A process for making a nonwoven fabric is provided comprising: (A) collecting multicomponent fibers to form a nonwoven web; wherein the multicomponent fiber comprises at least one water dispersible sulfopolyester and at least one water non-dispersible polymer; wherein said multicomponent fiber has a plurality of domains comprising the water non-dispersible polymer; wherein the domains are substantially isolated from each other by the water dispersible sulfopolyester intervening between the domains; (B) contacting the non-woven web with water at a sufficient temperature and pressure to remove a portion of the water dispersible sulfopolyester thereby forming a microfiber web; and (C) hydroentangling the microfiber web to produce the nonwoven fabric. A process is also provided wherein steps (B) and (C) are combined. Fibrous articles utilizing the nonwoven fabrics are also provided.
Fig. 1

MELT PUMPS

SPIN PACK

ASPIRATOR ASSEMBLY (DRAW)

PRIMARY EXTRUDER

SECONDARY EXTRUDER

CONSOLIDATION ROLLS

FORMING BELT

WIND-UP ROLL
NONWOVENS FABRICS PRODUCED FROM MULTICOMPONENT FIBERS COMPRISING SULFOPOLYESTERS

FIELD OF THE INVENTION

[0001] The present invention pertains to processes to produce nonwoven fabrics from multicomponent fibers wherein the multicomponent fibers comprise at least one water-dispersible sulfopolyester and at least one water non-dispersible polymer.

BACKGROUND OF THE INVENTION

[0002] Traditional textile fabrics are made by weaving or knitting. Nonwoven fabrics are similar to woven and knitted fabrics in that both are planar, inherently flexible, porous structures composed of polymer-based materials. The main difference between the two is the process in which the fabric is made. Woven fabrics are made by interlacing two or more sets of yarns at right angles to one another in a designated order. Nonwoven fabrics are made by placing a predetermined number of fibers or filaments into a two-dimensional array and locking them together. The nonwoven fabric can be assembled by mechanically, chemically, or thermally interlocking layers or networks of fibers, filaments or yarns. Typically, processes for producing nonwoven fabrics can be grouped into four general classifications: textile, paper, extrusion, and hybrid, meaning a combination of these technologies are utilized. These fabrics are technically sophisticated engineered structures that can be made to resemble woven fabrics and can exceed the properties of woven fabrics.

[0003] Extrusion technology is used to produce spunbond, meltblown, and porous-film nonwovens. These types of nonwovens are sometimes called polymer-laid nonwovens. Spunbond and meltblown processes are an advancement in the production of nonwoven fabrics since the nonwovens are made directly from the materials used to make the fibers themselves, thus eliminating the fiber production step. The spunbond process transforms polymer directly to fabric by extruding filaments, orienting them as bundles or groupings, layering them on a conveying screen, and interlocking them by thermal fusion, mechanical entanglement, chemical adhesives, or combinations of these methods. In the meltblown process, the polymer is heated to the liquid state and as it passes through an extrusion orifice, it is injected with sonic velocity air at about 250 to 500°C. The fast moving air stream stretches the molten polymer and solidifies it to produce fine fibers. The fibers are then separated from the air stream as an entangled web and compressed between heated rolls.

[0004] Nonwovens are utilized in textile applications such as, for example, clothing, curtains, upholstery, and uniforms. Nonwoven fabrics can also be utilized in personal care products, such as, but not limited to, wipes, feminine hygiene products, baby diapers, adult incontinence briefs, and hospital/surgical and other medical disposable. Other applications include, but are not limited to, protective fabrics and layers, geotextiles, industrial wipes, and filter media.

[0005] Accordingly, there is a need for new processes for producing nonwoven fabrics and fibrous articles prepared therefrom that are more efficient and/or less costly. This invention provides novel processes to produce nonwoven fabrics from multicomponent fibers comprising at least one water dispersible sulfopolyester and at least one water non-dispersible polymers.

SUMMARY OF THE INVENTION

[0006] In one embodiment of the invention, a process for making a nonwoven fabric is provided. The process comprises: (A) collecting multicomponent fibers to form a nonwoven web; wherein the multicomponent fiber comprises at least one water dispersible sulfopolyester and at least one water non-dispersible polymer; wherein the multicomponent fiber has a plurality of domains comprising the water non-dispersible polymer; wherein the domains are substantially isolated from each other by the water dispersible sulfopolyester intervening between the domains; (B) contacting the non-woven web with water at a sufficient temperature and pressure to remove a portion of the water dispersible sulfopolyester thereby forming a microfiber web; and (C) hydroentangling the microfiber web to produce the nonwoven fabric.

[0007] In another embodiment of the present invention, another process for making a nonwoven fabric is provided. The process comprises: (A) collecting the multicomponent fibers to form a non-woven web; wherein the multicomponent fiber comprises at least one water dispersible sulfopolyester and at least one water non-dispersible polymer; wherein the multicomponent fiber has a plurality of domains comprising the water non-dispersible polymer; wherein the domains are substantially isolated from each other by the water dispersible sulfopolyester intervening between the domains; and (B) contacting the non-woven web with water at a sufficient temperature and pressure to remove a portion of the water dispersible sulfopolyester thereby forming microfibers and simultaneously hydroentangling the microfibers to produce the nonwoven fabric.

[0008] Our invention thus offers a novel and inexpensive process to produce nonwoven fabric. The nonwoven fabric may be in the form of a flat fabric or a 3-dimensional shape and may be incorporated into a variety of fibrous articles such as those discussed previously.

BRIEF DESCRIPTION OF FIGURES

[0009] FIG. 1 illustrates the sponbond process.

[0010] FIG. 2 illustrates how the examples relate to one another.

DETAILED DESCRIPTION

[0011] The present invention may be understood more readily by reference to the following detailed description of preferred embodiments of the invention and the Examples included therein and to the Figures and their previous and following description.

[0012] Before the present compounds, compositions, articles, devices, and/or methods are disclosed and described, it is to be understood that this invention is not limited to specific syntactic methods, specific processes, or to particular apparatuses, 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.

[0013] 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.
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 a polymer includes one or more polymers.

Ranges may be expressed herein as from “about” one particular value, and/or to “about” another particular value. When such a range is expressed, another embodiment includes 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 forms another embodiment. It will be further understood that the endpoints of each of the ranges are significant both in relation to the other endpoint, and independently of the other endpoint. Further, the ranges stated in this disclosure and the claims are intended to include the entire range specifically and not just the endpoints.

“Optional” or “optionally” means that the subsequently described event or circumstance may or may not occur, and that the description includes instances where said event or circumstance occurs and instances where it does not. For example, the phrase “optionally heated” means that the material may or may not be heated, and that such phrase includes both heated and unheated processes.

Unless otherwise indicated, all numbers expressing quantities of ingredients, properties such as molecular weight, reaction conditions, and so forth used in the specification and claims are to be understood as being modified in all instances by the term “about.” Accordingly, unless indicated to the contrary, the numerical parameters set forth in the following specification and attached claims are approximations that may vary depending upon the desired properties sought to be obtained by the present invention. At the very least, each numerical parameter should at least be construed in light of the number of reported significant digits and by applying ordinary rounding techniques. Notwithstanding that the numerical ranges and parameters set forth the broad scope of the invention are approximations, the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical value, however, inherently contain certain errors necessarily resulting from the standard deviation found in their respective testing measurements.

As used herein, the term “nonwoven fabric” or “nonwoven web” means a structure of individual fibers or threads which are interlaid, but not in an identifiable manner as in a knitted fabric. The basis weight of nonwoven fabrics is usually expressed in ounces of material per square yard (osy) or grams per square meter (gsm).

The term “fiber” as used herein refers to an elongated extrudate formed by passing a polymer through a forming orifice such as a die. Unless noted as otherwise the term “fibers” include discontinuous strands having a definite length and continuous strands of material, such as filaments. The nonwoven fabric of the present invention may be formed from staple multicomponent fibers. Such staple fibers may be carded and bonded to form the nonwoven fabric. Desirably, however, the nonwoven fabric of the present invention is made with continuous multicomponent filaments which are extruded, drawn, and laid on a traveling forming surface.

As used herein, the term “microfibers” means small diameter fibers having an average diameter not greater than about 12 microns, for example, having an average diameter of from about 3 microns to about 8 microns. Fibers are also commonly discussed in terms of denier. A lower denier indicates a finer fiber and a higher denier indicates a thicker or heavier fiber. For example, a 15 micron polypropylene fiber has a denier of about 1.42 (15 x 0.089 x 0.00707 = 1.415). The microfibers utilized to produce the nonwoven fabric of this invention typically have d/f values of 1 or less, 0.5 or less, or 0.1 or less.

As used herein the term “multicomponent fibers” or “conjugate fibers” refers to fibers which have been formed from at least two polymer components. Such fibers are usually extruded from separate extruders but spun together to form one fiber. The polymers of the respective components are usually different from each other although multicomponent fibers may comprise separate components of similar or identical polymeric materials. The individual components are typically arranged in substantially constantly positioned distinct segments or zones across the cross-section of the multicomponent fiber and extend substantially along the entire length of the multicomponent fiber. The configuration of such multicomponent fibers may be, for example, a side-by-side arrangement, a side-by-side arrangement, a core/sheath arrangement, a pelt arrangement or other arrangement. Multicomponent fibers and methods of making the same are taught in U.S. Pat. No. 5,108,820 to Kaneko et al.; U.S. Pat. No. 4,795,668 to Krueger et al.; U.S. Pat. No. 5,382,400 to Pike et al.; U.S. Pat. No. 5,336,552 to Strack et al.; U.S. patent application Ser. No. 08/550,042 filed Oct. 30, 1996 to Cook; and U.S. patent application Ser. No. 11/344,320 filed Jan. 31, 2006 to Gupta et al. The fibers and individual components comprising the same may also have various irregular shapes such as those described in U.S. Pat. No. 5,277,976 to Hogle et al.; U.S. Pat. Nos. 5,162,074 and 5,466,410 to Hills; and U.S. Pat. Nos. 5,069,970 and 5,057,368 to Langman et al. The entire contents of the aforesaid patents and applications are incorporated herein by reference to the extent they do not contradict the statements herein.

As used herein, the term “polymer” generally includes, but is not limited to, homopolymers, copolymers, such as for example, block, graft, random and alternating copolymers, terpolymers, etc., and blends and modifications thereof. Furthermore, unless otherwise specifically limited, the term “polymer” shall include all possible geometric configurations of the molecules. These configurations include, but are not limited to, isotactic, syndiotactic and random symmetries.

The term “water-dispersible”, as used herein in reference to the water dispersible sulfopolyester as one component of a multicomponent fiber, is intended to be synonymous with the terms “water-dissipatable”, “water-disintegratable”, “water-dissoluble”, “water-dispensable”, “water soluble”, “water-removable”, “hydrosoluble”, and “hydrodispersible” and is intended to mean that the sulfopolyester component is sufficiently removed from the multicomponent fiber and is dispersed or dissolved by the action of water to enable the release and separation of the water non-dispersible polymer fibers contained therein. The terms “dispers-
ible”, “dissipate”, or “dissipatable” mean that, using a sufficient amount of deionized water (e.g., 100:1 water:fiber by weight) to form a loose suspension or slurry of the fibers, at a temperature of about 60°C, and within a time period of up to 5 days, the sulfopolyester component dissolves, disintegrates, or separates from the multicomponent fiber, leaving behind a plurality of microfibers from the water non-dispersible polymer segments. In the context of this invention, all of these terms refer to the activity of water or a mixture of water and a water-miscible cosolvent on the water dispersible sulfopolyesters described herein. Examples of such water-miscible cosolvents include, but are not limited to, alcohols, ketones, glycol ethers, esters and the like. It is intended for this terminology to include conditions where the water dispersible sulfopolyester is dissolved to form a true solution as well as those where the water dispersible sulfopolyester is dispersed within the aqueous medium. Often, due to the statistical nature of sulfopolyester compositions, it is possible to have a soluble fraction and a dispersed fraction when a single sulfopolyester sample is placed in an aqueous medium.

[0025] The term “segment” or “domain” or “zone” when used to describe the shaped cross section of a multicomponent fiber refers to the area within the cross section comprising the water non-dispersible polymers where these domains or segments are substantially isolated from each other by the water-dispersible sulfopolyester intervening between the segments or domains. The term “substantially isolated”, as used herein, is intended to mean that the segments or domains are set apart from each other to permit the segments domains to form individual fibers upon removal of the sulfopolyester. Segments or domains or zones can be of similar size and shape or varying size and shape. Again, segments or domains or zones can be arranged in any configuration. These segments or domains or zones are “substantially continuous” along the length of the multicomponent extrudate or fiber. The term “substantially continuous” means continuous along at least a 10 cm length of the multicomponent fiber.

[0026] The present invention provides processes to produce nonwoven fabrics from multicomponent fibers wherein the multicomponent fibers comprise at least one water-dispersible sulfopolyester and at least one water non-dispersible polymer.

[0027] In one embodiment, a process is provided comprising: (A) collecting multicomponent fibers to form a non-woven web, wherein the multicomponent fiber comprises at least one water dispersible sulfopolyester and at least one water non-dispersible polymer; wherein the multicomponent fiber has a plurality of domains comprising the water non-dispersible polymer, wherein the domains are substantially isolated from each other by the water dispersible sulfopolyester intervening between the domains; (B) contacting the non-woven web with water at a sufficient temperature and pressure to remove a portion of the water dispersible sulfopolyester thereby forming a microfiber web; and (C) hydroentangling the microfiber web to produce the nonwoven fabric.

[0028] The multicomponent fibers of our invention can be prepared by melt spinning a single sulfopolyester or sulfopolyester blend with the water non-dispersible polymer immiscible with the sulfopolyester. For example, in U.S. Pat. No. 5,916,678, a multicomponent fiber is prepared by extruding the sulfopolyester and one or more water non-dispersible polymers, which are immiscible with the sulfopolyester, separately through a spinneret having a shaped or engineered transverse geometry such as, for example, an “islands-in-the-sea”, sheath-core, side-by-side, or segmented pie configuration. The water dispersible sulfopolyester is later removed by dissolving the interfacial layers or pie segments and leaving the smaller filaments or microfibers of the water non-dispersible polymer(s). These fibers of the water non-dispersible polymer have fiber size much smaller than the multicomponent fiber before removing the water dispersible sulfopolyester.

[0029] In one embodiment, the multicomponent fiber is produced by feeding the sulfopolyester and water non-dispersible polymers to a polymer distribution system where the polymers are introduced into a segmented spinneret plate. The polymers follow separate paths to the fiber spinneret and are combined at the spinneret hole which comprises either two concentric circular holes or two parts to provide a sheath-core type fiber, or a circular spinneret hole divided along a diameter into multiple parts to provide a fiber having a side-by-side type. Alternatively, the immiscible water dispersible sulfopolyester and water non-dispersible polymers may be introduced separately into a spinneret having a plurality of radial channels to produce a multicomponent fiber having a segmented pie cross section. Typically, the sulfopolyester will form the “sheath” component of a sheath core configuration. In fiber cross sections having a plurality of segments, the water non-dispersible segments, typically, are substantially isolated from each other by the sulfopolyester. Alternatively, multicomponent fibers may be formed by melting the sulfopolyester and water non-dispersible polymers in separate extruders and directing the polymer flows into one spinneret with a plurality of distribution flow paths in form of small thin tubes or segments to provide a multicomponent fiber having an islands-in-the-sea shaped cross section. An example of such a spinneret is described in U.S. Pat. No. 5,366,804, herein incorporated by reference to the extent it does not contradict the statements herein. In the present invention, typically, the sulfopolyester will form the “sea” component and the water non-dispersible polymer will form the “islands” component.

