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(54) Title: THERMOTROPIC LIQUID CRYSTALLINE POLYMERS AS STABILIZERS IN THERMOPLASTIC POLYESTERS		
(57) Abstract <p>A method for lessening the shear or heat induced reduction in molecular weight of a thermoplastic polyester, and compositions used in and obtained from such method, comprising: admixing at least 50 parts by weight of a first polymer with less than or equal to 50 parts by weight of a second polymer, the first polymer being a thermoplastic polyester, and the second polymer being a TLCP, to obtain a polyester/TLCP mixture, and subjecting the polyester/TLCP mixture to heat or shear, wherein the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping melt processing temperature range; the first polymer and the second polymer are immiscible or partly immiscible when mixed within the overlapping temperature range; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.</p>		

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**THERMOTROPIC LIQUID CRYSTALLINE POLYMERS AS
STABILIZERS IN THERMOPLASTIC POLYESTERS**

5 **CROSS REFERENCE TO RELATED APPLICATIONS**

 This application claims priority upon provisional application Serial No. 60/062,016 filed October 10, 1997, and the 60/062,016 application is herein incorporated by this reference in its entirety.

10

BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION

15 This invention relates generally to polyester resins, to the use of thermotropic liquid crystalline polymers to improve the properties of such resins, and to polyester resins containing such thermoplastic liquid crystalline polymers.

 In many processes for producing articles from polyester resins the resins are
20 processed in a manner that subjects the resins to potentially damaging mechanical shear and/or heat. Process steps that cause such shear and heat include, for example, extrusion, compounding and molding. During these process steps the molecular weight of a polyester is often reduced. It is generally desirable to minimize the molecular weight loss during these processing operations, however,
25 in order to provide optimum mechanical properties in the final articles. Various techniques are known in the art for minimizing this molecular weight loss, although none has yet been totally satisfactory.

 This invention concerns the use of thermotropic liquid crystalline polymers

("TLCPs") to minimize the reduction of molecular weight in polyesters during processing. The use of TLCPs to reduce the viscosity of polyesters is known in the art, as described in Cogswell et al. in U.S. Patents 4,433,083 and 4,386,174, and by La Mantia in Thermotropic Liquid Crystal Polymer Blends, Technomic Publishing Co. (1993). The rheology of polyester/TLCP blends is complex, with numerous examples of significant negative deviations from linear mixing rules. As reported by Cogswell, for example, blends of miscible polyesters and TLCP can have viscosities substantially lower than either component individually.

In the present invention immiscible TLCPs that comprise aromatic hydroxyl residues are added to polyesters before the polyesters are processed in order to minimize the molecular weight loss that occurs during the processing. In contrast to Cogswell, a TLCP and polyester are chosen that are at least partly immiscible. The addition of TLCP to the polyester before processing minimizes the amount of polyester molecular weight loss during processing, when compared to the polyester molecular weight loss that occurs when the polyester is processed without TLCP. In many instances, the addition of TLCP even increases the viscosity of the polyester, in further contrast to the teachings of Cogswell et al.

SUMMARY OF THE INVENTION

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In accordance with the purpose(s) of this invention, as embodied and broadly described herein, this invention, in one aspect, relates to a composition comprising at least 50 parts by weight of a first polymer, the first polymer being a thermoplastic polyester; and less than or equal to 50 parts by weight of a second polymer that is a TLCP; wherein: the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping melt processing temperature range; the first polymer and the second polymer are immiscible or partly immiscible when mixed within the overlapping temperature range; and the first polymer and the second polymer are

provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.

5 In another aspect the invention relates to a composition comprising at least 50 parts by weight of a first polymer, the first polymer being a thermoplastic polyester; and less than or equal to 50 parts by weight of a second polymer that is a TLCP; wherein: the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping
10 melt processing temperature range; the first polymer and the second polymer are present in a weight ratio of at least 10:1; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.

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 In another aspect the invention relates to a method for lessening the shear or heat induced reduction in molecular weight of a thermoplastic polyester, comprising: admixing at least 50 parts by weight of a first polymer with less than or equal to 50 parts by weight of a second polymer, the first polymer being a
20 thermoplastic polyester, and the second polymer being a TLCP, to obtain a polyester/TLCP mixture, and subjecting the polyester/TLCP mixture to heat or shear, wherein the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping melt processing temperature range; the first polymer and the second polymer are
25 immiscible or partly immiscible when mixed within the overlapping temperature range; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.

In another aspect the invention relates to a method for lessening the shear or heat induced reduction in molecular weight of a thermoplastic polyester, comprising: admixing at least 50 parts by weight of a first polymer with less than or equal to 50 parts by weight of a second polymer, the first polymer being a thermoplastic polyester, and the second polymer being a TLCP, to obtain a polyester/TLCP mixture, and subjecting the polyester/TLCP mixture to at least 1500 s⁻¹ units of shear, wherein: the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping melt processing temperature range; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.

Additional advantages of the invention will be set forth in part in the description which follows, and in part will be obvious from the description, or may be learned by practice of the invention. The advantages of the invention will be realized and attained by means of the elements and combinations particularly pointed out in the appended claims. It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the invention, as claimed.

DESCRIPTION OF THE INVENTION

The present invention may be understood more readily by reference to the following detailed description of the invention and to the examples included herein. Before the present compounds, compositions and methods are disclosed and described, however, it is to be understood that this invention is not limited to specific synthetic methods, or to particular formulations or processing regimens, as

such may, of course, vary. It is also to be understood that the terminology used herein is for the purpose of describing particular embodiments only and is not intended to be limiting.

5 It must be noted that, as used in the specification and the appended claims, the singular forms "a," "an" and "the" include plural referents unless the context clearly dictates otherwise. Thus, for example, reference to "an aromatic compound" includes mixtures of aromatic compounds.

