with particular reference to preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention as described hereinabove and as defined in the appended claims.

We claim:

1. A fibrous article sized with a sizing composition comprising a linear, water-dissipatable polyester derived essentially from components
   (A) at least one dicarboxylic acid,
   (B) at least one diol, at least 20 mole percent of said diol component being a poly(ethylene glycol) having the formula
   \[ HOC(CH_2CH_2O)_nOH \]
   wherein \( n \) is an integer of from two to about ten, and
   (C) at least one difunctional dicarboxylic acid sulfonmonomer containing a \(-\text{SO}_3\text{H}\) group attached to an aromatic nucleus, wherein \( M \) is hydrogen or Na, Li, K, Mg\(^{2+}\), Ca\(^{2+}\), Cu\(^{2+}\), Fe\(^{3+}\), or a combination thereof, said sulfonmonomer component constituting at least about 8 mole percent to about 45 mole percent of the sum of the moles of said components (A) and (C).

2. A fibrous article as defined by claim 1 wherein said component (A) is isophthalic acid.

3. A fibrous article as defined by claim 1 wherein said component (A) is terephthalic acid.

4. A fibrous article as defined by claim 1 wherein said component (A) is adic acid.

5. A fibrous article as defined by claim 1 wherein said component (A) is hexahydraterhetherhalic acid.

6. A fibrous article as defined by claim 1 wherein said component (A) is a mixture of isophthalic, terephthalic, and hexahydrosisophthalic acids.

7. A fibrous article as defined by claim 1 wherein said component (B) is diethylene glycol.

8. A fibrous article as defined by claim 1 wherein said component (B) is triethylene glycol.

9. A fibrous article as defined by claim 1 wherein said component (B) is a mixture of 80 mole percent diethylene glycol and 20 mole percent ethylene glycol.

10. A fibrous article as defined by claim 1 wherein said component (B) is a mixture of 70 mole percent diethylene glycol and 30 mole percent 1,4-cyclohexanediol.

11. A fibrous article as defined by claim 1 wherein said component (C) is 5-sodiumsulfisophthalic acid.

12. A fibrous article as defined by claim 1 wherein said component (C) is 4-sodiumsulfophenyl-3,5-dicarboxypentene sulphonate.

13. A fibrous article as defined by claim 1 wherein said component (C) is 5-[4-(sodiumsulfophenoxy)]isophthalic acid.

14. A fibrous article as defined by claim 1 wherein said component (C) is 2(2'-sodiumsulfophenyl)-2-ethylmalonic acid.

15. A fibrous article as defined by claim 1 wherein said fibrous article is a textile yarn.

16. A fibrous article as defined by claim 15 wherein said textile yarn is made from a polyester.

17. A textile yarn as defined by claim 16 wherein said polyester is poly(ethylene terephthalate).

18. A textile yarn as defined by claim 16 wherein said polyester is poly(1,4-cyclohexylenedimethylene terephthalate).

19. A fibrous article as defined by claim 1 wherein said water-dissipatable polyester has an inherent viscosity of at least about 0.3, as measured at 25 °C, at a concentration of about 0.25 gram of said polyester per 100 mL of a solvent composed of 60 percent phenol and 40 percent tetrachloroethane.

20. A fibrous article as defined by claim 19 wherein said sulfo-monomer component constitutes at least about 12 mole percent of the sum of the moles of said component (A), (B), and (C).

21. A fibrous article sized with a sizing composition comprising a linear, water-dissipatable polyester or polysteramide derived from at least two difunctional monomer components which are dicarboxylic acid, hy-
droxycarboxylic acid having one aliphatic hydroxy group, aminocarboxylic acid, amino-alcohol having one aliphatic hydroxy group, a glycol having two aliphatic hydroxy groups, and an organic diamine including combinations thereof wherein the overall total moles of said monomer components comprises at least 10 mole percent of at least one or a mixture of poly(ethylene glycols) containing from 2 to 10 ethyleneoxy groups and at least 4 mole percent of at least one or a mixture of difunctional sulfonmonomer components containing at least one $-\text{SO}_3\text{M}$ group attached to an aromatic nucleus, said nucleus being present in at least one of the aforesaid monomer components wherein $\text{M}$ is hydrogen or $\text{Na}^+$, $\text{Li}^+$, $\text{K}^+$, $\text{Mg}^{++}$, $\text{Ca}^{++}$, $\text{Cu}^{++}$, $\text{Fe}^{++}$, $\text{Fe}^{+++}$, or a combination thereof, said polyester or polyesteramide having an inherent viscosity of at least about 0.1 measured as defined in this specification.

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U.S. Cl. X.R.

117—139.5; 260—40