[0030] As stated within this disclosure, the shaped cross section of a multicomponent fiber can, for example, be in the form of a sheath core, islands-in-the-sea, segmented pie, hollow segmented pie, off-centered segmented pie, etc.

[0031] The multicomponent fiber of the present invention is prepared from polymers or, more specifically sulfopolymers, comprising dicarboxylic acid monomer residues, sulfononimer residues, diol monomer residues, and repeating units. The sulfonomer may be a dicarboxylic acid, a diol, or hydroxycarboxylic acid. Thus, the term “monomer residue”, as used herein, means a residue of a dicarboxylic acid, a diol, or a hydroxycarboxylic acid. A “repeating unit”, as used herein, means an organic structure having 2 monomer residues bonded through a carboxyloxy group. The sulfopolymers of the present invention contain substantially equal molar proportions of acid residues (100 mole %) and diol residues (100 mole %) which react in substantially equal proportions such that the total moles of repeating units is equal to 100 mole %. The mole percentages provided in the present disclosure, therefore, may be based on the total moles of acid residues, the total moles of diol residues, or the total moles of repeating units. For example, a sulfopolyester containing 30 mole % of a sulfonomer, which may be a dicarboxylic acid, a diol, or hydroxycarboxylic acid, based on the total repeating units, means that the sulfopolyester contains
30 mole % sulfonmonomer out of a total of 100 mole % repeating units. Thus, there are 30 moles of sulfonmonomer residues among every 100 moles of repeating units. Similarly, a sulfopolyester containing 30 mole % of a dicarboxylic acid sulfonmonomer, based on the total acid residues, means the sulfopolyester contains 30 mole % sulfonmonomer out of a total of 100 mole % acid residues. Thus, in this latter case, there are 30 moles of sulfonmonomer residues among every 100 moles of acid residues.

[0032] The sulfopolyesters described herein have an inherent viscosity, abbreviated hereinafter as “Ih.V.”, of at least about 0.1 dl/g measured in a 60/40 parts by weight solution of phenol/tetrachloroethene solvent at 25° C, and at a concentration of about 0.5 g of sulfopolyester in 100 mL of solvent. The inherent viscosity of the sulfopolyester can also range from about 0.2 to about 0.3 dl/g. Another range for inherent viscosity is greater than about 0.3 dl/g. The term “polyester,” as used herein, encompasses both “homopolymers” and “copolyesters” and means a synthetic polymer prepared by the polycondensation of difunctional carboxylic acids with difunctional hydroxyl compound. As used herein, the term “sulfopolyester” means any polyester comprising a sulfonmonomer.

[0033] Typically the difunctional carboxylic acid is a dicarboxylic acid and the difunctional hydroxyl compound is a dihydric alcohol such as, for example glycols and diols. Alternatively, the difunctional carboxylic acid may be a hydroxy carboxylic acid such as, for example, p-hydroxybenzoic acid, and the difunctional hydroxyl compound may be an aromatic nucleus bearing 2 hydroxy substituents such as, for example, hydroquinone. The term “residue”, as used herein, means any organic structure incorporated into the polymer through a polycondensation reaction involving the corresponding monomer. Thus, the dicarboxylic acid residue may be derived from a dicarboxylic acid monomer or its associated acid halides, esters, salts, anhydrides, or mixtures thereof. As used herein, therefore, the term dicarboxylic acid is intended to include dicarboxylic acids and any derivative of a dicarboxylic acid, including its associated acid halides, esters, halfesters, salts, half-salts, anhydrides, mixed anhydrides, or mixtures thereof, useful in a polycondensation process with a diol to make a high molecular weight polyester.

[0034] The sulfopolyester of the present invention includes one or more dicarboxylic acid residues. Depending on the type and concentration of the sulfonmonomer, the dicarboxylic acid residue may comprise from about 60 to about 100 mole % of the acid residues. Other examples of concentration ranges of dicarboxylic acid residues are from about 60 mole % to about 95 mole %, and about 70 mole % to about 95 mole %. Examples of dicarboxylic acids that may be used include aliphatic dicarboxylic acids, alicyclic dicarboxylic acids, aromatic dicarboxylic acids, or mixtures of two or more of these acids. Thus, suitable dicarboxylic acids include, but are not limited to, succinic; glutaric; adipic; azelaic; sebacic; fumaric; maleic; itaconic; 1,3-cyclohexanedicarboxylic; 1,4-cyclohexanedicarboxylic; diglycolic; 2,5-norbornanedicarboxylic; phthalic; terephthalic; 1,4-naphthalenedicarboxylic; 2,5-naphthalenedicarboxylic; diphenic; 4,4'-oxydibenzoo; 4,4'-sulfonyldibenzoic; and isophthalic. The preferred dicarboxylic acid residues are isophthalic, terephthalic, and 1,4-cyclohexanedicarboxylic acids, or if diesters are used, dimethyl terephthalate, dimethyl isophthalate, and dimethyl-1,4-cyclohexanedicarboxylate. Although the dicarboxylic acid methyl ester can be utilized, it is also acceptable to include higher order alkyl esters, such as ethyl, propyl, isopropyl, butyl, and so forth. In addition, aromatic esters, particularly phenyl, also may be employed.

[0035] The sulfopolyester includes about 4 to about 40 mole %, based on the total repeating units, of residues of at least one sulfonmonomer having 2 functional groups and one or more sulfonate groups attached to an aromatic or cycloaliphatic ring wherein the functional groups are hydroxy, carboxyl, or a combination thereof. Additional examples of concentration ranges for the sulfonmonomer residues are about 4 to about 35 mole %, about 8 to about 30 mole %, and about 8 to about 25 mole %, based on the total repeating units. The sulfonmonomer may be a dicarboxylic acid or ester thereof containing a sulfonate group, a diol containing a sulfonate group, or a hydroxy acid containing a sulfonate group. The term “sulfonate” refers to a salt of a sulfonic acid having the structure “—SO₃M⁺” wherein M is the cation of the sulfonate salt. The cation of the sulfonate salt may be a metal ion such as Li⁺, Na⁺, K⁺, Mg²⁺, Ca²⁺, Ni²⁺, Fe²⁺, and the like. Alternatively, the cation of the sulfonate salt may be non-metallic such as a nitrogenous base as described, for example, in U.S. Pat. No. 4,304,901. Nitrogen-based cations are derived from nitrogen-containing bases, which may be aliphatic, cycloaliphatic, or aromatic compounds. Examples of such nitrogen containing bases include ammonia, dimethylethanolamine, diethanolamine, triethanolamine, pyridine, morpholine, and piperidine. Because monomers containing the nitrogen-based sulfonate salts typically are not thermally stable at conditions required to make the polymers in the melt, the method of this invention for preparing sulfopolyesters containing nitrogen-based sulfonate salt groups is to disperse, dissipate, or dissolve the polymer containing the required amount of sulfonate group in the form of its alkali metal salt in water and then exchange the alkali metal cation for a nitrogen-based cation.

[0036] When a monovalent alkali metal ion is used as the cation of the sulfonate salt, the resulting sulfopolyester is completely dispersible in water with the rate of dispersion dependent on the content of sulfonmonomer in the polymer, temperature of the water, surface area/thickness of the sulfopolyester, and so forth. When a divalent metal ion is used, the resulting sulfopolyesters are not readily dispersed by cold water but are more easily dispersed by hot water. Utilization of more than one counterion within a single polymer composition is possible and may offer a means to tailor or fine-tune the water-responsivity of the resulting article of manufacture. Examples of sulfonmonomers residues include monomer residues where the sulfonate salt group is attached to an aromatic acid nucleus, such as, for example, benzene; naphthalene; diphenyl; oxydiphenyl; sulfonyldiphenyl; and methylene-diphenyl or cyclaliphatic rings, such as, for example, cyclohexyl; cyclopentyl; cyclobutyl; cycloheptyl; and cyclooctyl. Other examples of sulfonmonomers residues which may be used in the present invention are the metal sulfonate salt of sulfophthalic acid, sulfitoterephthalic acid, sulfosulfophthalic acid, or combinations thereof. Other examples of sulfonmonomers which may be used are 5-sodiosulfophthalic acid and esters thereof. If the sulfonmonomer residue is from 5-sodiosulfophthalic acid, typical sulfonmonomer concentration ranges are about 4 to about 35 mole %, about 8 to about 30 mole %, and about 8 to 25 mole %, based on the total moles of acid residues.

[0037] The sulfonmonomers used in the preparation of the sulfopolyesters are known compounds and may be prepared...
using methods well known in the art. For example, sulfonylamides in which the sulfonate group is attached to an aromatic ring may be prepared by sulfonylating the aromatic compound with oleum to obtain the corresponding sulfonic acid and followed by reaction with a metal oxide or base, for example, sodium acetate, to prepare the sulfonate salt. Procedures for preparation of various sulfonylamides are described, for example, in U.S. Pat. Nos. 3,779,993; 3,018,272; and 3,528,947.

[0038] It is also possible to prepare the polyester using, for example, a sodium sulfonate salt, and ion-exchange methods to replace the sodium with a different ion, such as zinc, when the polymer is in the dispersed form. This type of ion exchange procedure is generally superior to preparing the polymer with divalent salts since the sodium salts are usually more soluble in the polymer reactant melt-phase.

[0039] The sulfopolyester includes one or more diol residues which may include aliphatic, cycloaliphatic, and aralkyl glycols. The cycloaliphatic diols, for example, 1,3- and 1,4-cyclohexanediol, may be present as their pure cis or trans isomers or as a mixture of cis and trans isomers. As used herein, the term “diol” is synonymous with the term “glycol” and means any dihydric alcohol. Examples of diols include, but are not limited to, ethylene glycol; diethylene glycol; triethylene glycol; polyethylene glycols; 1,3-propanediol; 2,4-dimethyl-1,3-dioxol; 2,2-dimethyl-1,3-propanediol; 2-ethyl-2-butyl-1,3-propanediol; 2-ethyl-2-isobutyl-1,3-propanediol; 1,3-butandiol; 1,4-butandiol; 1,5-pentanediol; 1,6-hexanediol; 2,2,4-trimethyl-1,6-hexanediol; thioglycol; 1,2-cyclohexanediol; 1,3-cyclohexanediol; 1,4-cyclohexanediol; 1,4-cyclohexanediol; 2,2,4,4-tetramethyl-1,3-cyclobutandiol; p-xylene glycol, or combinations of one or more of these glycols.

[0040] The diol residues may include from about 25 mole % to about 100 mole %, based on the total diol residues, of residue of a poly(ethylene glycol) having a structure

\[
\text{H} \rightarrow (\text{OCH}_2\text{-CH}_2)_n \rightarrow \text{OH}
\]

wherein \( n \) is an integer in the range of 2 to about 500. Non-limiting examples of lower molecular weight polyethylene glycols, e.g., wherein \( n \) is from 2 to 6, are diethylene glycol, triethylene glycol, and tetraethylene glycol. Of these lower molecular weight glycols, diethylene and triethylene glycol are preferred. Higher molecular weight polyethylene glycols (abbreviated herein as “PEG”), wherein \( n \) is from 7 to about 500, include the commercially available products known under the designation CARBOWAX®, a product of Dow Chemical Company (formerly Union Carbide). Typically, PEGs are used in combination with other diols such as, for example, diethylene glycol or ethylene glycol. Based on the values of \( n \), which range from greater than 6 to 500, the molecular weight may range from greater than 300 to about 22,000 g/mol. The molecular weight and the mole % are inversely proportional to each other; specifically, as the molecular weight is increased, the mole % will be decreased in order to achieve a designated degree of hydrophilicity. For example, it is illustrative of this concept to consider that a PEG having a molecular weight of 1000 may constitute up to 10 mole % of the total diol, while a PEG having a molecular weight of 10,000 would typically be incorporated at a level of less than 1 mole % of the total diol.

[0041] Certain dimer, trimer, and tetramer diols may be formed in situ due to side reactions that may be controlled by varying the process conditions. For example, varying amounts of diethylene, triethylene, and tetraethylene glycols may be formed from ethylene glycol from an acid-catalyzed dehydration reaction which occurs readily when the polycondensation reaction is carried out under acidic conditions. The presence of buffer solutions, well-known to those skilled in the art, may be added to the reaction mixture to retard these side reactions. Additional compositional latitude is possible, however, if the buffer is omitted and the dimerization, trimerization, and tetramerization reactions are allowed to proceed.

[0042] The sulfopolyester of the present invention may include from 0 to about 25 mole %, based on the total repeating units, of residues of a branching monomer having 5 or more functional groups wherein the functional groups are hydroxy, carboxyl, or a combination thereof. Non-limiting examples of branching monomers are 1,1,1-trimethylpropane, 1,1,1-trimethylolpropane, glycine, pentacyclotriol, erythritol, threitol, dipentaerythritol, sorbitol, trimellitic anhydride, pyromellitidianehydrate, dimethylpropionic acid, or combinations thereof. Further examples of branching monomer concentration ranges are from 0 to about 20 mole % and from 0 to about 10 mole %. The presence of a branching monomer may result in a number of possible benefits to the sulfopolyester of the present invention, including but not limited to, the ability to tailor rheological, solubility, and tensile properties. For example, at a constant molecular weight, a branched sulfopolyester, compared to a linear analog, will also have a greater concentration of end groups that may facilitate post-polymerization crosslinking reactions. At high concentrations of branching agent, however, the sulfopolyester may be prone to gelation.

[0043] The sulfopolyester used for the multicomponent fiber of the present invention has a glass transition temperature, abbreviated herein as “Tg”, of at least 25° C. as measured on the dry polymer using standard techniques, such as differential scanning calorimetry (DSC”), well known to persons skilled in the art. The Tg measurements of the sulfopolyesters of the present invention are conducted using a “dry polymer”, that is, a polymer sample in which adventitious or absorbed water is driven off by heating to polymer to a temperature of about 200° C. and allowing the sample to return to room temperature. Typically, the sulfopolyester is dried in the DSC apparatus by conducting a first thermal scan in which the sample is heated disc temperature above the water vaporization temperature, holding the sample at that temperature until the vaporization of the water absorbed in the polymer is complete (as indicated by an a large, broad endotherm), cooling the sample to room temperature, and then conducting a second thermal scan to obtain the Tg measurement. Further examples of glass transition temperatures exhibited by the sulfopolyester are at least 30° C., at least 35° C., at least 40° C., at least 50° C., at least 60° C., at least 65° C., at least 80° C., and at least 90° C. Although other glass transition temperatures are possible, typical glass transition temperatures of the dry sulfopolyesters in our invention are about 30° C., about 48° C., about 55° C., about 65° C., about 70° C., about 75° C., about 85° C., and about 90° C.