10 In this specification and in the claims which follow, reference will be made to a number of terms which shall be defined to have the following meanings:

 References in the specification and concluding claims to parts by weight, of a particular element or component in a composition or article, denotes the weight
15 relationship between the element or component and any other elements or components in the composition or article for which a part by weight is expressed. Thus, in a compound containing 2 parts by weight of component X and 5 parts by weight component Y, X and Y are present at a weight ratio of 2:5, and are present in such ratio regardless of whether additional components are contained in the
20 compound.

 A residue of a chemical species, as used in the specification and concluding claims, refers to the moiety that is the reaction product of the chemical species in a particular reaction scheme or subsequent formulation or chemical product,
25 regardless of whether the moiety is actually obtained from the chemical species. Thus, an ethylene glycol residue in a polyester refers to one or more -OCH₂CH₂O- units in the polyester, regardless of whether ethylene glycol was used to prepare the polyester. Similarly, a sebacic acid residue in a polyester refers to one or more -CO(CH₂)₈CO- moieties in the polyester, regardless of whether the residue is

obtained by reacting sebacic acid or an ester thereof to obtain the polyester.

"Optional" or "optionally" means that the subsequently described event or circumstance may or may not occur, and that the description includes instances
5 where said event or circumstance occurs and instances where it does not. For example, the phrase "optionally substituted lower alkyl" means that the lower alkyl group may or may not be substituted and that the description includes both unsubstituted lower alkyl and lower alkyl where there is substitution.

10 Ranges are often expressed herein as from about one particular value, and/or to about another particular value. When such a range is expressed, it is to be understood that a more preferred range is typically from the one particular value and/or to the other particular value. Similarly, when values are expressed as approximations, by use of the antecedent "about," it will be understood that the particular value is
15 typically more preferred.

By the term "effective amount" of a compound as provided herein is meant such amount as is capable of performing the function of the compound for which an effective amount is expressed. As will be pointed out below, the exact amount
20 required will vary from process to process, depending on a host of recognizable variables such as the compounds employed and the processing conditions observed. Thus, it is not possible to specify an exact "effective amount." However, an appropriate effective amount may be determined by one of ordinary skill in the art using only routine experimentation.

25 The terms "polymer," and "resin" are used interchangeably herein, and each term is meant to include any of various substances made by chemical synthesis, especially those used in the making of plastics, any compound formed by polymerization, and any of two or more polymeric compounds.

The term "modified" is often used herein to describe polymers and means that a particular monomeric unit that would typically make up the pure polymer has been replaced by another monomeric unit that shares a common polymerization
5 capacity with the replaced monomeric unit. Thus, for example, it is possible to substitute diol residues for glycol in poly(ethylene glycol), in which case the poly(ethylene glycol) will be "modified" with the diol. If the poly(ethylene glycol) is modified with a mole percentage of the diol, then such a mole percentage is based upon the total number of moles of glycol that would be present in the pure
10 polymer but for the modification. Thus, in a poly(ethylene glycol) that has been modified by 50 mole % with a diol, the diol and glycol residues are present in equimolar amounts.

"TLCP" is the abbreviated term for "thermotropic liquid crystalline
15 polymer", as distinct from lyotropic liquid crystalline polymer. Liquid crystalline polymers are well known in the art and are characterized by being capable of maintaining a high degree of intermolecular order in a liquid state, even in the absence of anisotropic shear forces, and by exhibiting a behavior pattern characteristic of a crystalline liquid. Thermotropic LCP's such as Vectra, give rise
20 to this ordered system in the molten state, while lyotropic LCP's such as Kevlar, give rise to this ordered system in solution. These ordered systems may generally be classified as nematic, smectic, or cholesteric, and all of these classes of TLCP are included in this invention. TLCP's are reviewed, for example, in F.P.
LaMantia, Thermotropic Liquid Crystalline Blends, Technomic Publishing Co.
25 (1993). The disclosure of this publication is hereby incorporated by reference as if fully set forth herein.

"PET-HB" is the abbreviated term for poly(ethylene terephthalate-co-p-hydroxybenzoate), meaning a copolymer of p-hydroxybenzoyl and

oxyethyleneoxyterephthaloyl residues. Such polymers may be prepared by modifying poly(ethylene terephthalate) to contain the p-hydroxybenzoate residues. A numeric designation, such as the 80 in PET-80HB, refers to the molar percentage of p-hydroxybenzoate residues in the copolymer.

5

The term "melt processing range" as used herein refers to the temperature range between which the polymer begins to flow and where the thermal degradation results in unacceptable loss of properties.

10

The term "polyester" includes copolyesters.

When referenced herein, inherent viscosity of TLCPs is measured at 25 C using 0.5 wt.% polymer in pentafluorophenol. The inherent viscosity of polyesters is measured using 0.5 wt.% polyester in 60/40 phenol/tetrachloroethane at 25 C.

15

In one aspect the invention relates to a composition comprising at least 50 parts by weight of a first polymer, the first polymer being a thermoplastic polyester; and less than or equal to 50 parts by weight of a second polymer that is a TLCP; wherein: the second polymer comprises at least one monomeric aromatic hydroxyl
20 residue; the first polymer and the second polymer have an overlapping melt processing temperature range; the first polymer and the second polymer are immiscible or partly immiscible when mixed within the overlapping temperature range; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat
25 processing is greater than the molecular weight of the first polymer alone after shear or heat processing. The composition is preferably in the form of an inhomogeneous solid phase blend. In a separate embodiment, the polyester and TLCP are chosen in amounts so that the viscosity of the polyester without the TLCP is less than the viscosity of a composition containing both the polyester and

the TLCP. Moreover, in one embodiment the melt viscosity of the polyester/TLCP mixture, measured at 1000 s^{-1} or higher, is 5% or more higher than the melt viscosity of the first polymer.

5 In another aspect the invention relates to a composition comprising at least 50 parts by weight of a first polymer, the first polymer being a thermoplastic polyester; and less than or equal to 50 parts by weight of a second polymer that is a TLCP; wherein: the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping
10 melt processing temperature range; the first polymer and the second polymer are present in a weight ratio of at least 10:1; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.

