

(12) STANDARD PATENT
(19) AUSTRALIAN PATENT OFFICE

(11) Application No. **AU 2015305567 B2**

(54) Title
Synthetic fibers with enhanced stain resistance and methods of making the same

(51) International Patent Classification(s)
C08L 23/04 (2006.01)

(21) Application No: **2015305567** (22) Date of Filing: **2015.08.19**

(87) WIPO No: **WO16/028840**

(30) Priority Data

| | | |
|-------------------|-------------------|--------------|
| (31) Number | (32) Date | (33) Country |
| 62/039,637 | 2014.08.20 | US |

(43) Publication Date: **2016.02.25**

(44) Accepted Journal Date: **2019.10.10**

(71) Applicant(s)
INVISTA Textiles (U.K.) Limited

(72) Inventor(s)
Tung, Wae-Hai; Fielitz, Glenn

(74) Agent / Attorney
Davies Collison Cave Pty Ltd, Level 15 1 Nicholson Street, MELBOURNE, VIC, 3000, AU

(56) Related Art
US 6117550 A



- (51) International Patent Classification:
C08L 23/04 (2006.01)
- (21) International Application Number:
PCT/US2015/045797
- (22) International Filing Date:
19 August 2015 (19.08.2015)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
62/039,637 20 August 2014 (20.08.2014) US
- (71) Applicant (for all designated States except US): **INVISTA TECHNOLOGIES S.A.R.L.** [LU/CH]; Zweigniederlassung St. Gallen, Flurhofstrasse 160, 9000 St. Gallen (CH).
- (71) Applicant (for US only): **INVISTA NORTH AMERICA S.A.R.L.** [LU/US]; Three Little Falls Centre, 2801 Centerville Road, Wilmington, Delaware 19808 (US).
- (72) Inventors: **TUNG, Wae-Hai**; 3412 Stillbrook Way, Marietta, Georgia 30062-1377 (US). **FIELITZ, Glenn**; 63 Shoreline Drive, Columbia, South Carolina 29229 (US).
- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM,

AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))

- (54) Title: SYNTHETIC FIBERS WITH ENHANCED STAIN RESISTANCE AND METHODS OF MAKING THE SAME

- (57) Abstract: Synthetic fibers with enhanced stain resistance, yarns and carpets prepared from these fibers and compounds and methods for their production are provided.

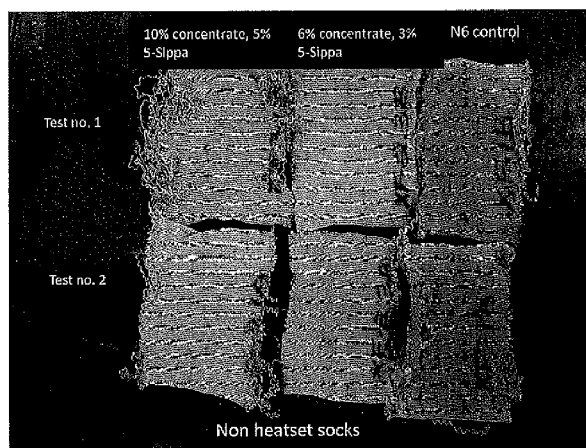


FIGURE 1

Synthetic Fibers with Enhanced Stain Resistance and Methods of Making the Same

Field of Invention

[0001] The present disclosure relates to synthetic fibers with enhanced stain resistance, articles prepared from these fibers and methods for their production.

Background

[0002] Polyamides are used in a variety of applications and often include additives used to modify physical properties including, but not limited to viscosity, tensile strength, fire resistance, stain resistance, flowability, color and texture. Many of these additives can be difficult to work with for various reasons, such as volatility, moisture absorptivity, photosensitivity and thermal sensitivity and subsequent degradation processes, and melt viscosity incompatibility.

Accordingly, use of such additives on a commercial scale can be inefficient, resulting in production of polyamides with inferior physical properties.

[0003] In one application, polyamides are used to form fibers in textiles such as fabrics, carpets and rugs. These fibers oftentimes suffer from a lack of stain resistance, due to chemical interactions of acidic sites in the polyamides and complementary sites in staining compositions. These chemical interactions typically fix stains on a substrate, and make stain removal difficult. Accordingly, it is known in the art to add stain blocking additives to the polyamide composition, in order to increase its stain resistance by a reduction in the chemical interactions, thereby making removal of the stains easier, and more efficient.