[0044] In another embodiment, however, the sulfopolyesters of this invention may be a single polyester or may be blended with one or more supplemental polymers to modify the properties of the resulting multicomponent fiber. The supplemental polymer may or may not be water-dispersible depending on the application and may be miscible or immiscible with the sulfopolyester. If the supplemental polymer is
water non-dispersible, it is preferred that the blend with the sulfopolyester is immiscible. The term “miscible”, as used herein, is intended to mean that the blend has a single, homogeneous amorphous phase as indicated by a single composition-dependent Tg. For example, a first polymer that is miscible with second polymer may be used to “plasticize” the second polymer as illustrated, for example, in U.S. Pat. No. 6,211,309. By contrast, the term “immiscible”, as used herein, denotes a blend that shows at least 2, randomly mixed, phases and exhibits more than one Tg. Some polymers may be immiscible and yet compatible with the sulfopolyester. A further general description of miscible and immiscible polymer blends and the various analytical techniques for their characterization may be found in Polymer Blends Volumes 1 and 2, Edited by D. R. Paul and C. B. Bucknall, 2000, John Wiley & Sons, Inc.

[0045] Non-limiting examples of water-dispersible polymers that may be blended with the sulfopolyester are poly-methacrylic acid, polyvinyl pyrrolidone, polyethylene-acrylic acid copolymers, polyvinyl methyl ether, polyvinyl alcohol, polyethylene oxide, hydroxy propyl cellulose, hydroxypropyl methyl cellulose, methyl cellulose, ethyl hydroxyethyl cellulose, isopropyl cellulose, methyl ether starch, polyacrylamides, poly(N-vinyl caprolactam), poly-ethyl oxazoline, poly(2-isopropyl-2-oxazoline), polyvinyl methyl oxazolidine, water-dispersible sulfopolymesters, polyvinyl methyl oxazolidine, poly(2,4-dimethyl-6-triazinyl-ethylene), and ethylene oxide-propylene oxide copolymers.

Examples of polymers which are water non-dispersible that may be blended with the sulfopolyester include, but are not limited to, polyolefins, such as homo- and copolymers of polyethylene and propylene; poly(ethylene terephthalate); poly(ethylene terephthalate); and polyamides, such as nylon-6; polyamide; caprolactone; Eastar Bios (poly(tetramethylene adipate-co-terephthalate), a product of Eastman Chemical Company); polycarbonate; polyurethane; and polyvinyl chloride.

[0046] According to our invention, blends of more than one sulfopolyester may be used to tailor the end-use properties of the resulting nonwoven fabric or web. The blends of one or more sulfopolymers will have glass transition temperatures of at least 25°C. Thus, blending may also be exploited to alter the processing characteristics of a sulfopolyester to facilitate the fabrication of the nonwoven fabric.

[0047] The sulfopolyester and supplemental polymer may be blended in batch, semicontinuous, or continuous processes. Small scale batches may be readily prepared in any high-intensity mixing devices well-known to those skilled in the art, such as Banbury mixers, prior to melt-spinning fibers. The components may also be blended in solution in an appropriate solvent. The melt blending method includes blending the sulfopolyester and supplemental polymer at a temperature sufficient to melt the polymers. The blend may be cooled and pelletized for further use or the melt blend can be melt spun directly from this molten blend into fiber form. The term “melt” as used herein includes, but is not limited to, merely softening the polymer. For melt mixing methods generally known in the polymers art, see mixing and compounding of polymers (1. Manus-Zwozower & Z. Tadmor editors, Carl Hanser Verlag Publisher, 1994, New York, N.Y.).

[0048] In another embodiment of the invention, the sulfopolyester in the multicomponent fiber has a glass transition temperature (Tg) of at least 25°C, and the sulfopolyester comprises:

(A) about 50 to about 96 mole % of one or more residues of isophthalic acid or terephthalic acid, based on the total acid residues;

(B) about 4 to about 30 mole %, based on the total acid residues, of a residue of sodium sulfophthalic acid;

(C) one or more diol residues wherein at least 25 mole %, based on the total diol residues, is a poly(ethylene glycol) having a structure

$$\text{H} - (\text{OCH}_2 - \text{CH}_2)\_n - \text{OH}$$

wherein n is an integer in the range of 2 to about 500; (iv) 0 to about 20 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein the functional groups are hydroxyl, carboxyl, or a combination thereof. As described hereinabove, the multicomponent fiber may optionally include a first water-dispersible polymer blended with the sulfopolyester; and,

[0049] optionally, a water non-dispersible polymer blended with the sulfopolyester such that the blend is an immiscible blend. The first water-dispersible polymer is as described hereinabove. The sulfopolyester can have a glass transition temperature (Tg) of at least 25°C, but may have, for example, a Tg of about 35°C, about 48°C, about 55°C, about 65°C, about 70°C, about 75°C, about 85°C, and about 90°C. The sulfopolyester may contain other concentrations of isophthalic acid residues, for example, about 60 to about 95 mole %, and about 75 to about 95 mole %. Further examples of isophthalic acid residue concentrations ranges are about 70 to about 85 mole %, about 85 to about 95 mole % and about 90 to about 95 mole %. The sulfopolyester also may comprise about 25 to 95 mole % of the residues of diethylene glycol. Further examples of diethylene glycol residue concentration ranges include about 50 to about 95 mole %, about 70 to about 95 mole %, and about 75 to about 95 mole %.

The sulfopolyester also may include the residues of ethylene glycol and/or 1,4-cyclohexanediol, abbreviated herein as “CHDM”. Typical concentration ranges of CHDM residues are about 10 to about 75 mole %, about 25 to about 65 mole %, and about 40 to about 60 mole %. Typical concentration ranges of ethylene glycol residues are about 10 to about 75 mole %, about 25 to about 65 mole %, and about 40 to about 60 mole %. In another embodiment, the sulfopolyester comprises about 75 to about 96 mole % of the residues of isophthalic acid and about 25 to about 95 mole % of the residues of diethylene glycol.

[0050] The sulfopolyesters of the instant invention are readily prepared from the appropriate dicarboxylic acids, esters, anhydrides, or salts, sulfonmonomers, and the appropriate diol or diol mixtures using typical polycondensation reaction conditions. They may be made by continuous, semicontinuous, and batch modes of operation and may utilize a variety of reactor types. Examples of suitable reactor types include, but are not limited to, stirred tank, continuous stirred tank, slurry, tubular, wiped-film, falling film, or extrusion reactors. The term “continuous” as used herein means a process whereby reactants are introduced and products withdrawn simultaneously in an uninterrupted manner. By “continuous” it is meant that the process is substantially or completely continuous in operation and is to be contrasted with a “batch” process. “Continuous” is not meant in any way to prohibit normal interruptions in the continuity of the process due to, for example, start-up, reactor maintenance, or scheduled shut down periods. The term “batch” process as used herein means a process wherein all the reactants are
added to the reactor and then processed according to a predetermined course of reaction during which no material is fed or removed into the reactor. The term “semicontinuous” means a process where some of the reactants are charged at the beginning of the process and the remaining reactants are fed continuously as the reaction progresses. Alternatively, a semicontinuous process may also include a process similar to a batch process in which all the reactants are added at the beginning of the process except that one or more of the products are removed continuously as the reaction progresses. The process is operated advantageously as a continuous process for economic reasons and to produce superior coloration of the polymer as the sulfopolyester may deteriorate in appearance if allowed to reside in a reactor at an elevated temperature for too long a duration.

[0051] The sulfopolyesters of the present invention are prepared by procedures known to persons skilled in the art. The sulfononomer is most often added directly to the reaction mixture from which the polymer is made, although other processes are known and may also be employed, for example, as described in U.S. Pat. Nos. 3,018,272, 3,075,952, and 3,033,822. The reaction of the sulfononomer, diol component and the dicarboxylic acid component may be carried out using conventional polyester polymerization conditions.

[0052] For example, when preparing the sulfopolyesters by means of an ester interchange reaction, i.e., from the ester form of the dicarboxylic acid components, the reaction process may comprise two steps. In the first step, the diol component and the dicarboxylic acid component, such as, for example, dimethyl isophthalate, are reacted at elevated temperatures, typically, about 150°C to about 250°C, for about 0.5 to about 8 hours at pressures ranging from about 0.0 kPa gauge to about 414 kPa gauge (60 pounds per square inch, “psig”). Preferably, the temperature for the ester interchange reaction ranges from about 180°C to about 230°C for about 1 to about 4 hours while the preferred pressure ranges from about 103 kPa gauge (15 psig) to about 276 kPa gauge (40 psig).

[0053] Thereafter, the reaction product is heated under higher temperatures and under reduced pressure to form sulfopolyester with the elimination of diol, which is readily volatilized under these conditions and removed from the system. This second step, or polycondensation step, is continued under higher vacuum and a temperature which generally ranges from about 230°C to about 350°C, preferably about 250°C to about 310°C, and most preferably about 260°C, for about 0.1 to about 6 hours, or preferably, for about 0.2 to about 2 hours, until a polymer having the desired degree of polymerization, as determined by inherent viscosity, is obtained. The polycondensation step may be conducted under reduced pressure which ranges from about 53 kPa (400 torr) to about 0.013 kPa (0.1 torr). Stirring or appropriate conditions are used in both stages to ensure adequate heat transfer and surface renewal of the reaction mixture. The reactions of both stages are facilitated by appropriate catalysts such as, for example, alkoxyl titanium compounds, alkali metal hydroxides and alcoholates, salts of organic carboxylic acids, alkyl tin compounds, metal oxides, and the like. A three-stage manufacturing procedure, similar to that described in U.S. Pat. No. 5,290,631, may also be used, particularly when a mixed monomer feed of acids and esters is employed.

[0054] To ensure that the reaction of the diol component and dicarboxylic acid component by an ester interchange reaction mechanism is driven to completion, it is preferred to employ about 1.05 to about 2.5 moles of diol component to one mole dicarboxylic acid component. Persons of skill in the art will understand, however, that the ratio of diol component to dicarboxylic acid component is generally determined by the design of the reactor in which the reaction process occurs.

[0055] In the preparation of sulfopolyester by direct esterification, i.e., from the acid form of the dicarboxylic acid component, sulfopolyesters are produced by reacting the dicarboxylic acid or a mixture of dicarboxylic acids with the diol component or a mixture of diol components. The reaction is conducted at a pressure of from about 7 kPa gauge (1 psig) to about 1379 kPa gauge (200 psig), preferably less than 689 kPa (100 psig) to produce a low molecular weight, linear or branched sulfopolyester product having an average degree of polymerization of from about 1.4 to about 10. The temperatures employed during the direct esterification reaction typically range from about 180°C to about 280°C. Another range is from about 220°C to about 270°C. This low molecular weight polymer may then be polymerized by a polycondensation reaction.

[0056] The multicomponent fibers, nonwoven fabrics, and fibrous articles of this invention also may contain other conventional additives and ingredients which do not deleteriously affect their end use. For example, additives such as fillers, surface friction modifiers, light and heat stabilizers, extrusion aids, antistatic agents, colorants, dyes, pigments, fluorescent brighteners, antimicrobials, anticounterfeit markers, hydrophobic and hydrophilic enhancers, viscosity modifiers, slip agents, tougheners, adhesion promoters, and the like may be used.

[0057] The multicomponent fibers, nonwoven fabrics, and fibrous articles of our invention do not require the presence of additives such as, for example, pigments, fillers, oils, waxes, or fatty acid finishes, to prevent blocking or fusing of the fibers during processing. The terms “blocking or fusing”, as used herein, is understood to mean that the fibers or fibrous articles stick together or fuse into a mass such that the fiber cannot be processed or used for its intended purpose. Blocking and fusing can occur during processing of the fiber or fibrous article or during storage over a period of days or weeks and is exacerbated under hot, humid conditions.

[0058] In one embodiment of the invention, the multicomponent fibers, nonwoven fabrics, and fibrous articles can contain less than 10 wt % of such anti-blocking additives, based on the total weight of the multicomponent fiber or fibrous article. For example, the multicomponent fibers and fibrous articles may contain less than 10 wt % of a pigment or filler. In other examples, the multicomponent fibers, nonwoven fabrics, and fibrous articles may contain less than 9 wt %, less than 5 wt %, less than 3 wt %, less than 1 wt %, and 0 wt % of a pigment or filler, based on the total weight of the multicomponent fiber. Colorants, sometimes referred to as dyes, may be added to impart a desired neutral hue and/or brightness to the sulfopolyester. When colored multicomponent fibers are desired, pigments or colorants may be included in the sulfopolyester reaction mixture during the reaction of the diol monomer and the dicarboxylic acid monomer or they may be melt blended with the preformed sulfopolyester. A preferred method of including colorants is to use a colorant having thermally stable organic colored compounds having reactive
groups such that the colorant is copolymerized and incorporated into the sulfopolyester to improve its hue. For example, colorants such as dyes possessing reactive hydroxyl and/or carboxyl groups, including, but not limited to, blue and red substituted anthraquinones, may be copolymerized into the polymer chain. When dyes are employed as colorants, they may be added to the copolyester reaction process after an ester interchange or direct esterification reaction.

[0059] In one embodiment of the invention, the sulfopolyester contained in the multicomponent fiber has a glass transition temperature (Tg) of at least 57° C. A glass transition temperature (Tg) of at least 57° C. has been found to particularly useful for multicomponent fibers to prevent blocking and fusing of the multicomponent fiber during spinning and take up. Thus, in one embodiment of the invention, a multicomponent fiber having shaped cross section is utilized to produce the nonwoven fabric comprising:

(A) a water dispersible sulfopolyester having a glass transition temperature (Tg) of at least 57° C., the sulfopolyester comprising:

[0060] (i) residues of one or more dicarboxylic acids;
[0061] (ii) about 4 to about 40 mole %, based on the total repeating units, of residues of at least one sulfononomer having 2 functional groups and one or more sulfonate groups attached to an aromatic or cycloaliphatic ring wherein the functional groups are hydroxyl, carboxyl, or a combination thereof;
[0062] (iii) one or more diol residues wherein at least 25 mole %, based on the total diol residues, is a poly(ethylene glycol) having a structure

\[ \text{H} \rightarrow \text{(OCH}_2\text{CH}_3)_n \rightarrow \text{OH} \]

[0063] wherein n is an integer in the range of 2 to about 500; and

[0064] (iv) 0 to about 25 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein the functional groups are hydroxyl, carboxyl, or a combination thereof; and

(B) a plurality of segments comprising one or more water non-dispersible polymers immiscible with the sulfopolyester, wherein the segments are substantially isolated from each other by the sulfopolyester intervening between the segments;

[0065] wherein the fiber has an islands-in-the-sea or segmented pie cross section.

[0066] The dicarboxylic acids, diols, sulfopolyester, sulfononomers, and branching monomers residues are as described previously for other embodiments of the invention. For multicomponent fibers, it is advantageous that the sulfopolyester have a Tg of at least 57° C. Further examples of glass transition temperatures that may be exhibited by the sulfopolyester or sulfopolyester blend of the multicomponent fiber are at least 60° C., at least 65° C., at least 70° C., at least 75° C., at least 80° C., at least 85° C., and at least 90° C. Further, to obtain a sulfopolyester with a Tg of at least 57° C., blends of one or more sulfopolyesters may be used in varying proportions to obtain a sulfopolyester blend having the desired Tg. The Tg of a sulfopolyester blend may be calculated by using a weighted average of the glass transition temperatures of the sulfopolyester components. For example, sulfopolyester having a Tg of 48° C. may be blended in a 25:75 wt:wt ratio with another sulfopolyester having Tg of 65° C. to give a sulfopolyester blend having a Tg of approximately 61° C.