15

 In another aspect the invention provides a method for lessening the shear or heat induced reduction in molecular weight of a thermoplastic polyester, comprising: admixing at least 50 parts by weight of a first polymer with less than or equal to 50 parts by weight of a second polymer, the first polymer being a
20 thermoplastic polyester, and the second polymer being a TLCP, to obtain a polyester/TLCP mixture, and subjecting the polyester/TLCP mixture to heat or shear, wherein the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping melt processing temperature range; the first polymer and the second polymer are
25 immiscible or partly immiscible when mixed within the overlapping temperature range; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.

In still another aspect the invention relates to a method for lessening the shear or heat induced reduction in molecular weight of a thermoplastic polyester, comprising: admixing at least 50 parts by weight of a first polymer with less than or equal to 50 parts by weight of a second polymer, the first polymer being a thermoplastic polyester, and the second polymer being a TLCP, to obtain a polyester/TLCP mixture, and subjecting the polyester/TLCP mixture to at least 1500 s⁻¹ units of shear, wherein: the second polymer comprises at least one monomeric aromatic hydroxyl residue; the first polymer and the second polymer have an overlapping melt processing temperature range; and the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing. Because the process lessens the reduction in polyester molecular weight, compositions obtained by the process often have improved physical properties such as modulus and elongation to break when compared with compositions not produced by the process.

The invention is particularly suitable for application to extrusion processes, wherein the shear induced reduction in molecular weight is applied by extrusion. The process has further particular application to processes in which the composition is subjected to over about 250 s⁻¹ units of shear. The invention may preferably be employed in processes in which the composition is subjected to over about 1000 s⁻¹ units of shear, even more preferably to processes in which the composition is subjected to over about 1500 s⁻¹ units of shear, and still even more preferably to processes in which the composition is subjected to over about 2500 s⁻¹ units of shear. A preferred range in which to practice the invention is from about 500 to about 25,000 s⁻¹ units of shear, and an even more preferred range is from about 3,000 to about 9,000 s⁻¹ units of shear and a still even further preferred range is

from about 4,000 to about 10,000 s⁻¹ units of shear.

A particular advantage from the process of the present invention is the reduction in shear-induced damage to molecular weight of the polyesters in thermoplastic polymers. In the process of the current invention the molecular weight loss of the polyester is preferably reduced on average by 5% or more, when compared to polyesters processed conventionally, and even more preferably, the loss in molecular weight is reduced on average by 10% or more, when measured by GPC.

10

The first and second polymers preferably should be chosen so that they are immiscible or partly immiscible in an effective amount to reduce the shear induced reduction in molecular weight that otherwise would occur during processing of the first polymer in the absence of the TLCP. The polymers are compatible, and may be used in the compositions of the present invention, if they evidence any effective amount of immiscibility. The miscibility and potential compatibility of polymers is discussed in M.M. Coleman et al., Specific Interactions and the Miscibility of Polymers, Technomic Publishing, Lancaster, Pennsylvania, Chapter 2 (1991), the disclosure of which is hereby incorporated by reference as if fully set forth herein.

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The effective amount of miscibility of the polymers can, in many instances, be expressed numerically by measuring or identifying the difference between the solubility parameters for the polymers. Polymers that have solubility parameters that differ by about 0.3 (J/cm³)^{0.5} and greater are generally immiscible, and are particularly suitable for practicing the present invention. In another embodiment the difference in solubility parameters of the first polymer and second polymer is greater than about 0.5 (J/cm³)^{0.5}, in another embodiment the difference in solubility parameters of the first polymer and second polymer is greater than about 0.7 (J/cm³)^{0.5}, in yet another embodiment the difference in solubility parameters of the

first polymer and second polymer is greater than about $1.0 \text{ (J/cm}^3\text{)}^{0.5}$, in still another embodiment the difference in solubility parameters of the first polymer and second polymer is greater than about $1.3 \text{ (J/cm}^3\text{)}^{0.5}$, and in a still even further embodiment the difference in solubility parameters of the first polymer and second polymer is
5 greater than about $1.6 \text{ (J/cm}^3\text{)}^{0.5}$.

The composition of the present invention may comprise the first and second polymers at any ratio, although it is generally preferred that the first and second polymers be present in the composition in effective amounts to reduce the shear
10 induced reduction in molecular weight that otherwise would occur during processing of the first polymer in the absence of the TLCP. The polymers should also be present in an effective amount to prevent brittle failure problems in the final product due to the immiscibility of the polymers. The first and second polymers may preferably be present in effective amounts in which the weight ratio of second
15 polymer to first polymer is less than 1:10, and greater than 1:49. Even more preferably, the weight ratio of second polymer to first polymer is from about 1:14 to about 1:25, and most preferably the ratio is about 1:19.

Preferred compositions comprise from about 20 to about 99.5 percent by
20 weight of the first polymer, and from about 0.1 to about 50 percent by weight of the second polymer. Even more preferred compositions comprise from about 1 to about 20 percent by weight of the second polymer.

In order to obtain a suitable melt processable composition for the present
25 invention it is necessary that the first and second polymers be melt processable, and that the melt processing range for each of the first and second polymers overlap. Preferably the melt processing ranges overlap by at least 5 C, more preferably the ranges overlap by at least 10 C, more preferably at least 20 C, and most preferably the ranges overlap by from about 20 C to about 30 C. In order to optimize the

overlap of the melt processing temperature ranges of the first and second polymers, preferred thermoplastics for the first polymer of this invention, and preferred TLCPs for the second polymer, each have a melting point from about 200 C to about 400 C, and more preferably from about 250 C to about 350 C. Various means
5 are known in the art for modifying the melting point of a TLCP to increase the overlap of its melt processing range with the melt processing range of the polyester. For example, the melting point of a TLCP may be lowered by adding side groups (e.g., replacing hydroquinone with phenylhydroquinone), reducing the aromatic content (e.g., replacing hydroquinone with ethylene glycol), or adding bends into
10 the rigid sections (e.g., replacing hydroquinone with resorcinol). Care should be taken, however, not to substantially modify or replace the aromatic hydroxyl residues of the TLCP, or to affect the properties of the TLCP.

The first polymer may preferably be formed from one or more aliphatic,
15 aromatic, or alicyclic dibasic acids containing from about 2 to about 40 carbon atoms, and one or more aliphatic or alicyclic glycols containing from about 2 to about 40 carbon atoms. In a particularly preferred embodiment the first polymer comprises poly(ethylene terephthalate) (PET); poly(tetramethylene terephthalate) (PBT); poly(1,4-cyclohexylenedimethylene terephthalate (PCT); poly(ethylene
20 2,6-naphthalenedicarboxylate) (PEN); poly(ethylene 1,4-cyclohexanedicarboxylate) (PEC); a PET copolyester containing isophthalic acid residues; a PET copolyester containing 1,4-cyclohexane dimethanol residues; a PEN copolyester containing 1,4-cyclohexane dimethanol residues; a PEN copolyester containing terephthalic acid residues; a PEN copolyester containing 1,4-cyclohexane dimethanol residues;
25 and/or a PET copolyester containing isophthalic acid residues.

The first polymer may also be advantageously modified with from about 0.1 to about 25 mole % of aliphatic dibasic acids containing from 2 to about 40 carbon atoms. A particularly suitable first polymer comprises PEN formed from 2,6-, 2,7-,

1,4-, and/or 1,5-naphthalenedicarboxylic acid. The first polymer also preferably has an inherent viscosity of from about 0.4 to about 2.0 dl/g.

In general, any TLCP which contains an aromatic hydroxyl residue, and
5 which has a melt processing range that overlaps the processing range of the thermoplastic polyester used in the invention is operable. The TLCP can thus be formed from monomers that contain aromatic hydroxyl functionalities, and from monomers that before reacting to form the TLCP are converted to contain an aromatic hydroxyl functionality. Processes for preparing the TLCP are known in
10 the art, and include those discussed by Jackson, et al., in U.S. Patent 4,169,933 (the disclosure of which is hereby incorporated by reference). Particularly suitable TLCPs for the second polymer comprise residues of ethylene glycol, terephthalic acid, naphthalene-2,6-dicarboxylic acid, naphthalene-6-carboxyl-2-ol, hydroquinone, p-hydroxybenzoic acid, methylhydroquinone, phenylhydroquinone,
15 naphthalene-6-carboxyl-2-amide, p-aminobenzoic acid, and/or isophthalic acid. Of these, residues of terephthalic acid, naphthalene-2,6-dicarboxylic acid, hydroquinone, p-hydroxybenzoic acid or mixtures thereof, are more preferred.

In a particularly preferred embodiment of the current invention, the second
20 polymer is a poly(ethylene terephthalate) copolyester containing from about 50 to about 90 mole percent p-hydroxybenzoic acid residues (PET-HB). In an even more preferred embodiment of the current invention, the second polymer is a PET-HB containing from about 60 to about 85 mole percent p-hydroxybenzoic acid residues. The PET-HB copolyester may also be modified by up to 20 mole % with
25 one or more dibasic acids comprising from 2 to 40 carbon atoms. Particularly suitable dibasic acids include succinic, glutaric, adipic, sebacic, 1,4-cyclohexanedicarboxylic, isophthalic, naphthalenedicarboxylic, trans-stilbene-4,4'-dicarboxylic, and dimer acid. Preferred dibasic acids are terephthalic acid, naphthalene-2,6-dicarboxylic acid, hydroquinone, and p-hydroxybenzoic acid as

disclosed by Jackson, et al., in U.S. Patent 4,169,933 (the disclosure of which is hereby incorporated by reference). The PET-HB can similarly be modified with up to 20 mole % of one or more glycols containing from 2 to about 12 carbon atoms. Preferred modifying glycols include propylene glycol, 1,3-propanediol, 1,4-
5 butanediol, 1,6-hexanediol, neopentyl glycol, 2,2,4,4-tetramethyl-1,3-cyclobutanediol, and 1,4-cyclohexane dimethanol.

The inherent viscosity of the TLCP is a further consideration in the selection of preferred second polymers. A preferred second polymer is PET-HB
10 having an inherent viscosity of from about 0.3 to about 10.0 dl/g. An even more preferred second polymer is PET-HB having an inherent viscosity of from about 0.5 to about 3.5 dl/g.

Beneficial results are obtained using either pellet blends or melt blends of
15 the TLCP and of the thermoplastic polyesters. The melt blends may be made in any conventional melt blending apparatus such as single or twin screw extruders, Brabender plastographs, and the like. Beneficial results are also obtained when the TLCP is used as one component in a polymeric formulation. In one embodiment, for example, various other polymers are blended with the TLCP prior to blending
20 with the thermoplastic.

The compositions of this invention may optionally comprise one or more additives which lend to the composition desirable qualities such as, for example, flame retardants, glass fibers, carbon fibers, fibrous reinforcing materials, inert
25 fillers, reinforcing agents, pigments, dyes, colorants, stabilizers, antioxidants, plasticizers, epoxides, nucleators, and processing aids. Preferred fillers and reinforcing agents include glass fibers, glass beads, mica, and polymeric fibers, including preformed or in-situ TLCP fibers or fibrils. In a particularly suitable embodiment, the composition comprises from about 20 to about 98.9% by wt. of

the first polymer, from about 0.1 to about 50% by wt. of the second polymer, and from about 1 to about 70 wt. % of the one or more additive components.

Compositions comprising from about 15 to about 40 wt. % of the one or more additive components are more preferred.

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The compositions of the present invention optionally can be used during the manufacture of various products, and can also be incorporated into final products. Thus, the composition can be a raw material that is further processed before a final product is prepared, and the compositions can be present as the final
10 article. The composition can, for example, be incorporated into shaped articles, including extruded shaped articles and molded shaped articles. Particularly suitable products that can be obtained from the present invention include molded parts, film sheeting, and fibers.