[0067] In another embodiment of the invention, the water dispersible sulfopolyester component of the multicomponent fiber presents properties which allow at least one of the following:

(A) the multicomponent fibers to be spun to a desired low denier, and

(B) the multicomponent fibers to be spun to a desired low denier, and

[0068] wherein the multicomponent fiber has an as-spun denier of less than about 6 denier per filament;

[0069] wherein the water dispersible sulfopolyester exhibits a melt viscosity of less than about 12,000 poise measured at 240° C. at a shear rate of 1 rad/sec, and wherein the sulfopolyester comprises less than about 25 mole % of residues of at least one sulfononomer, based on the total moles of diacid or diol residues.

[0070] The sulfopolyester utilized in these multicomponent fibers has a melt viscosity of generally less than about 12,000 poise. Other ranges of melt viscosity of the sulfopolyester is less than 10,000 poise, less than 6,000, and less than 4,000 poise measured at 240° C. and 1 rad/sec shear rate. In another aspect, the sulfopolyester exhibits a melt viscosity ranging from about 1000 to about 12,000 poise from about 2000 to about 6000 poise, or from about 2500 to about 4000 poise measured at 240° C. and 1 rad/sec shear rate. Prior to determining the viscosity, the solutions are dried at 60° C. in a vacuum oven for 2 days. The melt viscosity is measured using a rheometer having a 25 mm diameter parallel-plate geometry at 1 mm gap setting. A dynamic frequency sweep is run at a strain rate range of 1 to 400 rad/sec and 10% strain amplitude. The viscosity is then measured at 240° C. and strain rate of 1 rad/sec.

[0071] The level of sulfononomer residues in the sulfopolyester polymers for use in this embodiment of the invention is generally less than about 25 mole % or less than 20 mole %, reported as a percentage of the total diacid or diol residues in the sulfopolyester. Other ranges for the level of sulfononomer residues in the sulfopolyester polymers range from about 4 to about 20 mole %, about 5 to about 12 mole %, and between about 7 to about 10 mole %. In another aspect of this embodiment, the sulfonomomers for use with the invention can have 2 functional groups and one or more sulfonate groups attached to an aromatic or cycloaliphatic ring wherein the functional groups are hydroxyl, carboxyl, or a combination thereof, for example, sodiumsulfosulfo-isophalic acid monomer.

[0072] In addition to the sulfononomer described previously, the sulfopolyester can comprise residues of one or more dicarboxylic acids, one or more diol residues wherein at least 25 mole %, based on the total diol residues, is a poly(ethylene glycol) having a structure

\[ \text{H} \rightarrow \text{(OCH}_2\text{CH}_3)_n \rightarrow \text{OH} \]
wherein \( n \) is an integer in the range of 2 to about 500, and 0 to about 20 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein the functional groups are hydroxyl, carboxyl, or a combination thereof.

[0073] In one embodiment of the invention, the sulfopolyester comprises from about 80-90 mole % dicarboxylic acid residues, from about 4 to about 20 mole % sulfonmonomer residues, and 100 mole % diol residues (there being a total mole % of 200%, i.e., 100 mole % dicarboxylic acid and 100 mole % diol). More specifically, the dicarboxylic portion of the sulfopolyester comprises between about 60-80 mole % terephthalic acid, about 0-30 mole % isophthalic acid, and about 4-20 mole % 5-sodiumulisciophthalic acid (5-SSIPA). The diol portion comprises from about 0-50 mole % diethylene glycol and from about 50-100 mole % ethylene glycol. An exemplary formulation according to this embodiment of the invention is set forth subsequently.

<table>
<thead>
<tr>
<th>Approximate Mole % (based on total moles of diol or diacid residues)</th>
<th>Terephthalic acid</th>
<th>Isophthalic acid</th>
<th>5-SSIPA</th>
<th>Diethylene glycol</th>
<th>Ethylene glycol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Approximately Mole %</td>
<td>71</td>
<td>20</td>
<td>9</td>
<td>35</td>
<td>65</td>
</tr>
</tbody>
</table>

[0074] The water non-dispersible component of the multicomponent fiber may comprise any of those water non-dispersible polymers described herein. Spinning of the multicomponent fiber may also occur according to any method described herein. However, the improved rheological properties of multicomponent fibers in accordance with this aspect of the invention provide for enhanced drawdown speeds. When the water dispersible sulfopolyester has a melt viscosity of less than about 12,000 poise measured at 240° C. at a strain rate of 1 rad/sec, and wherein the sulfopolyester comprises less than about 25 mole % of residues of at least one sulfonmonomer, based on the total moles of diacid or diol residues, and is extruded to produce multicomponent extrudates, the multicomponent extrudate is capable of being melt drawn to produce the multicomponent fiber, using any of the methods disclosed herein, at a speed of at least about 2000 m/min. In another aspect of this embodiment, the multicomponent extrudate is capable of being melt drawn at a speed in the following ranges: at least about 3000 m/min, at least about 4000 m/min, or at least about 4500 m/min. Although not intending to be bound by theory, melt drawing of the multicomponent extrudates at these speeds results in at least some oriented crystallinity in the water non-dispersible component of the multicomponent fiber. This oriented crystallinity can increase the dimensional stability of nonwoven fabrics made from the multicomponent fibers during subsequent processing.

[0075] Another advantage of the multicomponent extrudate produced from sulfopolyesters having a melt viscosity of less than about 12,000 poise measured at 240° C. at a strain rate of 1 rad/sec, and wherein the sulfopolyester comprises less than 25 mole % of residues of at least one sulfonmonomer, based on the total moles of diacid or diol residues, is that it can be melt drawn to a multicomponent fiber having an as-spun denier of less than 6 deniers per filament. Other ranges of multicomponent fiber sizes include an as-spun denier of less than 4 deniers per filament and less than 2.5 deniers per filament.

[0076] The multicomponent fiber comprises a plurality of segments or domains of one or more water non-dispersible polymers immiscible with the sulfopolyester in which the segments or domains are substantially isolated from each other by the sulfopolyester intervening between the segments or domains. The term “substantially isolated”, as used herein, is intended to mean that the segments or domains are set apart from each other to permit the segments domains to form individual fibers upon removal of the sulfopolyester. For example, the segments or domains may be touching each others as in, for example, a segmented pie configuration but can be split apart by impact or when the sulfopolyester is removed.

[0077] The ratio by weight of the sulfopolyester to water non-dispersible polymer component in the multicomponent fiber of the invention is generally in the range of about 60:40 to about 2:98 or, in another example, in the range of about 50:50 to about 5:95. Typically, the sulfopolyester comprises 50% by weight or less of the total weight of the multicomponent fiber.

[0078] The segments or domains of the multicomponent fiber comprise one of more water non-dispersible polymers. Examples of water non-dispersible polymers which are used in segments of the multicomponent fiber include, but are not limited to, polyeleins, polystyres, polynamides, polylactides, polycaprolactone, polycarbonate, polyurethane, and polyvinyl chloride. For example, the water non-dispersible polymer may be polyester such as poly(ethylene) terephthalate, poly(ethylene) terephthalate, poly(cyclohexylene) cyclohexanediacarblylate, poly(cyclohexylene) terephthalate, poly(tetramethylene) terephthalate, and the like.

[0079] In one embodiment of the invention, the water non-dispersible polymer is poly(ethylene) terephthalate having an inherent viscosity of less than 0.6 Dl/g, measured in a 60/40 parts by weight solution of phenol/tetrachloroethane solvent at 25° C. and at a concentration of about 0.5 g of poly(ethylene) terephthalate in 100 mL of solvent. Other ranges are less than 0.55 Dl/g, less than 0.4 Dl/g, and less than 0.3 Dl/g. PET having this inherent viscosity can produce improved nonwoven fabrics having good fiber entanglement and less loose fibers on the surface of the nonwoven fabric.

[0080] In another example, the water non-dispersible polymer can be biodisintegratable as determined by DIN Standard 54900 and/or biodegradable as determined by ASTM Standard Method, D6340-98. Examples of biodegradable polyesters and polyester blends are disclosed in U.S. Pat. Nos. 5,599,858; 5,580,911; 5,446,079; and 5,559,171. The term “biodegradable”, as used herein in reference to the water non-dispersible polymers of the present invention, is understood to mean that the polymers are degraded under environmental influences such as, for example, in a composting environment, in an appropriate and demonstrable time span as defined, for example, by ASTM Standard Method, D6340-98, entitled “Standard Test Methods for Determining Aerobic Biodegradation of Radiolabeled Plastics Materials in an Aerobic Environment”. The water non-dispersible polymers of the present invention also may be “biodisintegratable”, meaning that the polymers are easily fragmented in a composting environment as defined, for example, by DIN Standard 54900. For example, the biodegradable polymer is initially reduced in molecular weight in the environment by
the action of heat, water, air, microbes, and other factors. This reduction in molecular weight results in a loss of physical properties (tenacity) and often in fiber breakage. Once the molecular weight of the polymer is sufficiently low, the monomers and oligomers are then assimilated by the microbes. In an aerobic environment, these monomers or oligomers are ultimately oxidized to CO₂, H₂O, and new cell biomass. In an anaerobic environment, the monomers or oligomers are ultimately converted to CO₂, H₂, acetate, methane, and cell biomass.

[0081] For example, water non-dispersible polymer may be an aliphatic-aromatic polyester, abbreviated herein as “AAPE”. The term “aliphatic-aromatic polyester”, as used herein, means a polyester comprising a mixture of residues from aliphatic or cycloaliphatic dicarboxylic acids or diols and aromatic dicarboxylic acids or diols. The term “non-aromatic”, as used herein with respect to the dicarboxylic acid and diol monomers of the present invention, means that the backbone or hydroxyl groups of the monomer are not connected through an aromatic nucleus. For example, adipic acid contains no aromatic nucleus in its backbone, i.e., the chain of carbon atoms connecting the carboxylic acid groups, thus is “non-aromatic”. By contrast, the term “aromatic” means the dicarboxylic acid or diol contains an aromatic nucleus in the backbone such as, for example, terephthalic acid or 2,6-naphthalene dicarboxylic acid. “Non-aromatic”, therefore, is intended to include both aliphatic and cycloaliphatic structures such as, for example, diols and dicarboxylic acids which contain as a backbone a straight or branched chain or cyclic arrangement of the constituent carbon atoms which may be saturated or unsaturated in nature, unsaturated, i.e., containing non-aromatic carbon-carbon double bonds, or acetylenic, i.e., containing carbon-carbon triple bonds. Thus, in the context of the description and the claims of the present invention, non-aromatic is intended to include linear and branched, chain structures (referred to herein as “aliphatic”) and cyclic structures (referred to herein as “alicyclic” or “cycloaliphatic”). The term “non-aromatic”, however, is not intended to exclude any aromatic substituents which may be attached to the backbone of an aliphatic or cycloaliphatic diol or dicarboxylic acid. In the present invention, the functional carboxylic acid typically is a aliphatic dicarboxylic acid such as, for example, adipic acid, or an aromatic dicarboxylic acid such as, for example, terephthalic acid. The functional hydroxyl compound may be cycloaliphatic diol such as, for example, 1,4-cyclohexanediol, a linear or branched aliphatic diol such as, for example, 1,4-butanediol, or an aromatic diol such as, for example, hydroquinone.

[0082] The AAPE may be a linear or branched random copolyester and/or chain extended copolyester comprising diol residues which comprise the residues of one or more substituted or unsubstituted, linear or branched, diols selected from aliphatic diols containing 2 to about 8 carbon atoms, polyalkylene ether glycols containing 2 to 8 carbon atoms, and cycloaliphatic diols containing about 4 to about 12 carbon atoms. The substituted diols, typically, will comprise 1 to about 4 substituents independently selected from halo, C₆-C₁₀ aryl, and C₁₋₄ alkoxy. Examples of diols which may be used include, but are not limited to, ethylene glycol, diethylene glycol, propylene glycol, 1,3-propanediol, 2,2-dimethyl-1,3-propanediol, 1,3-butadiol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, polyethylene glycol, diethylene glycol, 2,2,4-trimethyl-1,6-hexanediol, thio-diethanol, 1,3-cyclohexanediol, 1,4-cyclohexanediol, 2,2,4, 4-tetramethyl-1,3-cyclobutanediol, triethylene glycol, and tetracyethylene glycol with the preferred diols comprising one or more diols selected from 1,4-butanediol; 1,3-propanediol; ethylene glycol; 1,6-hexanediol; diethylene glycol; or 1,4-cyclohexanediol. The AAPE also comprises diacid residues which contain about 35 to about 99 mole %, based on the total moles of diacid residues, of the residues of one or more substituted or unsubstituted, linear or branched, non-aromatic dicarboxylic acids selected from aliphatic dicarboxylic acids containing 2 to about 12 carbon atoms and cycloaliphatic diacids containing about 5 to about 10 carbon atoms. The substituted non-aromatic dicarboxylic acids will typically contain 1 to about 4 substituents selected from halo, C₆-C₁₀ aryl, and C₁₋₄ alkoxy. Non-limiting examples of non-aromatic diacids include malonic, succinic, glutaric, adipic, pimelic, azelaic, sebacic, fumaric, 2,2-dimethyl glutaric, suberic, 1,3-cyclopentanedicarboxylic, 1,4-cyclohexanedicarboxylic, 1,3-cyclohexanediacarboxylic, diglycolic, itaconic, maleic, and 2,5-norbornedicarboxylic. In addition to the non-aromatic dicarboxylic acids, the AAPE comprises about 1 to about 65 mole %, based on the total moles of diacid residues, of the residues of one or more substituted or unsubstituted aromatic dicarboxylic acids containing 6 to about 10 carbon atoms. In the case where substituted aromatic dicarboxylic acids are used, they will typically contain 1 to about 4 substituents selected from halo, C₆-C₁₀ aryl, and C₁₋₄ alkoxy. Non-limiting examples of aromatic dicarboxylic acids which may be used in the AAPE of the present invention are terephthalic acid, isophthalic acid, salts of 5-sulfoisophthalic acid, and 2,6-naphthalenedicarboxylic acid. More preferably, the non-aromatic dicarboxylic acid will comprise adipic acid, the aromatic dicarboxylic acid will comprise terephthalic acid, and the diol will comprise 1,4-butanediol.

[0083] Other possible compositions for the AAPE’s of our invention are those prepared from the following diols and dicarboxylic acids (or polyester-forming equivalents thereof such as diesters) in the following mole percentages, based on 100 mole percent of diacid component and 100 mole percent of diol component:

[0084] 1. glutaric acid (about 30 to about 75%); terephthalic acid (about 25 to about 70%); 1,4-butanediol (about 90 to 100%); and modifying diol (0 about 10%);

[0085] 2. succinic acid (about 30 to about 95%); terephthalic acid (about 5 to about 70%); 1,4-butanediol (about 90 to 100%); and modifying diol (0 about 10%); and

[0086] 3. adipic acid (about 30 to about 75%); terephthalic acid (about 25 to about 70%); 1,4-butanediol (about 90 to 100%); and modifying diol (0 about 10%).