15 The compounds and components of the invention may be readily synthesized and prepared using techniques generally known to synthetic organic or polymer chemists. Suitable experimental methods for making and deriving the first and second polymers are described, for example, in the references cited in this document, the disclosures of which being hereby incorporated by this reference for
20 their general teachings and for their synthesis teachings. Methods for making specific and preferred compositions of the present invention are described in detail in examples set forth below.

Experimental

25 The following examples are put forth so as to provide those of ordinary skill in the art with a complete disclosure and description of how the compounds claimed herein are made and evaluated, and are intended to be purely exemplary of the invention and are not intended to limit the scope of what the inventors regard as their invention. Efforts have been made to ensure accuracy with respect to numbers

(e.g., amounts, temperature, etc.) but some errors and deviations should be accounted for. Unless indicated otherwise, parts are parts by weight, temperature is in C or is at room temperature, and pressure is at or near atmospheric.

5

Example 1

- A. 540 grams of PCT pellets (I.V. 0.78 dl/g) were mixed with 60 grams of PET-80HB pellets (PET-HB polymer containing 80 mole % HB). The pellet blend was compounded using a Brabender D6 twin screw extruder, with the first zone set to 280 C and subsequent zones set to 305 C. The blend was processed at a throughput of approximately 1000 grams/hour and at a screw speed of 50 rpm. The system was allowed to equilibrate for 20 minutes, and then samples were collected (sample 1A).
- B. A blend consisting of 570 grams of PCT pellets (from the same lot as 1A) and 24 grams of PET-80HB pellets was processed under identical conditions to 1A (sample 1B).
- C. 600 grams of PCT pellets (also from the same lot as 1A and 1B) were processed under identical conditions to 1A (sample 1C).

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The viscosities of these three samples were measured using a capillary rheometer, at 305 C and 1000 s⁻¹, and the results are given in Table 1.

Approximately 25 grams of sample 1A were then dried overnight in a 100 C oven with a 1 to 10 scf/hour nitrogen sweep. An aliquot of this dried material was then re-melted in a Tinius-Olsen rheometer. The sample was allowed to equilibrate for 5 minutes at 300 C, at which time a sample was collected. Three subsequent samples were collected at 5, 10, and 15 minutes elapsed from the initial sample. During

these hold times, no force was placed on the molten sample.

The above testing was repeated for samples 1B and 1C. The compounded samples and the Tinius-Olsen extrudates were submitted for GPC molecular weight analysis, with the weight-average molecular weights (in PET molecular weight equivalents) given in Table 2. The samples containing PET-80HB show lower molecular weight loss, both as a result of compounding and the Tinius-Olsen melt stability testing.

10

Example 2

A. 540 grams of PET pellets (I.V.= 0.72) were mixed with 24 grams of PET-80HB pellets using conditions similar to Example 1, except that the processing temperatures were 20 C lower.

15

B. 600 grams of PET pellets were processed under identical conditions to Example 2A.

20

C. 540 grams of Bisphenol A polycarbonate (PC) were mixed with 24 grams of PET-80HB, using conditions similar to Example 1.

D. 600 grams of PC pellets were processed under identical conditions to Example 2C.

25

The viscosities of these four samples were measured using a capillary rheometer, at 305 C and 1000 s⁻¹, and the results are given in Table 1. The melt stability of these samples was tested using the same Tinius-Olsen procedure as in Example 1, with results given in Table 2. Sample 2A showed less molecular weight loss during compounding and improved melt stability relative to sample 2B.

Samples 2C and 2D did not differ in molecular weight loss during compounding or in melt stability.

Example 3

5

- A. 360 grams of PCT pellets, 45 grams of PET-80HB pellets, and 180 grams of 1/2" long by 12 micron diameter chopped glass fiber were compounded under conditions identical to Example 1.
- 10 B. 360 grams of PCT pellets, 45 grams of PET-60HB pellets, and 180 grams of 1/2" long by 12 micron diameter chopped glass fiber were compounded under conditions identical to Example 1.
- 15 C. 420 grams of PCT pellets and 180 grams of 1/2" long by 12 micron diameter chopped glass fiber were compounded under conditions identical to Example 1.

The viscosities of these three samples were measured using a capillary rheometer, at

- 20 305 C and 1000 s^{-1} , and the results are given in Table 1. The melt stability of these samples was tested using the same Tinius-Olsen procedure as in Example 1, with results given in Table 2. Samples 3A and 3B showed less molecular weight loss during compounding and improved melt stability relative to sample 3C.

25

Example 4

- A. 540 grams of PCT pellets were compounded, under conditions identical to example 1, with 60 grams of a thermotropic liquid crystalline polymer prepared from 1 part chloroterephthalic acid, 3 parts naphthalene-2,6-

dicarboxylic acid, 40 parts hydroquinone, and 60 parts p-hydroxybenzoic acid.

5 The viscosities of this sample was measured using a capillary rheometer, at 305 C and 1000 s⁻¹, and the results are given in Table 1. The melt stability of the sample was tested using the same Tinius-Olsen procedure as in Example 1, with results given in Table 2. This sample showed improved melt stability, relative to sample 1C.

10

Example 5

A. 540 grams of PCT pellets were mixed with 60 grams of a thermotropic liquid crystalline polymer prepared from 1 part dimethyl *trans*-stilbene-4,4'-dicarboxylate, 0.9 parts butane-1,4-diol, and 0.1 parts ethylene glycol.

15 This pellet blend was processed under conditions identical to Example 1.

The viscosities of this sample was measured using a capillary rheometer, at 305 C and 1000 s⁻¹, and the results are given in Table 1. The melt stability of the sample was tested using the same Tinius-Olsen procedure as in Example 1, with results
20 given in Table 2. This sample did not show molecular weight preservation on compounding in comparison to sample 1C. Neither did the sample show improved melt stability relative to sample 1C.

Table 1Sample viscosities at 305 C, 1000 s⁻¹, in Pa · sec:

<u>Sample #</u>	—
1A	45.11
1B	37.82
1C	25.63
2A	79.41
2B	109.49
2C	228.71
2D	297.12
3A	149.90
3B	137.75
3C	80.72
4A	33.54
5A	22.03

Table 2

Molecular weights (Mw, by GPC, in daltons) after processing:

Sample #	after processing	300 C, 0 minutes	300 C, 5 minutes	300 C, 10 minutes	300 C, 15 minutes
1A	36,130	35,500	33,260	32,020	31,870
1B	35,030	34,810	32,650	30,970	28,490
1C	34,760	33,430	31,530	30,120	26,460
2A	45,400	44,130	38,590	37,500	36,880
2B	44,600	44,010	38,060	36,650	35,400
2C	29,730	28,030	24,030	22,540	19,730
2D	29,760	28,430	24,190	21,730	19,910
3A	39,330	38,890	37,530	36,280	34,990
3B	37,770	37,150	35,920	33,990	32,010
3C	33,480	33,430	31,070	29,920	27,520
4A	34,330	34,250	31,500	29,520	29,180
5A	28,600	28,730	26,440	24,960	24,370

5

Throughout this application, various publications are referenced. The disclosures of these publications in their entireties are hereby incorporated by reference into this application in order to more fully describe the state of the art to which this invention pertains.

10

It will be apparent to those skilled in the art that various modifications and variations can be made in the present invention without departing from the scope or spirit of the invention. Other embodiments of the invention will be apparent to
5 those skilled in the art from consideration of the specification and practice of the invention disclosed herein. It is intended that the specification and examples be considered as exemplary only, with a true scope and spirit of the invention being indicated by the following claims.

What is claimed is:

1. A composition comprising:
 - 5 (a) at least 50 parts by weight of a first polymer, the first polymer being a thermoplastic polyester; and
 - (b) less than or equal to 50 parts by weight of a second polymer that is a TLCP;wherein:
 - 10 (i) the second polymer comprises at least one monomeric aromatic hydroxyl residue;
 - (ii) the first polymer and the second polymer have an overlapping melt processing temperature range;
 - (iii) the first polymer and the second polymer are immiscible or
15 partly immiscible when mixed within the overlapping temperature range; and
 - (iv) the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer
20 in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.
2. The composition of Claim 1 in the form of an inhomogeneous solid phase
blend.
- 25 3. The composition of Claim 1 wherein the difference in solubility parameters of the first polymer and second polymer is greater than 0.3 $(\text{J}/\text{cm}^3)^{0.5}$.

4. The composition of Claim 1 wherein the difference in solubility parameters of the first polymer and second polymer is greater than 0.5 $(\text{J}/\text{cm}^3)^{0.5}$.
- 5 5. The composition of Claim 1 wherein the difference in solubility parameters of the first polymer and second polymer is greater than 1.0 $(\text{J}/\text{cm}^3)^{0.5}$.
6. The composition of Claim 1 wherein the second polymer and first polymer are present in the composition at a weight ratio of second polymer to first polymer of less than 1:10.
- 10
7. The composition of Claim 1 wherein the weight ratio of the second polymer to the first polymer ranges from about 1:14 to about 1:25.
- 15
8. The composition of Claim 1 comprising:
- (a) from about 20 to about 99.5% by wt. of the first polymer; and
- (b) from about 0.1 to about 50% by wt. of the second polymer.
- 20
9. The composition of Claim 8 comprising from about 1 to about 20% by weight of the second polymer.
10. The composition of Claim 9, further comprising from about 1 to about 70 wt. % of one or more additive components comprising a flame retardant, a glass fiber, carbon fiber, an inert filler, a reinforcing agent, a pigment, a dye, a colorant, a stabilizer, an antioxidant, a plasticizer, an epoxide, a nucleator, or a processing aid.
- 25

11. The composition of Claim 1 wherein the overlapping temperature range is 5 C or greater.
12. The composition of Claim 1 wherein the overlapping temperature range is 5
20 C or greater.
13. The composition of Claim 1 wherein the second polymer is a PET-HB copolyester containing from about 50 to about 90 mole percent p-hydroxybenzoic acid residues.
- 10
14. The composition Claim 1 wherein the second polymer is a PET-HB copolyester containing from about 60 to about 85 mole percent p-hydroxybenzoic acid residues.
- 15
15. The composition of Claim 13 wherein the second polymer has an inherent viscosity of from about 0.3 to about 10.0 dl/g.
16. The composition of Claim 13 wherein the inherent viscosity of the second polymer is from about 0.5 to about 3.5 dl/g.
- 20
17. The composition of Claim 13 wherein the PET-HB copolyester is modified by up to 20 mole % with one or more dibasic acids comprising from 2 to about 40 carbon atoms.
- 25
18. The composition of Claim 17 wherein the one or more dibasic acids comprise succinic, glutaric, adipic, sebasic, 1,4-cyclohexanedicarboxylic, isophthalic, naphthalenedicarboxylic, trans-stilbene-4,4'-dicarboxylic, dimer acid, or mixtures thereof.

19. The composition of Claim 13 wherein the PET-HB copolyester is modified with one or more glycols containing from 2 to about 12 carbon atoms.
20. The composition of Claim 19 wherein the one or more glycols comprise
5 propylene glycol, 1,3-propanediol, 1,4-butanediol, 1,6-hexanediol, neopentyl glycol, 2,2,4,4-tetramethyl-1,3-cyclobutanediol, 1,4-cyclohexane dimethanol, or mixtures thereof.
21. The composition of Claim 1 wherein the second polymer is formed from
10 one or more dibasic acids comprising terephthalic acid, naphthalene-2,6-dicarboxylic acid, hydroquinone, p-hydroxybenzoic acid or mixtures thereof.
22. The composition of Claim 1 wherein the first polymer is formed from one
15 or more aliphatic, aromatic, or alicyclic dibasic acids containing from 2 to about 40 carbon atoms, and one or more aliphatic or alicyclic glycols containing from about 2 to about 40 carbon atoms.
23. The composition of Claim 22 wherein the first polymer is modified with
20 from about 0.1 to about 25 mole % of aliphatic dibasic acids containing from 2 to about 40 carbon atoms.