[0087] The modifying diol preferably is selected from 1,4-cyclohexanediol, triethylene glycol, polyethylene glycol and neopentyl glycol. The most preferred AAPE’s are linear, branched or chain extended copolyesters comprising about 50 to about 60 mole percent adipic acid residues, about 40 to about 50 mole percent terephthalic acid residues, and at least 95 mole percent 1,4-butanediol residues. Even more preferably, the adipic acid residues comprise about 55 to about 60 mole percent, the terephthalic acid residues comprise about 40 to about 45 mole percent, and the diol residues comprise about 95 mole percent 1,4-butanediol residues. Such compositions are commercially available under the trademark EASTAR BIO® copolyester from Eastman Chemical Company, Kingsport, Tenn., and under the trademark ECOFLEX® from BASF Corporation.
Additional, specific examples of preferred AAPE’s include a poly(tetra-methylene glutarate-co-terephthalate) containing (a) 50 mole percent glutaric acid residues, 50 mole percent terephthalic acid residues, and 100 mole percent 1,4-butenediol residues, (b) 60 mole percent glutaric acid residues, 40 mole percent terephthalic acid residues, and 100 mole percent 1,4-butenediol residues or (c) 40 mole percent glutaric acid residues, 60 mole percent terephthalic acid residues, and 100 mole percent 1,4-butenediol residues; a poly(tetramethylene-succinate-co-terephthalate) containing (a) 85 mole percent succinic acid residues, 15 mole percent terephthalic acid residues, and 100 mole percent 1,4-butenediol residues or (b) 70 mole percent succinic acid residues, 30 mole percent terephthalic acid residues, and 100 mole percent 1,4-butenediol residues; a poly(ethylene succinate-co-terephthalate) containing 70 mole percent succinic acid residues, 30 mole percent terephthalic acid residues, and 100 mole percent ethylene glycol residues; and a poly(tetramethylene adipate-co-terephthalate) containing (a) 85 mole percent adipic acid residues, 15 mole percent terephthalic acid residues, and 100 mole percent 1,4-butenediol residues; or (b) 55 mole percent adipic acid residues, 45 mole percent terephthalic acid residues, and 100 mole percent 1,4-butenediol residues.

The AAPE preferably comprises from about 10 to about 1,000 repeating units and preferably, from about 15 to about 600 repeating units. The AAPE may have an inherent viscosity of about 0.4 to about 2.0 dl/g, or more preferably about 0.7 to about 1.6 dl/g, as measured at a temperature of 25°C, using a concentration of 0.5 gram copolyester in 100 ml of a 60/40 by weight solution of phenol/tetrachloroethane.

The AAPE, optionally, may contain the residues of a branching agent. The mole percentage ranges for the branching agent are from about 0 to about 2 mole %, preferably about 0.1 to about 1 mole %, and most preferably about 0.1 to about 0.5 mole % based on the total moles of diacid or diol residues (depending on whether the branching agent contains carboxylic or hydroxyl groups). The branching agent preferably has a weight average molecular weight of about 50 to about 5000, more preferably about 92 to about 3000, and a functionality of about 3 to about 6. The branching agent, for example, may be the esterified residue of a polyol having 3 to 6 hydroxyl groups, a polycarboxylic acid having 3 to 4 carboxyl groups (or ester-forming equivalent groups) or a hydroxy acid having a total of 3 to 6 hydroxyl and carboxyl groups. In addition, the AAPE may be branched by the addition of a peroxide during reactive extrusion.

Each segment of the water non-dispersible polymer may be different from others in fineness and may be arranged in any shaped or engineered cross-sectional geometry known to persons skilled in the art. For example, the sulfopolyester and a water non-dispersible polymer may be used to prepare a multicomponent fiber having an engineered geometry such as, for example, a side-by-side, “islands-in-the-sea”, segmented pie, other splittables, sheath/core, or other configurations known to persons skilled in the art. Other multi-component configurations are also possible. Subsequent removal of a side, the “sea”, or a portion of the “pie” can result in very fine fibers. The process of preparing multicomponent fibers is also well known to persons skilled in the art. In a multicomponent fiber, the sulfopolyester fibers of this invention may be present in amounts of about 10 to about 50 weight % and will generally be used in the sheath portion of sheath/core fibers. The other component may be from a wide range of other polymeric materials such as, for example, poly(ethylene terephthalate), poly(butylene terephthalate), poly(trimethylene terephthalate), polyactides and the like as well as polyolefins, cellulose esters, and polyamides. Typically, when a water-insoluble or water non-dispersible polymer is used, the resulting bicomponent or multicomponent fiber is not completely water-dispersible. Side by side combinations with significant differences in thermal shrinkage can be utilized for the development of a spiral crimp. If crimping is desired, a saw tooth or stuffer box crimp is generally suitable for many applications. If the second polymer component is in the core of a sheath/core configuration, such a core optionally may be stabilized.

The sulfopolyesters are particularly useful for fibers having an “islands-in-the-sea” or “segmented pie” cross section as they only need requires neutral or slightly acidic (i.e., “soft” water) to disperse, as compared to the caustic-containing solutions that are sometimes required to remove other water dispersible polymers from multicomponent fibers. Thus, another aspect of our invention is when the multicomponent fiber comprises:

(A) a water dispersible sulfopolyester having a glass transition temperature (Tg) of at least 57°C, the sulfopolyester comprising:

(i) about 50 to about 96 mole % of one or more residues of isophthalic acid or terephthalic acid, based on the total acid residues;

(ii) about 4 to about 30 mole %, based on the total acid residues, of a residue of sodiosulfoisophthalic acid;

(iii) one or more diol residues wherein at least 25 mole %, based on the total diol residues, is a poly(ethylene glycol) having a structure

H−(OCH2−CH2)n−OH

wherein n is an integer in the range of 2 to about 500;

(iv) 0 to about 20 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein the functional groups are hydroxyl, carboxyl, or a combination thereof; and

(B) a plurality of segments comprising one or more water non-dispersible polymers immiscible with the sulfopolyester, wherein the segments are substantially isolated from each other by the sulfopolyester intervening between the segments;

wherein the fiber has an islands-in-the-sea or segmented pie cross section.

The dicarboxylic acids, diols, sulfopolyester, sulfonomomers, branching monomers residues, and water non-dispersible polymers are as described previously. For multicomponent fibers, it is advantageous that sulfopolyester have a Tg of at least 57°C. The sulfopolyester may be a single sulfopolyester or a blend of one or more sulfopolyester polymers. Further examples of glass transition temperatures that may be exhibited by the sulfopolyester or sulfopolyester blends are at least 65°C, at least 70°C, at least 75°C, and at least 85°C, and at least 90°C. For example, the sulfopolyester may comprise about 75 to about 95 mole % of one or more residues of isophthalic acid or terephthalic acid and about 25 to about 95 mole % of a residue of diethylene glycol. As described hereinafter, examples of the water non-dispersible polymers are polyolefins, polyesters, polyamides, polyactides, polycaprolactone, polycarbonate, polyurethane, and polyvinyl chloride. In addition, the water non-dispersible polymer may be
biodegradable or biodisintegrable. For example, the water non-dispersible polymer may be an aliphatic-aromatic polyester as described previously.

[0099] The multicomponent fiber may be prepared by any number of methods known to persons skilled in the art. In one embodiment of the invention, the multicomponent fiber having a shaped cross section is produced by a process comprising: spinning a water dispersible sulfoxopolyester having a glass transition temperature (Tg) of at least 57°C and one or more water non-dispersible polymers immiscible with the sulfoxopolyester into a fiber, the sulfoxopolyester comprising:

0100 (i) residues of one or more dicarboxylic acids;

0101 (ii) about 4 to about 40 mole %, based on the total repeating units, of residues of at least one sulfoxonomer having 2 functional groups and one or more sulfone groups attached to an aromatic or cycloaliphatic ring wherein the functional groups are hydroxyl, carboxyl, or a combination thereof;

0102 (iii) one or more diol residues wherein at least 25 mole %, based on the total diol residues, is a poly(ethylene glycol) having a structure

$$\text{H} = \text{(OCH}_2\text{CH}_2\text{)}\text{n} = \text{OH}$$

0103 wherein n is an integer in the range of 2 to about 500; and

0104 (iv) 0 to about 25 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein the functional groups are hydroxyl, carboxyl, or a combination thereof; wherein the fiber has a plurality of segments comprising the water non-dispersible polymers and the segments are substantially isolated from each other by the sulfoxopolyester intervening between the segments.

0105 For example, the multicomponent fiber may be prepared by melting the sulfoxopolyester and one or more water non-dispersible polymers in separate extruders and directing the individual polymer flows into one spinneret or extrusion die with a plurality of distribution flow paths such that the water non-dispersible polymer component form small segments or thin strands which are substantially isolated from each other by the intervening sulfoxopolyester. The cross section of such a fiber may be, for example, a segmented pie arrangement or an islands-in-the-sea arrangement. In another example, the sulfoxopolyester and one or more water non-dispersible polymers are separately fed to the spinneret orifices and then extruded in sheath-core form in which the water non-dispersible polymer forms a “core” that is substantially enclosed by the sulfoxopolyester “sheath” polymer. In the case of such concentric fibers, the orifice supplying the “core” polymer is in the center of the spinning orifice outlet and flow conditions of core polymer fluid are strictly controlled to maintain the concentricity of both components when spinning. Modifications in spinneret orifices enable different shapes of core and/or sheath to be obtained within the fiber cross-section. In yet another example, a multicomponent fiber having a side-by-side cross section or configuration may be produced by coextruding the water dispersible sulfoxopolyester and water non-dispersible polymer through orifices separately and converging the separate polymer streams at substantially the same speed to merge side-by-side as a combined stream below the face of the spinneret; or (2) by feeding the two polymer streams separately through orifices, which converge at the surface of the spinneret, at substantially the same speed to merge side-by-side as a combined stream at the surface of the spinneret. In both cases, the velocity of each polymer stream, at the point of merge, is determined by its metering pump speed, the number of orifices, and the size of the orifice.

0106 The dicarboxylic acids, diols, sulfoxopolyester, sulfoxonomers, branching monomers residues, and water non-dispersible polymers are as described previously. The sulfoxopolyester has a glass transition temperature of at least 57°C. Further examples of glass transition temperatures that may be exhibited by the sulfoxopolyester or sulfoxopolyester blend are at least 65°C, at least 70°C, at least 75°C, at least 85°C, and at least 90°C. In one example, the sulfoxopolyester may comprise about 50 to about 96 mole % of one or more residues of isophthalic acid or terephthalic acid, based on the total acid residues; and about 4 to about 30 mole %, based on the total acid residues, of a residue of sodiumisophthalic acid; and 0 to about 20 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein the functional groups are hydroxyl, carboxyl, or a combination thereof. In another example, the sulfoxopolyester may comprise about 75 to about 96 mole % of one or more residues of isophthalic acid or terephthalic acid and about 25 to about 95 mole % of a residue of diethylene glycol. As described hereinabove, examples of the water non-dispersible polymers are polyolefins, polyesters, polynamides, polyactides, polyacetals, polycarbonates, polyurethanes, and polyvinyl chloride. In addition, the water non-dispersible polymer may be biodegradable or biodisintegratable. For example, the water non-dispersible polymer may be an aliphatic-aromatic polyester as described previously. Examples of shaped cross sections include, but are not limited to, islands-in-the-sea, side-by-side, sheath-core, or segmented pie configurations.

0107 In another embodiment of the invention, the multicomponent fiber having a shaped cross section is produced by a process comprising: spinning at least one water dispersible sulfoxopolyester and one or more water non-dispersible polymers immiscible with the sulfoxopolyester to produce a multicomponent fiber, wherein the multicomponent fiber has a plurality of domains comprising the water non-dispersible polymers and the domains are substantially isolated from each other by the sulfoxopolyester intervening between the domains; wherein the water dispersible sulfoxopolyester exhibits a melt viscosity of less than about 1,200 poise measured at 240°C at a strain rate of 1 rad/sec, and wherein the sulfoxopolyester comprising less than about 25 mole % of residues of at least one sulfoxonomer, based on the total moles of diacid or diol residues; and wherein the multicomponent fiber has an as-spun denier of less than about 6 denier per filament.

0108 The sulfoxopolyester utilized in these multicomponent fiber and the water non-dispersible polymers were discussed previously in this disclosure.

0109 In another embodiment of this invention, the multicomponent fiber having a shaped cross section can be produced by a process comprising: (A) extruding at least one water dispersible sulfoxopolyester and one or more water non-dispersible polymers immiscible with said sulfoxopolyester to produce a multicomponent extrudate, wherein the multicomponent extrudate has a plurality of domains comprising the water non-dispersible polymers and the domains are substantially isolated from each other by the sulfoxopolyester intervening between the domains; and
(B) melt drawing the multicomponent extrudate at a speed of at least about 2000 m/min to produce the multicomponent fiber.

[0110] It is also a feature of this embodiment that the process includes the step of melt drawing the multicomponent extrudate at a speed of at least about 2000 m/min, more preferably, at least about 3000 m/min, and most preferably at least 4500 m/min.

[0111] Typically, upon exiting the spinneret, the fibers are quenched with a cross flow of air whereupon the fibers solidify. Various finishes and sizes may be applied to the fiber at this stage. The cooled fibers, typically, are subsequently drawn and wound up on a take up spool. Other additives may be incorporated in the finish in effective amounts like emulsifiers, antistatics, antimicrobials, antifoams, lubricants, thermostabilizers, UV stabilizers, and the like.

[0112] Optionally, the drawn fibers may be textured and wound-up to form a bulky continuous filament. This one-step technique is known in the art as spin-draw-texturizing. Other embodiments include flat filament (non-textured) yarns, or cut staple fiber, either crimped or uncrimped.

[0113] Step (A) of the inventive process comprises collecting the multicomponent fibers to form a non-woven web. The multicomponents fibers produced by the processes discussed previously can be collected in a manner to produce a non-woven web such that a support for the multicomponent fibers is not necessary. This can be accomplished by any method known in the art. For example, the multicomponent fibers can be subjected to such processes as mechanical needling, chemical bonding, thermally calendaring, ultrasonic fusing, or hydroentangling, in order to provide enough consolidation where a support for the multicomponent fibers is not necessary. An example of mechanical needling is needle punching.

[0114] In one embodiment of the invention, when hydroentangling is chosen to consolidate or bond the multicomponent fibers only sufficient hydroentangling is conducted in this step such that a support is not necessary for the multicomponent fibers. In one embodiment of the invention, the amount of hydroentangling energy expended in this process can range from about 20% to about 50% of the hydroentangling energy expended in Step (C) of the process. As will be discussed later in this invention, hydroentangling after sulfopolyester removal, (Step C) of the process, is the step where the majority of the energy is expended to interlock the multicomponent fibers to produce the nonwoven fabric. Since the fibers are finer when the sulfopolyester is removed, this provides for an improved nonwoven fabric and also requires less hydroentangling energy than traditional systems that hydroentangle fibers prior to sulfopolyester removal.

[0115] In another embodiment of the invention, extrusion technology can be utilized to produce the nonwoven web of Step (A) from the materials used to make the multicomponent fibers themselves, thus eliminating the multicomponent fiber production step. These processes include spunbond and meltblown processes. The spunbond process transforms the water dispersible sulfopolyester and water non-dispersible polymer directly into a nonwoven web by extruding multicomponent fibers, orienting them as bundles or groupings, and layering them on a conveying screen, without a separate fiber production step. In conventional processes, the multicomponent fiber is first spun and collected, then formed into a fabric by a separate process. Therefore, the water dispersible sulfopolyester and water non-dispersible polymer are routed through the spunbond process to yield a nonwoven web of Step (A). FIG. 1 shows the spunbond process. In the meltblown process, the water dispersible sulfopolyester and water non-dispersible polymer are heated to the liquid state and as it passes through an extrusion orifice, it is injected with sonic velocity air at about 250 to 500°C. The fast moving air stream stretches the molten polymer and solidifies it to produce fine multicomponent fibers. The multicomponent fibers are then separated from the air stream as an entangled web and compressed between heated rolls to produce the nonwoven web of Step (A).

[0116] Generally, the weight of the nonwoven web can range from about 10 grams/m² to about 800 grams/m². Other ranges are 10 grams/m² to 600 grams/m², 10 grams/m² to 400 grams/m², 50 grams/m² to 300 grams/m², and about 50 grams/m² to about 150 grams/m².

[0117] Step (B) of the inventive process comprises contacting the nonwoven web with water at a sufficient extraction temperature and pressure to remove a portion of the water dispersible sulfopolyester thereby forming a microfiber web. Typically, the multicomponent fiber can be contacted with water at an extraction temperature of about 50°C to about 100°C. Other extraction temperature ranges are from 25°C to about 100°C and from about 40°C to about 90°C.

[0118] The extraction temperature is defined by the following procedure. A nonwoven web consisting of multicomponent fibers with a known water dispersible sulfopolyester content is weighed and then taped to a metal backing plate and covered with a 100-200 mesh stainless steel screen on top. The backing plate with nonwoven fabric is placed in a deionized water bath at a test temperature in the range about 20°C to about 60°C for 10 minutes to extract the water dispersible sulfopolyester. After the conditioning, the specimen is removed from the water, and the excess water is removed by placing an absorbent towel above the screen mesh and squeezing. The conditioned fabric web is dried in a forced air oven at 60°C and the final weight is measured. The weight loss due to extraction is calculated as a percentage of the starting weight. The temperature of the conditioning water at which greater than 80% of the sulfopolyester polymer in the starting fiber is removed during the conditioning is designated as the sulfopolyester extraction temperature.

[0119] Generally, the temperature, pressure, contact time of the water are such that the water dispersible sulfopolyester is removed, and the nonwoven web remains sufficiently intact as a structure. The pressure of the water can range from about 30 bar to about 600 bar. Other pressure ranges are 50 bar to 300 bar.

[0120] In one embodiment of the invention, the nonwoven web can be contacted with water for a time sufficient to remove about 30% by weight to about 100% by weight of the total water dispersible sulfopolyester contained in the multicomponent fiber. In another embodiment of the invention, greater than 90% by weight of the total water dispersible sulfopolyester is removed from the nonwoven web. In another embodiment, greater than 95% by weight of the total water dispersible sulfopolyester is removed from the nonwoven web.

[0121] In another embodiment, the nonwoven web is contacted with water for a time period of from about 10 to about 600 seconds whereby the water dispersible sulfopolyester is dissipated or dissolved.

[0122] After removal of the sulfopolyester, the remaining water non-dispersible polymer microfibers can have an average fineness of less than 30% of the denier of the starting
multicomponent fiber. The remaining water non-dispersible polymer microfibers typically will have an average fineness of 1 d/f or less, typically, 0.5 d/f or less, or more typically, 0.11 d/f or less.

[0123] Generally, the nonwoven web is contacted with water via any method known in the art. For example, water jets can be utilized. In one embodiment of the invention, one to eight water jet heads can be utilized. The amount of water used ranges from less than 500 times to less than 1000 times the weight of the nonwoven web prior to contacting with water.

[0124] The wash water containing the water-dispersible sulfolon polyester can be recycled. In one embodiment of the invention, 80% or more of the wash water is recycled. This is quite an advancement since in conventional processes to produce nonwoven fabrics, the wash water generally cannot be recycled due to the water dispersible sulfolon polyester that may plug equipment and may cause excess machine wear. In another embodiment of the invention, substantially all of the sulfolon polyester is removed from the wash water. Sulfolon polyester recycling processes are described in U.S. patent application Ser. No. 11/343,955 filed Jan. 31st, 2006 to Gupta entitled “Sulfolon Polyester Recovery”, which is hereby incorporated by reference to the extent it does not contradict the statements herein.

[0125] Step (C) comprises hydroentangling the microfiber web to produce the nonwoven fabric. Generally, the water temperature during hydroentangling is less than 40° C. The pressure can range from about 150 bar to about 220 bar. During this hydroentangling step, less than 1% by weight of the water-dispersible sulfolon polyester is removed from the microfiber web. In other embodiments of the invention, the amount of water-dispersible sulfolon polyester removed can be less than 0.8% by weight, less than 0.5% by weight, less than 0.25% by weight, less than 0.1% by weight, less than 0.08% by weight, less than 0.05% by weight, and less than 0.02% by weight based on the weight of the microfiber web. The water from the hydroentangling step can be recycled as discussed previously. Due to the low level of water-dispersible sulfolon polyester, the sulfolon polyester may not need to be removed from the wash water before reuse.

[0126] In one embodiment of the invention, the hydroentangling is conducted by water jets. The number of waterjet heads can range from 1 to about 20, or from 2 to about 15, or 2 to about 10. Because a substantial amount of the sulfolon polyester has been removed, the fibers contained in the microfiber web are much finer, and during the hydroentangling step, a superior nonwoven fabric is produced having a tighter configuration with less fraying and loose fibers.

[0127] In addition, the hydroentangling process of this invention can be simplified over conventional nonwoven hydroentangling processes where hydroentangling occurs prior to removal of the water-dispersible polymer. Since the water non-dispersible polymer fibers in the microfiber web are finer, less hydraulic energy is needed to entangle the fibers. Furthermore, in conventional nonwoven processes, hydroentangling typically occurs prior to the removal of the water-dispersible sulfolon polyester, and often times, further hydroentangling after removal of the sulfolon polyester is required. However, in the present invention, a quality nonwoven fabric can be obtained without a further hydroentangling step.

[0128] In another embodiment of the invention, a process for making a nonwoven fabric is provided comprising:

[0129] (A) collecting multicomponent fibers to form a non-woven web; wherein the multicomponent fiber comprises at least one water-dispersible sulfolon polyester and at least one water non-dispersible polymer; wherein the multicomponent fiber has a plurality of domains comprising the water non-dispersible polymer; wherein the domains are substantially isolated from each other by the water-dispersible sulfolon polyester intervening between the domains; and

[0130] (B) contacting the non-woven web with water at a sufficient temperature and pressure to remove a portion of said sulfolon polyester thereby forming a microfibers and simultaneously hydroentangling the microfibers to produce the nonwoven fabric.

Steps (A) and (B) have been previously described in this disclosure.

[0131] The non-woven fabric can undergo a heat setting step comprising heating the non-woven fabric to a temperature of at least about 100° C., and more preferably at least about 120° C. The heat setting step relaxes out internal fiber stresses and aids in producing a dimensionally stable fabric product. It is preferred that when the heat set material is reheated to the temperature to which it was heated during the heat setting step that it exhibits surface area shrinkage of less than about 5% of its original surface area. More preferably, the shrinkage is less than about 2% of the original surface area, and most preferably the shrinkage is less than about 1%.

[0132] The nonwoven fabric can optionally be held together by 1) mechanical fiber cohesion and interlocking in a web or mat; 2) various techniques of fusing fibers, including the use of binder fibers, utilizing the thermoplastic properties of certain polymers and polymer blends; 3) use of a binding resin such as starch, casein, a cellulose derivative, or a synthetic resin, such as an acrylic latex or urethane; 4) powder adhesive binders; or 5) combinations thereof. The fibers in the nonwoven fabric are often deposited in a random manner, although orientation in one direction is possible, followed by bonding using one of the methods described above.

[0133] The instant invention produces nonwoven fabrics that can be utilized in clothing, curtains, upholstery, and uniforms. Nonwoven fabrics can also be utilized in personal care products, such as, but not limited to, wipes, feminine hygiene products, baby diapers, training pants, adult incontinence briefs, and hospital/surgical and other medical disposables, such as, but not limited to, surgical gowns, gauze, bandages and the like. Other applications include, but are not limited to, multilayer nonwovens, laminates and composites, protective fabrics and layers, geotextiles, industrial wipes, and filter media. Further, the fibrous articles may include replacement inserts for various personal hygiene and cleaning products. The fibrous article of the present invention may be bonded, laminated, attached to, or used in conjunction with other materials which may or may not be water-dispersible. The fibrous article, for example, a nonwoven fabric layer, may be bonded to a flexible plastic film or backing of a water non-dispersible material, such as polyethylene. Such an assembly, for example, could be used as one component of a disposable diaper.

EXAMPLES

[0134] The multicomponent fibers and nonwoven fabrics described in the following examples were produced in the manner described below. A pilot scale bicomponent spun-
Separate extruders and spin pumps were utilized to melt and meter the water dispersible sulfopolyester and the PET into the bicomponent spin pack. One extruder melted and metered the water dispersible sulfopolyester and the second extruder melted and metered the PET polymer. In this process, the water dispersible sulfopolyester was extruded at a melt temperature of 260°C, and the PET component was extruded at a 285°C melt temperature. The total combined polymer flow was about 1.33 kg/min., resulting in an average polymer flow rate exiting each die hole of about 0.60 grams/hole per minute from a spinneret having 2222 holes.

Both polymer melt streams entered a segmented pie bicomponent spin pack (manufactured by Hills Inc., Melbourne, Fla.) which distributed the polymer melts such that the final combined melt stream was in the form of 2222 bicomponent fibers having the segmented pie polymer distribution. The lobes contained the PET component while the area between these lobes is comprised of the water dispersible sulfopolyester co-extruded with the PET during this fiber spinning process. The spinneret in this spin pack was constructed with 2222 die holes each having a diameter of 0.35 millimeters.

An aspirator assembly using compressed air was utilized to form a high velocity downward flow stream which drew the extruded polymer melt streams exiting the spinneret into fine multicomponent fibers. In the present example, sufficient air pressure was applied to draw the multicomponent fibers to a fineness of about 1.0 to about 1.5 denier per filament (dpf) or nominal filament diameters of about 8 to about 12 microns.

The drawn fibers were laid down on a collection belt to form a continuous nonwoven web where the density (basis weight) of the nonwoven web was controlled by the speed of the belt and the extrusion rate. Nonwoven webs having a typical basis weight of about 100 to about 150 grams/sq. meter or as specified were produced in the examples.

A compacting roll and wind up station was utilized to produce rolls of the lightly compacted nonwoven webs of multicomponent fibers with the segmented pie fiber cross-section structure. The compaction of the nonwoven web was conducted using calendar rolls at room temperature with sufficient nip pressure to consolidate the nonwoven web so it could be handled on a roll and unwound for further treatment. The nonwoven web produced by the preceding process was subsequently hydroentangled using a pilot scale hydroentangling line (manufactured by Fleissner, Engelsbach, Germany) having five hydroentangling heads. The hydroentangling equipment was utilized in the examples using different number of hydroentangling heads and pressures as specified in the examples. The hydroentangling equipment had three high pressure hydroentangling heads which contacted the nonwoven web as it passed under the water jets supported by a 103 mesh screen belt. The first head operated as a pre-wetting station using only about 30 bar water pressure while hydroentangling heads 2 and 3 were operated at a typical water pressure ranging from about 100 to about 200 bar. The water jets of the final two hydroentangling heads impacted the nonwoven web as it was conveyed on a hydroentangling drum with a porous surface capable of drawing the water from the hydroentangling jets into the drum using vacuum within the drum. These final two hydroentangling jets were operated at about 200 bar water pressure, and the jets contacted the side of the fabric opposite the side contacted by the first three hydroentangling jets.

In each hydroentangling head, a jet strip was inserted which consisted of a line of finely machined holes having a diameter of about 120 microns with a separation between holes of about 0.8 mm. The water pressure of about 200 bar on one side of the jet strip inside the hydroentangling head caused a high velocity jet of water to be formed in each hole. These high velocity water jets caused the multicomponent fibers in the nonwoven web to entangle as the web passes under the hydroentangling jets. In operation about 1200 water jets are applied per meter of nonwoven web width and typically the water flow through a jet strip is about 150 liters/minute or about 0.12 liter/min. of water flowing through each individual hole in the jet strip. During the hydroentangling process, the nonwoven web was passed under the hydroentangling jets at a speed ranging from about 10 meters/min. to about 50 meters/min. The fabric speed can be adjusted to vary the degree of hydroentangling treatment. The temperature of the water during the hydroentangling process was about 30°C-40°C C. and no treatment of the process water such as deionization or demineralization was performed. The hydroentangling process water used in these examples was inherently low in metal ion content. In practice, demineralization of the water used in the hydroentangling operation is desirable.

These process details are general and apply to the specific pilot equipment available for production of the fabrics of the following examples. The conditions described are not meant to be limiting, rather to serve to demonstrate how the materials described in the examples could be produced using other types of nonwoven fabric equipment than the specific equipment used in the examples.

Example 1

Preparation of the Nonwoven Web

A nonwoven web comprised of bicomponent fibers having a segmented pie structure with 16 segments was prepared according to the procedure previously described. The non water dispersible component of the bicomponent fibers was IS3HC grade of PET polyester with inherent viscosity of about 0.53 (Eastman Chemical Company, Kingsport, Tenn., U.S.A.) and extruded along with a water dispersible sulfopolyester as the second component. The water dispersible sulfopolyester had designation SP051 Lot TP06038931 (Eastman Chemical Company) and exhibited a melt viscosity of about 3000 poise as measured at 240°C C. and 1 rad/sec shear rate.

Both polymers were extruded through a segmented pie bicomponent spin pack (Hills Inc., Melbourne Fla.) with a spinneret having 2222 holes at a total extrusion rate through each hole of about 0.6 grams/hole-min (g/hm) with the two polymers in a 70 (PET) to 30 (sulfopolyester) weight ratio. The molten fiber extrudates were drawn using an aspirator assembly to form fibers with an average fiber diameter of about 9 microns having the desired segmented pie polymer distribution. The extruded multicomponent fibers were laid down onto a forming belt to produce a nonwoven web having a basis weight of 135 grams/sq. meter (gsm).
[0145] The nonwoven web of Example 1 was consolidated by pressing between calendar rolls at room temperature. The multicomponent fibers were melt drawn at an estimated speed in excess of about 5000 meters/min during this process. When the nonwoven web of Example 1 was conditioned unrestricted in an oven at 120°C, the nonwoven web shrank to 60±5% of the starting dimensions in the MD (machine direction) and CD (cross direction) respectively due to stress relaxation of the multicomponent fibers.

[0146] This nonwoven web had minimal inter-fiber bonding and served as the precursor for producing hydroentangled nonwoven fabrics by subsequent processing of this nonwoven web of Example 1.