24. The composition of Claim 1 wherein the first polymer comprises poly(ethylene terephthalate); poly(tetramethylene terephthalate); poly(1,4-cyclohexylenedimethylene terephthalate); poly(ethylene 2,6-naphthalenedicarboxylate); poly(ethylene 1,4-cyclohexanedicarboxylate); a
5 poly(ethylene terephthalate) copolyester containing isophthalic acid residues; a poly(ethylene terephthalate) copolyester containing 1,4-cyclohexane dimethanol residues; a poly(ethylene 2,6-naphthalenedicarboxylate) copolyester containing 1,4-cyclohexane dimethanol residues; a poly(ethylene 2,6-naphthalenedicarboxylate)
10 copolyester containing terephthalic acid residues; a poly(ethylene 2,6-naphthalenedicarboxylate) copolyester containing 1,4-cyclohexane dimethanol residues; a poly(ethylene terephthalate) copolyester containing isophthalic acid residues; or mixtures thereof.
- 15 25. The composition of Claim 1 wherein the first polymer comprises poly(ethylene 2,6-naphthalenedicarboxylate) formed from 2,6-, 2,7-, 1,4-, or 1,5-naphthalenedicarboxylic acid.
26. The composition of Claim 1 wherein the first polymer has an inherent
20 viscosity of from about 0.4 to about 2.0 dl/g.
27. The composition of Claim 1 wherein the melt viscosity of the composition, measured at 1000 s^{-1} or higher, is at least 5% higher than the melt viscosity of the first polymer.
25
28. A molded part comprising the composition of Claim 1.
29. A film sheeting comprising the composition of Claim 1.

30. A fiber comprising the composition of Claim 1.
31. A method for lessening the shear or heat induced reduction in molecular weight of a thermoplastic polyester, comprising:
- 5 (a) admixing at least 50 parts by weight of a first polymer with less than or equal to 50 parts by weight of a second polymer, the first polymer being a thermoplastic polyester, and the second polymer being a TLCP, to obtain a polyester/TLCP mixture, and
- (b) subjecting the polyester/TLCP mixture to heat or shear, wherein
- 10 (i) the second polymer comprises at least one monomeric aromatic hydroxyl residue;
- (ii) the first polymer and the second polymer have an overlapping melt processing temperature range;
- (iii) the first polymer and the second polymer are immiscible or
- 15 partly immiscible when mixed within the overlapping temperature range; and
- (iv) the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater
- 20 than the molecular weight of the first polymer alone after shear or heat processing.
32. The method of Claim 31 wherein the polyester/TLCP mixture is in the form of an inhomogeneous solid phase blend.
- 25
33. The method of Claim 31 wherein the overlapping temperature range is at least 5°C.

34. The method of Claim 31 wherein the overlapping temperature range is at least 20°C.
35. The method of Claim 31 wherein the polyester/TLCP mixture comprises:
5
(a) from about 20 to about 99.5% by wt. of the first polymer; and
(b) from about 0.1 to about 50% by wt. of the second polymer.
36. The method of Claim 35 wherein the polyester/TLCP mixture comprises
10 from about 1 to about 20% by weight of the second polymer.
37. The method of Claim 35 wherein the weight ratio of the second polymer to the first polymer in the polyester/TLCP mixture ranges from about 0.1 to about 1.0.
15
38. The method of Claim 31, further comprising admixing one or more additive components comprising a flame retardant, a glass fiber, an inert filler, a reinforcing agent, a pigment, a dye, a colorant, a stabilizer, an antioxidant, a plasticizer, an epoxide, a nucleator, or a processing aid.
20
39. The method of Claim 38 wherein the polyester/TLCP mixture comprises:
(a) from about 20 to about 98.9% by wt. of the first polymer;
(b) from about 0.1 to about 50% by wt. of the second polymer; and
(c) from about 1 to about 70 wt. % of the one or more additive
25 components.
40. The method of Claim 39 wherein the polyester/TLCP mixture comprises from about 1 to about 20% by wt. of the second polymer.

41. The method of Claim 39 wherein, in the polyester/TLCP mixture, the weight ratio of the second polymer to the first polymer is from about 0.1 to about 1.0.
- 5 42. The method of Claim 31 wherein the melt viscosity of the polyester/TLCP mixture, measured at 1000 s^{-1} or higher, is at least 5% higher than the melt viscosity of the first polymer.
- 10 43. The method of Claim 31 wherein the second polymer is a PET-HB copolyester containing from about 50 to about 90 mole percent p-hydroxybenzoic acid residues.
44. The method of Claim 31 wherein the second polymer contains about 60 to about 85 mole percent p-hydroxybenzoic acid residues.
- 15 45. The method of Claim 43 wherein the second polymer has an inherent viscosity of from about 0.3 to about 10.0 dl/g.
- 20 46. The method of Claim 43 wherein the inherent viscosity of the second polymer is from about 0.5 to about 3.5 dl/g.
47. The method of Claim 43 wherein the PET-HB copolyester is modified by up to 20 mole % with one or more dibasic acids comprising from 2 to 40 carbon atoms.
- 25 48. The method of Claim 47 wherein the one or more dibasic acids comprise succinic, glutaric, adipic, sebacic, 1,4-cyclohexanedicarboxylic, isophthalic, naphthalenedicarboxylic, trans-stilbene-4,4'-dicarboxylic, or dimer acid.

49. The method of Claim 48 wherein the PET-HB copolyester is modified with one or more glycols containing from 2 to about 12 carbon atoms.
50. The method of Claim 49 wherein the PET-HB copolyester is modified with
5 one or more glycols comprising propylene glycol, 1,3-propanediol, 1,4-butanediol, 1,6-hexanediol, neopentyl glycol, 2,2,4,4-tetramethyl-1,3-cyclobutanediol, or 1,4-cyclohexane dimethanol.
51. The method of Claim 31 wherein the second polymer is formed from one or
10 more dibasic acids comprising terephthalic acid, naphthalene-2,6-dicarboxylic acid, hydroquinone, or p-hydroxybenzoic acid.
52. The method of Claim 31 wherein the first polymer is formed from one or
15 more aliphatic, aromatic, or alicyclic dibasic acids containing from about 2 to about 40 carbon atoms, and one or more aliphatic or alicyclic glycols containing from about 2 to about 40 carbon atoms.