Example 2

[0147] The nonwoven web of Example 1 was hydroentangled with high velocity water jets using a hydroentangling unit described previously. During the hydroentangling process, the fabric passed through the machine at a 40 meters/min speed where the nonwoven web was exposed to two high pressure hydroentangling jets operating with 150 bar water pressure. The first jet was applied to the nonwoven web and was transported on a 103 mesh screen belt to apply a fine mesh pattern to the nonwoven web. The second hydroentangling jet was applied to the opposite side of the web as it was supported on a hydroentangling drum operating under vacuum to yield the nonwoven fabric.

[0148] In the final step of the hydroentangling process, the wet nonwoven fabric was passed through a drum dryer where 70°C air passed through the nonwoven fabric by the action of vacuum maintained within the 1.4 meter diameter drum. In the process, the nonwoven fabric was only partly dried when heated in the oven and exited in the wet state. The residence time of the nonwoven fabric in the oven was measured to be about 5 seconds. As the nonwoven fabric was wound onto a core, some excess water was squeezed out, and it was noted that the water was cloudy due to the presence of emulsified sulfopolyester and also formed a sticky film when dried. Both observations indicated that some of sulfopolyester was emulsified during this drying step.

[0149] The energy expended during the hydroentangling process is inversely proportional to the speed of the nonwoven fabric passing under the jets where slower speed translates to increased hydroentangling energy applied to the nonwoven fabric. Similarly, the energy applied is directly proportional to the number of hydroentangling jet stations under which the nonwoven fabric passes where the hydroentangling energy applied by each jet station is proportional to the square root of the water pressure. Using these relationships, the relative amount of hydroentangling energy applied to a nonwoven web can be approximated for comparison. In the present case of Example 2, the hydroentangling energy is arbitrarily assigned a value of 1.0, where in subsequent examples the value will represent the increase in hydroentangling energy applied to the nonwoven web relative to the process in Example 2.

[0150] The basis weight of the hydroentangled nonwoven fabric of Example 2 was 139 gsm (on dry basis), which was similar to the value of the starting nonwoven web and indicated that none or only minimal sulfopolyester was removed during hydroentangling.

[0151] The wet nonwoven fabric of Example 2 was rinsed 3 times in ambient temperature deionized water to remove the sulfopolyester emulsified in the water phase soaked in the nonwoven fabric. After rinsing and drying, a soft nonwoven fabric was recovered. The nonwoven fabric recovered was very loose, exhibited limited strength, and had a fuzzy texture due to loose fiberson fabric surface. The nonwoven fabric had limited utility as a textile fabric. The weight loss during rinsing steps was 29% indicating about full removal of water dispersible sulfopolyester from the nonwoven fabric.

[0152] This nonwoven fabric of Example 2 had limited utility as a finished product but consisted of a nonwoven web of microfibers where the sulfopolyester was removed from the multicomponent fibers to expose the PET microfibers. This precursor nonwoven fabric of Example 2 was further processed to make nonwoven fabrics of subsequent examples 5 and 7.

Example 3

[0153] The nonwoven web of Example 1 was hydroentangled with high velocity water jets using a hydroentangling unit described previously. During the hydroentangling process, the nonwoven web passed through the unit at 10 meters/min speed where the nonwoven web was exposed to four high pressure hydroentangling jets operating with jet pressures of 100 bar, 150 bar, 200 bar, and 200 bar, respectively. The first two jets were applied to the nonwoven web as it was transported on a 103 mesh screen belt to apply a fine mesh pattern to the nonwoven web. The third and fourth hydroentangling jets were applied to the opposite side of the nonwoven web as it was supported on a hydroentangling drum operating under vacuum to produce the nonwoven fabric.

[0154] In the final step of the hydroentangling process, the wet nonwoven fabric was passed through a drum dryer where 70°C air passed through the nonwoven fabric by the action of vacuum maintained within the 1.4 meter diameter drum. In the process, the nonwoven fabric was only partly dried when heated in the oven and exited in the wet state. The residence time of the nonwoven fabric in the oven was measured to be about 18 seconds. As the nonwoven fabric was wound, some excess water was squeezed out, and it was noted that the water was cloudy due to the presence of emulsified sulfopolyester. The emulsified sulfopolyester also formed a sticky film on the nonwoven fabric when dried.

[0155] The energy expended during the hydroentangling process is inversely proportional to the speed of the nonwoven fabric passing under the jets where slower speed translates to increased hydroentangling energy applied to the nonwoven fabric. Similarly, the energy applied is directly proportional to the number of hydroentangling jet stations under which the nonwoven fabric passes where the hydroentangling energy applied by each jet station is proportional to the square root of the water pressure. Using these relationships, the relative amount of hydroentangling energy applied to this nonwoven web of Example 3 relative to Example 2 was calculated for comparison. In the present case, the hydroentangling energy amounted to a value 8.3 times larger than the base value assigned to Example 2. The higher degree of hydroentanglement in Example 3 is due to 2 times the number of hydroentangling jets used coupled with a ¼ process line speed which provided 4 times the residence time under the hydroentangling jets.

[0156] The basis weight of the hydroentangled nonwoven fabric of Example 3 was 136 gsm, which was similar to the value of the starting nonwoven web of Example 1 and indicated that none or only minimal amount of sulfopolyester was removed during hydroentangling.
The wet nonwoven fabric of Example 3 was rinsed 3 times in ambient temperature deionized water to remove any sulfopolyester emulsified in the water phase soaked in the nonwoven fabric. After rinsing and drying, a soft nonwoven fabric was recovered. A weight loss of about 28% was observed indicating removal of essentially all of sulfopolyester from the nonwoven fabric. This nonwoven fabric was very flexible, exhibited good strength, but had a fuzzy surface texture due to loose surface fibers. This precursor fabric of Example 3 was used for further processing to make fabrics of Examples 6 and 9.

Example 4

The wet nonwoven fabric of Example 2 was passed through a hydroentangling unit described previously using minimal water contact in order to remove the sulfopolyester from the nonwoven fabric without applying much additional entanglement of the multicomponent fibers in the fabric. As noted in Example 2, the sulfopolyester in the nonwoven fabric was found to be present in the absorbed water phase rather than the multicomponent fibers, and a simple water rinse was found to remove the sulfopolyester from the nonwoven fabric. The nonwoven fabric passed through the hydroentangling unit at a 50 meters/min speed where the nonwoven fabric was exposed to a single high pressure hydroentangling jets operating at 100 bar pressure. The temperature of the water during the process was about 20°C.

The fabric of Example 4 was dried at ambient temperatures and subsequently washed at 50°C in deionized water to calculate the amount of residual sulfopolyester in the nonwoven fabric that was not removed by the single water jet of the process of Example 4. Only a 3.0% weight loss was measured after washing the nonwoven fabric of Example 4 indicating that about 90% by weight of the sulfopolyester in the nonwoven fabric of Example 2 was removed by this single water head hydroentangling step. The single water head was effective for removing the bulk of sulfopolyester in the nonwoven fabric and removal would have been more efficient if the temperature was higher, preferably in the range of about 40°C to about 50°C for sulfopolyester removal.

Example 5

The precursor nonwoven fabric of Example 2 was further hydroentangled with high velocity water jets using the hydroentangling unit described previously. During this subsequent second hydroentangling step, the precursor nonwoven fabric of Example 2 was passed through the hydroentangling unit at a speed of 10 meters/min and was exposed to four high pressure hydroentangling jets operating with jet pressures of 130 bar, 150 bar, 200 bar, and 200 bar respectively. The first two jets were applied to the nonwoven fabric as it was transported on a 61 mesh screen belt obtained from Asten Johnson in Charleston, S.C. to apply a mesh pattern to the nonwoven fabric. The third and fourth hydroentangling jets were applied to the opposite side of the nonwoven fabric as it was supported on a hydroentangling drum operating under vacuum.

Using the relationships previously described, the relative amount of hydroentangling energy applied to this nonwoven fabric in the process of Example 5 relative to the process of Example 2 was calculated for comparison. The hydroentangling energy in Example 5 amounts to a value 8.5 times larger than the base value of 1 assigned to Example 2. The overall hydroentangling energy applied to the nonwoven fabric in the combined process of Example 2 and 5 amounts to a value about 9.5 times the hydroentangling energy applied in the process of Example 2 alone. This fabric exhibited very good fiber entanglement with no loose fibers on the surface. This fabric exhibited good smooth surface texture.

Example 6

The precursor nonwoven fabric of Example 3 was further hydroentangled with high velocity water jets using the hydroentangling unit. During this second hydroentangling step, the nonwoven fabric passed through the hydroentangling unit at a speed of 10 meters/min where the nonwoven fabric was exposed to four high pressure hydroentangling jets operating with jet pressures of 130 bar, 150 bar, 200 bar, and 200 bar, respectively. The first two jets were applied to the nonwoven fabric as it was transported on a Formtec 14 (14 mesh screen) belt obtained from Albany International in Albany, N.Y. to apply a coarse mesh pattern to the nonwoven fabric. The difference between the fabric of Example 5 and the fabric of Example 7 is the forming belt used, where the belt in Example 7 conferred a coarser basket weave appearance to the fabric surface compared to the fabric of Example 5. The third and fourth hydroentangling jets were applied to the opposite side of the nonwoven fabric as it was supported on a hydroentangling drum operating under vacuum.

Using the relationships previously described, the relative amount of hydroentangling energy applied to this nonwoven fabric in the process of Example 7 relative to the process of Example 2 was calculated for comparison. The hydroentangling energy in Example 7 amounts to a value 8.5 times larger than the arbitrary value of 1 assigned to Example 2. The overall hydroentangling energy applied to the non-
woven fabric in the combined process of Example 2 and 7 amounts to a value about 9.5 times the hydroentangling energy applied in the process of Example 2 alone. This fabric exhibited very good fiber entanglement with no loose fibers on the surface. This fabric exhibited good smooth surface texture.

Example 8

[0166] The nonwoven fabric of Example 5 was heat set by placing pieces of fabric on a restraining frame and conditioning the nonwoven fabric in a forced air oven at 130°C for 5 minutes conditioning time. The restraining frame was a 14 inch x 14 inch frame with pins around the periphery of the frame, spaced with ½ inch separation between pins. The restraining action of the pins prevented the nonwoven fabric from shrinking when heated. The conditioning at elevated temperature causes stress relaxation in the fibers.

[0167] This fabric exhibited very good fiber entanglement with no loose fibers on the surface. The fabric exhibited good smooth surface texture.

Example 9

[0168] The fabric of Example 6 was heat set by placing pieces of nonwoven fabric on a restraining frame and conditioning the nonwoven fabric in a forced air oven at 130°C for 5 minutes conditioning time. The restraining frame was a 14 inch x 14 inch frame with pins around the periphery of the frame, spaced with ½ inch separation between pins. The restraining action of the pins prevented the nonwoven fabric from shrinking when heated. The conditioning at elevated temperature causes stress relaxation in the fibers.

[0169] This fabric exhibited very good fiber entanglement with no loose fibers on the surface. This fabric exhibited good smooth surface texture.

Example 10

[0170] The nonwoven fabric of Example 7 was heat set by placing pieces of nonwoven fabric on a restraining frame and conditioning the nonwoven fabric in a forced air oven at 130°C for 5 minutes conditioning time. The restraining frame was a 14 inch x 14 inch frame with pins around the periphery of the frame, spaced with ½ inch separation between pins. The restraining action of the pins prevented the nonwoven fabric from shrinking when heated. The conditioning at elevated temperature causes stress relaxation in the fibers.

[0171] This fabric exhibited very good fiber entanglement with no loose fibers on the surface. This fabric exhibited good smooth surface texture.

Comparative Example 1

[0172] The fabric of Example 1 was hydroentangled with high velocity water jets using the hydroentangling unit described previously. During the hydroentangling process the fabric passed through the machine at a 10 meters/min. speed where the nonwoven web was exposed to four high pressure hydroentangling jets operating with jet pressures of 100 bar, 150 bar, 200 bar, and 200 bar respectively. The first two jets were applied to the fabric as it was transferred on a 103 mesh screen belt to apply a fine mesh pattern to the fabric. The third and fourth hydroentangling jets were applied to the opposite side of the web as it was supported on a hydroentangling drum operating under vacuum.

[0173] In the final step of the hydroentangling process the wet fabric was passed through a drum dryer where 70°C air passed through the fabric by the action of vacuum maintained within the 1.4 meter diameter drum. In the process the fabric was only partly dried when heated in the oven and exited in the wet state. The residence time of the fabric in the oven was measured to be about 18 seconds. As the fabric was wound onto a core some excess water was squeezed out and it was noted that the water was cloudy indicating some emulsification of sulfopolyester.

[0174] The fabric formed by this hydroentangling step was rinsed in both in deionized water at 45°C and in a home washing machine using a hot water (48°C) cycle. The fabric had a highly wrinkled appearance with a large number of loose fibers on the surface. The fabric was very "tacky" or "clingy" due to the loose nature of the surface fibers in the fabric and not desirable for textile applications.

Comparative Example 2

[0175] The nonwoven fabric of Comparative Example 1 was heat set by placing pieces of fabric on a restraining frame and conditioning the nonwoven fabric in a forced air oven at 130°C for 5 minutes conditioning time. The restraining frame was a 14 inch x 14 inch frame with pins around the periphery of the frame, spaced with ½ inch separation between pins. The restraining action of the pins prevented the nonwoven fabric from shrinking when heated. The conditioning at elevated temperature causes stress relaxation in the fibers.

[0176] This fabric exhibited loose fibers on the surface and had "tacky" or "clingy" feel due to the excessive amount of loose fibers on the surface.

We claim:

1. A process for making a nonwoven fabric comprising:
   (A) collecting multicomponent fibers to form a non-woven web; wherein said multicomponent fiber comprises at least one water dispersible sulfopolyester and at least one water non-dispersible polymer; wherein said multicomponent fiber has a plurality of domains comprising said water non-dispersible polymer; wherein said domains are substantially isolated from each other by said water dispersible sulfopolyester intervening between said domains;
   (B) contacting said non-woven web with water at a sufficient temperature and pressure to remove a portion of said water dispersible sulfopolyester thereby forming a microfiber web; and
   (C) hydroentangling said microfiber web to produce said nonwoven fabric.

2. A process according to claim 1 wherein said sulfopolyester comprises dicarboxylic acid monomer residues, sulfonmonomer residues, diol monomer residues, and repeating units.

3. A process according to claim 2 wherein said dicarboxylic acids are selected from aliphatic diacids, cycloaliphatic dicarboxylic acids, aromatic dicarboxylic acids, and combinations thereof.

4. A process according to claim 3 wherein said dicarboxylic acids are selected from succinic, glutaric, adipic, azelaic, sebacic, fumaric, maleic, itaconic, 1,3-cyclohexane dicarboxylic, 1,4-cyclohexanedimdicarboxylic, diglycolic, 2,5-norbornanedicarboxylic, phthalic, terephthalic, 1,4-naphthalenedicarboxylic, 2,5-naphthalenedicarboxylic, 2,6-naphthalenedicarboxylic, 2,7-naphthalenedicarboxylic,
diphenic, 4,4'-oxydibenzoic, 4,4'-sulfonyldibenzoic, isophthalic, and combinations thereof.

5. A process according to claim 2 wherein said sulfonamide is a metal sulfonate salt of a sulfophthalic acid, sulfoterephthalic acid, sulfosioisophthalic acid, or combinations thereof.

6. A process according to claim 2 wherein said diol residues are selected from the group consisting of ethylene glycol, diethylene glycol, triethylene glycol, poly(ethylene) glycols, 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, 1,3-butanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 2,2,4,4-trimethyl-1,6-hexanediol, 1,3-cyclohexanediol, 1,2-cyclohexanediethanol, 1,4-cyclohexane-dimethanol, 2,2,4,4-tetramethyl-1,3-cyclobutanediol, p-xylene1ediol, and combinations thereof.

7. A process according to claim 2 wherein said sulfopolyester further comprises at least one branching monomer.

8. A process according to claim 7 wherein said branching monomer is at least one selected from the group consisting of 1,1,1-trimethylol propane, 1,1,1-trimethylol ethane, glycerin, pentaerythritol, erythritol, xylitol, dipentaerythritol, sorbitol, trimellitic anhydride, pyromellitic dianhydride, dimethylol propionic acid, or combinations thereof.

9. A process according to claim 1 wherein said water-dispersible polymers are selected from polyolefins, polysteres, polyanilides, polylecctides, polycaprolactone, polycarbonate, polyurethane, polyvinyl chloride, and combinations thereof.

10. A process according to claim 1 wherein the shaped cross section of said multicomponent fiber is an island-in-the-sea, segmented pie, or shell-core configuration.

11. A process according to claim 1 wherein said sulfopolyester has a glass transition temperature (Tg) of at least 57°C, and said water dispersible sulfopolyester comprises:

(i) residues of one or more dicarboxylic acids;
(ii) about 4 to about 40 mole %, based on the total repeating units, of residues of at least one sulfonamide having 2 functional groups and one or more sulfonate groups attached to an aromatic or cycloaliphatic ring wherein said functional groups are hydroxyl, carbonyl, or a combination thereof;
(iii) one or more diol residues wherein at least 25 mole %, based on the total diol residues, is a poly(ethylene glycol) having a structure

\[ -\text{HO}-\overset{\cdot}{\text{O}}\text{CH}_{2}-\text{CH}_{2}\text{-}\overset{\cdot}{\text{O}}\text{H} \]

wherein \( n \) is an integer in the range of 2 to about 500; and
(iv) 0 to about 25 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein said functional groups are hydroxyl, carbonyl, or a combination thereof;

wherein said fiber has a plurality of segments comprising said water-nondispersible polymers and said segments are substantially isolated from each other by said sulfopolyester intervening between said segments.

12. A process according to claim 1 wherein said sulfopolyester has a melt viscosity of less than 12,000 poise measured at 240°C and 1 rad/sec shear rate.

13. A process according to claim 12 wherein said sulfopolyester has a melt viscosity less than 6,000 poise measured at 240°C and 1 rad/sec shear rate.

14. A process according to claim 13 wherein said sulfopolyester has a melt viscosity less than 4,000 poise measured at 240°C and 1 rad/sec shear rate.

15. A process according to claim 1 wherein said collecting is accomplished by at least one method selected from the group consisting of mechanical needling, chemical binding, thermally calendaring, ultrasonic fusing, and hydroentangling.

16. A process according to claim 1 wherein said collecting is accomplished by hydroentangling and the hydroentangling energy in said hydroentangling step involves about 20% to about 50% of the amount of hydroentangling energy expended in Step (C).

17. A process according to claim 1 wherein said multicomponent fibers are produced by a spunbond process or meltblown process.

18. A process according to claim 1 wherein the weight of said nonwoven web ranges from about 10 grams/m² to about 800 grams/m².

19. A process according to claim 18 wherein the weight of said nonwoven web ranges from about 10 grams/m² to about 400 grams/m².

20. A process according to claim 19 wherein the weight of said nonwoven web ranges from about 50 grams/m² to about 150 grams/m².

21. A process according to claim 1 wherein the extraction temperature of said water dispersible sulfopolyester ranges from about 20°C to about 100°C.

22. A process according to claim 1 wherein the pressure of the water during Step (B) ranges from about 30 bar to about 600 bar.

23. A process according to claim 22 wherein the pressure of the water during Step (B) ranges from about 50 bar to about 300 bar.

24. A process according to claim 24 wherein said nonwoven web is contacted with the water in Step (B) for a time sufficient to remove about 30% by weight to about 100% by weight of the total water dispersible sulfopolyester contained in said nonwoven web.

25. A process according to claim 24 wherein said nonwoven web is contacted with the water in Step (B) for a time sufficient to remove greater than 90% by weight of the total water dispersible sulfopolyester from said nonwoven web.

26. A process according to claim 25 wherein said nonwoven web is contacted with the water in Step (B) for a time sufficient to remove greater than 95% by weight of the total water dispersible sulfopolyester from said nonwoven web.

27. A process according to claim 26 wherein said nonwoven web is contacted with water for a time period of from about 10 to about 600 seconds whereby said water dispersible sulfopolyester is dissipated or dissolved.

28. A process according to claim 1 wherein the microfibers in the microfiber web have an average fineness of less than 30% of the denier of said multicomponent fiber.

29. A process according to claim 28 wherein said microfibers in said microfiber web have an average fineness of 1 dpf or less.

30. A process according to claim 29 wherein said microfibers in said microfiber web have an average fineness of 0.5 dpf or less.

31. A process according to claim 1 wherein said nonwoven web is contacted with water in Step (B) using water jets.

32. A process according to claim 31 wherein one to eight water jet heads are utilized.
33. A process according to claim 31 wherein the amount of water used in Step (B) ranges from about 500 times to about 1000 times the weight of said nonwoven web.

34. A process according to claim 3 wherein the wash water from Step (B) and Step (C) can be reclaimed.

35. A process according to claim 34 wherein 80% or more of the wash water is reclaimed.

36. A process according to claim 1 wherein the temperature of the water in Step (C) is less than 40°C.

37. A process according to claim 1 wherein the pressure of the water in Step (C) ranges from about 150 bar to about 250 bar.

38. A process according to claim 1 wherein less than 1% by weight of the water dispersible sulfopolyester in Step (C) is removed from said microfiber web.

39. A process according to claim 38 wherein in Step (C) less than 0.5% by weight of the water dispersible sulfopolyester is removed from the microfiber web.

40. A process according to claim 38 wherein in Step (C) less than 0.1% by weight of the water dispersible sulfopolyester is removed from the microfiber web.

41. A process according to claim 1 wherein said hydroentangling in Step (C) is conducted by water jet heads.

42. A process according to claim 41 wherein number of water jet heads ranges from 1 to about 20.

43. A process for making a nonwoven fabric comprising:
   (A) collecting multicomponent fibers to form a non-woven web wherein said multicomponent fiber comprises at least one water dispersible sulfopolyester and at least one water non-dispersible polymer wherein said multicomponent fiber has a plurality of domains comprising said water non-dispersible polymer wherein said domains are substantially isolated from each other by said water dispersible sulfopolyester between said domains; and
   (B) contacting said non-woven web with water at a sufficient temperature and pressure to remove a portion of said sulfopolyester thereby forming a microfibers and simultaneously hydroentangling said microfibers to produce said nonwoven fabric.

44. A process according to claim 43 wherein said sulfopolyester comprises dicarboxylic acid monomer residues, sulfonmonomer residues, diol monomer residues, and repeating units.

45. A process according to claim 44 wherein said dicarboxylic acids are selected from aliphatic dicacids, cycloaliphatic dicarboxylic acids, aromatic dicarboxylic acids, and combinations thereof.

46. A process according to claim 45 wherein said dicarboxylic acids are selected from succinic, glutaric, adipic, azelaic, sebacic, fumaric, maleic, itaconic, 1,3-cyclohexane dicarboxylic, 1,4-cyclohexanedicarboxylic, diglycolic, 2,5-norbornanedicarboxylic, phthalic, terephthalic, 1,4-naphthalenedicarboxylic, 2,5-naphthalenedicarboxylic, 2,6-naphthalenedicarboxylic, 2,7-naphthalenedicarboxylic, diphenic, 4,4'-oxydibenzonic, 4,4'-sulfonyldibenzonic, isophthalic, and combinations thereof.

47. A process according to claim 44 wherein said sulfonmonomer is a metal sulfonate salt of a sulfophthalic acid, sulfterephthalic acid, sulfosulfonic acid, or combinations thereof.

48. A process according to claim 44 wherein said diol residues are selected from the group consisting of ethylene glycol, diethylene glycol, triethylene glycol, poly(ethylene) glycols, 1,3-propanediol, 2,4-dimethyl-2-ethylhexane-1,3-diol, 2,2-dimethyl-1,3-propanediol, 2-ethyl-2-butyl-1,3-propanediol, 2-ethyl-2-isobutyl-1,3-propanediol, 1,3-butanediol, 1,4-butanediol, 1,5-pentanediol, 1,6-hexanediol, 2,2,4-trimethyl-1,6-hexanediol, thiodiethanol, 1,2-cyclohexanediol, 1,3-cyclohexanediol, 1,4-cyclohexane-dimethanol, 2,2,4,4-tetramethyl-1,3-cyclobutanediol, p-xylene, and combinations thereof.

49. A process according to claim 44 wherein said sulfopolyester further comprises at least one branching monomer.

50. A process according to claim 49 wherein said branching monomer is at least one selected from the group consisting of 1,1,1-trimethylol propane, 1,1,1-trimethylolethane, glycerin, pentaerythritol, erythritol, threitol, dipentaerythritol, sorbitol, trimellitic anhydride, pyromellitic dianhydride, dimethylol propionic acid, or combinations thereof.

51. A process according to claim 43 wherein said water-nondispensible polymers are selected from polyolefins, polyesters, poliamides, poly lactides, polycaprolactone, polycarbonate, polyurethane, polyvinyl chloride, and combinations thereof.

52. A process according to claim 43 wherein the shaped cross section of said multicomponent fiber is an islands-in-the-sea, segmented pie, or sheath-core configuration.

53. A process according to claim 43 wherein said sulfopolyester has a glass transition temperature (Tg) of at least 57°C and said water dispersible sulfopolyester comprises:
   (i) residues of one or more dicarboxylic acids;
   (ii) about 4 to about 40 mole %, based on the total repeating units, of residues of at least one sulfonmonomer having 2 functional groups and one or more sulfonate groups attached to an aromatic or cycloaliphatic ring wherein said functional groups are hydroxyl, carboxyl, or a combination thereof;
   (iii) one or more diol residues wherein at least 25 mole %, based on the total diol residues, is a poly(ethylene glycol) having a structure

\[
\text{H} \rightarrow \left(\text{OCH}_{2}-\text{CH}_{2}\right)_{n} \rightarrow \text{OH}
\]

wherein \(n\) is an integer in the range of 2 to about 500; and
(iv) 0 to about 25 mole %, based on the total repeating units, of residues of a branching monomer having 3 or more functional groups wherein said functional groups are hydroxyl, carboxyl, or a combination thereof;

wherein said fiber has a plurality of segments comprising said water-nondispensible polymers and said segments are substantially isolated from each other by said sulfopolyester intervening between said segments.

54. A process according to claim 43 wherein said sulfopolyester has a melt viscosity of less than 12,000 poise measured at 240°C and 1 rad/sec shear rate.

55. A process according to claim 54 wherein said sulfopolyester has a melt viscosity of less than 6,000 poise measured at 240°C and 1 rad/sec shear rate.

56. A process according to claim 56 wherein said sulfopolyester has a melt viscosity of less than 4,000 poise measured at 240°C and 1 rad/sec shear rate.

57. A process according to claim 43 wherein said collecting is accomplished by at least one method selected from the group consisting of mechanical needling, chemical binding, thermally calendaring, ultrasonic fusing, and hydroentangling.
58. A process according to claim 57 wherein said collecting is accomplished by hydroentangling and the hydroentangling energy in said hydroentangling step involves about 20% to about 50% of the amount of hydroentangling energy expended in Step (C).

59. A process according to claim 43 wherein said multicomponent fibers are produced by a spunbond process or meltblown process.

60. A process according to claim 43 wherein the weight of said nonwoven web ranges from about 10 grams/m² to about 800 grams/m².

61. A process according to claim 60 wherein the weight of said nonwoven web ranges from about 10 grams/m² to about 400 grams/m².

62. A process according to claim 61 wherein the weight of said nonwoven web ranges from about 50 grams/m² to about 150 grams/m².

63. A process according to claim 43 wherein the extraction temperature of said water dispersible sulfopolyester ranges from about 20°C to about 100°C.

64. A process according to claim 43 wherein the pressure of the water during Step (B) ranges from about 30 bar to about 600 bar.

65. A process according to claim 64 wherein the pressure of the water during Step (B) ranges from about 50 bar to about 300 bar.

66. A process according to claim 43 wherein said nonwoven web is contacted with the water in Step (B) for a time sufficient to remove about 50% by weight to about 100% by weight of the total water dispersible sulfopolyester contained in said nonwoven web.

67. A process according to claim 66 wherein said nonwoven web is contacted with the water in Step (B) for a time sufficient to remove greater than 90% by weight of the total water dispersible sulfopolyester from said nonwoven web.

68. A process according to claim 67 wherein said nonwoven web is contacted with the water in Step (B) for a time sufficient to remove greater than 95% by weight of the total water dispersible sulfopolyester from said nonwoven web.

69. A process according to claim 43 wherein said nonwoven web is contacted with water for a time period of from about 10 to about 600 seconds whereby said water dispersible sulfopolyester is dissipated or dissolved.

70. A process according to claim 43 wherein the microfibers in the microfiber web have an average fineness of less than 30% of the denier of said multicomponent fiber.

71. A process according to claim 70 wherein said microfibers in said microfiber web have an average fineness of 1 dpf or less.

72. A process according to claim 71 wherein said microfibers in said microfiber web have an average fineness of 0.5 dpf or less.

73. A process according to claim 43 wherein said nonwoven web is contacted with water in Step (B) using water jets.

74. A process according to claim 73 wherein one to eight water jet heads are utilized.

75. A process according to claim 74 wherein the amount of water used in Step (B) ranges from about 500 times to about 1000 times the weight of said nonwoven web.

76. A process according to claim 43 wherein said water is used in Step (B) and Step (C) can be reclaimed.

77. A process according to claim 76 wherein 80% or more of the water is reclaimed.

78. A process according to claim 43 wherein the temperature of the water in Step (C) is less than 40°C.

79. A process according to claim 43 wherein the temperature of the water in Step (C) ranges from about 150 bar to about 250 bar.

80. A process according to claim 43 wherein less than 1% by weight of the water dispersible sulfopolyester in Step (C) is removed from said microfiber web.

81. A process according to claim 80 wherein in Step (C) less than 0.5% by weight of the water dispersible sulfopolyester is removed from the microfiber web.

82. A process according to claim 81 wherein in Step (C) less than 0.1% by weight of the water dispersible sulfopolyester is removed from the microfiber web.

83. A process according to claim 43 wherein said hydroentangling in Step (C) is conducted by water jet heads.

84. A process according to claim 83 wherein number of water jet heads range from 1 to about 20.

85. A process according to claim 1 or 43 wherein said nonwoven fabric is heat set.

86. A nonwoven fabric produced by the process of claim 1 or claim 43 wherein said nonwoven fabric is utilized in at least one of the following end uses: clothing, curtains, upholstery, uniforms, personal care products, hospital/surgical and other medical disposables, multilayer nonwovens, laminates and composites, protective fabrics and layers, geotextiles, industrial wipes, and filter media.

* * * * *