53. The method of Claim 31 wherein the first polymer comprises poly(ethylene terephthalate); poly(tetramethylene terephthalate); poly(1,4-cyclohexylenedimethylene terephthalate); poly(ethylene 2,6-naphthalenedicarboxylate); poly(ethylene 1,4-cyclohexanedicarboxylate); a
5 poly(ethylene terephthalate) copolyester containing isophthalic acid residues; a poly(ethylene terephthalate) copolyester containing 1,4-cyclohexane dimethanol residues; a poly(ethylene 2,6-naphthalenedicarboxylate) copolyester containing 1,4-cyclohexane dimethanol residues; a poly(ethylene 2,6-naphthalenedicarboxylate) copolyester containing terephthalic acid
10 residues; a poly(ethylene 2,6-naphthalenedicarboxylate) copolyester containing 1,4-cyclohexane dimethanol residues; a poly(ethylene terephthalate) copolyester containing isophthalic acid residues; or mixtures thereof.
- 15 54. The method of Claim 52 wherein the first polymer is modified with from about 0.1 to about 25 mole % of aliphatic dibasic acids containing from 2 to about 40 carbon atoms.
- 20 55. The method of Claim 31 wherein the first polymer comprises poly(ethylene 2,6-naphthalenedicarboxylate) formed from 2,6-, 2,7-, 1,4-, or 1,5-naphthalenedicarboxylic acid.
- 25 56. The method of Claim 50 wherein the first polymer has an inherent viscosity of from about 0.4 to about 2.0 dl/g.
57. The product produced by the method of Claim 31.
58. The product of Claim 57 in the form of a molded part.

59. The product of Claim 57 in the form of a film sheeting.
60. The product of Claim 57 in the form of a fiber.
- 5 61. A method for lessening the shear or heat induced reduction in molecular weight of a thermoplastic polyester, comprising:
- 10 (a) admixing at least 50 parts by weight of a first polymer with less than or equal to 50 parts by weight of a second polymer, the first polymer being a thermoplastic polyester, and the second polymer being a TLCP, to obtain a polyester/TLCP mixture, and
- 15 (b) subjecting the polyester/TLCP mixture to at least 1500 s⁻¹ units of shear, wherein
- (i) the second polymer comprises at least one monomeric aromatic hydroxyl residue;
- (ii) the first polymer and the second polymer have an overlapping melt processing temperature range; and
- 20 (iii) the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.
62. The process of Claim 61 wherein the composition is subjected to over 2500 s⁻¹ units of shear.
- 25 63. A composition comprising:
- (a) at least 50 parts by weight of a first polymer, the first polymer being a thermoplastic polyester; and

- (b) less than or equal to 50 parts by weight of a second polymer that is a TLCP;

wherein:

- 5
- (i) the second polymer comprises at least one monomeric aromatic hydroxyl residue;
 - (ii) the first polymer and the second polymer have an overlapping melt processing temperature range;
 - (iii) the first polymer and the second polymer are present in a weight ratio of greater than 10:1; and
 - 10 (iv) the first polymer and the second polymer are provided in such quantities that the molecular weight of the first polymer in the composition after shear or heat processing is greater than the molecular weight of the first polymer alone after shear or heat processing.
- 15

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/20284

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C08L67/00 C08J3/00 C08J5/18 D01F6/92

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C08L C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>WO 94 20552 A (AKZO NV) 15 September 1994</p> <p>see claims 1-7; examples 2-5 see page 1, line 3 - page 2, line 2</p> <p style="text-align: center;">--- -/--</p>	<p>1-9, 11, 12, 21, 22, 24, 26, 30-37, 43-45, 51-53, 56, 57, 60-63</p>

Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

Special categories of cited documents :

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/20284

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	<p>EP 0 030 417 A (ICI LTD.) 17 June 1981</p> <p>see claims 1-10; examples 2,10,16 see page 1, line 3 - page 9, line 4 & US 4 433 083 A cited in the application & US 4 386 174 A cited in the application</p> <p style="text-align: center;">---</p>	<p>1-15,21, 22,24, 26,28, 31-41, 43-45, 51-53, 56-58, 61-63</p>
X	<p>F.P. LA MANTIA: "Thermotropic liquid crystal polymer blends" 1993, TECHNOMIC PUBL. CO., LANCASTER, PENNSYLVANIA, USA XP002092727 cited in the application</p> <p>see page 113 - page 120 see page 169 see page 173 - page 174</p> <p style="text-align: center;">---</p>	<p>1-9, 11-14, 21,22, 24, 28-37, 43,44, 51-53, 57-63</p>
X	<p>DATABASE WPI Section Ch, Week 8727 Derwent Publications Ltd., London, GB; Class A23, AN 87-189291 XP002092728 & JP 62 119265 A (DIAFOIL CO LTD) , 30 May 1987 see abstract</p> <p style="text-align: center;">---</p>	<p>1-14, 21-26, 29, 31-41, 43,44, 51-57, 59,61-63</p>
X	<p>DATABASE WPI Section Ch, Week 9303 Derwent Publications Ltd., London, GB; Class A23, AN 93-021891 XP002092729 & JP 04 348154 A (TORAY IND INC) , 3 December 1992 see abstract</p> <p style="text-align: center;">---</p>	<p>1-12,21, 22,24, 26,28, 31-41, 51-53, 56-58, 61-63</p>
X	<p>EP 0 071 968 A (ALLIED CORP) 16 February 1983</p> <p>see claims 1-5; examples 18,31</p> <p style="text-align: center;">-----</p>	<p>1-5,8,9, 11,12, 21,22, 24, 30-37, 51-53, 57,60-62</p>

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 98/20284

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