[0004] Known stain blocking additives added to polyamides during polymerization include sulfoisophthalic acids, sulfobenzoic acids, and sulfonic acids. However, these agents are typically hygroscopic, oftentimes agglomerate when used on a commercial scale, and can clog industrial reactors thus leading to production inefficiencies, cost increases, and formation of inferior fibers.

[0005] While different approaches such as use of powdered stain blocking agents, spray solutions of stain blocking agents and physical distribution of stain blocking agents via masterbatch compounds in polyamide melts have been developed, each of these approaches still suffers from production inefficiencies and forms fibers that can still be improved.

[0006] Polyesters have commonly been used in the past as the carrier polymer of a masterbatch compound. There are numerous problems, however, when a masterbatch having polyester,

particularly polyethylene terephthalate (PET) as the carrier polymer is combined with polyamides such as polycaprolactam, also known as nylon 6 or N6. First, PET has a melting point (m.p. 252-260° C) which is higher than N6 (m.p. 215-220° C). This difference in polymer melting point and the associated differences in melt phase polymer flow causes process issues that complicate extrusion, transfer of extrudate to a spinneret, fiber formation, and fiber draw. Additionally, above a given use rate, PET will undergo macrophase separation when used with N6, which will cause fibrillation of PET in the fiber. PET is also comparatively expensive when used as a base masterbatch component.

[0007] Alternatively, N6 can be used as a carrier in a masterbatch composition. Should that masterbatch then be used in combination with an N6 polymer base, concerns associated with melting point and polymer melt phase flow would be effectively mitigated, because the base fiber forming polymer and the masterbatch carrier polymer would be the same. However, as is understood by those skilled in the art, N6 requires careful control of its moisture content prior to melt spinning into filament. Inadequate control of moisture content can cause major process issues. For example, a N6 masterbatch with excessive moisture content will cause a drop in the melt viscosity of the polymer melt during melt spinning, thus making it difficult to draw fiber from the extruder. This issue can be resolved by an additional polymer drying, or conditioning step, before the N6 masterbatch is fed to an extruder or by the removal of moisture or other volatiles during the melt spinning process. The removal of moisture or volatiles is taught in a number of patents, including U.S. Pat. No. 6537475 B1, U.S. Pat. No. 6753385 B2, and U.S. Pat. No. 6861480 B2. Additional patents include U.S. Pat. No. 6117550, U.S. Pat. No. 6420044, U.S. Pat. No. 6433107, U.S. Pat. No. 7115224, U.S. Pat. No. 6680018, U.S. Pat. No. 6635346, U.S. Pat. No. 6334877, and U.S. Pat. No. 6589466.

[0008] U.S. Pat. No. 8,501,898, also herein incorporated by reference, discloses a method of adding a masterbatch that includes a stain blocking ingredient, into a reactor during the polymerization of caprolactam monomer to N6. The masterbatch is added into the reactor, prior or during polymerization, for the purpose of incorporating the stain blocking ingredient into N6.

Summary of the Invention

[0009] An aspect of the present disclosure relates to a synthetic fiber comprising a first fiber forming polymer, a second polymer having a melt point that is lower than the melt point of the first fiber forming polymer, and a stain blocking additive.

[0010] In one nonlimiting embodiment, the first fiber forming polymer is a polyamide, a polyester, a polyolefin, or a combination thereof. In another nonlimiting embodiment, the first fiber forming polymer is nylon 6.

[0011] In one nonlimiting embodiment, the second polymer is a polyolefin. In another nonlimiting embodiment the second polymer is an unmodified polyolefin. In one nonlimiting embodiment, the second polymer is polypropylene.

[0012] In one nonlimiting embodiment, the stain blocking additive is an aromatic sulfonate or an alkali metal salt thereof. In another nonlimiting embodiment, the stain blocking additive is 5-sulphoisophthalic acid, sodium salt (NaSIPA).

[0013] Another aspect of the present disclosure relates to yarns formed from these synthetic fibers.

[0014] Another aspect of the present disclosure relates to a fabric knitted from these synthetic fibers and yarns.

[0015] Another aspect of the present disclosure relates to carpet formed from these yarns.

[0016] Another aspect of the present disclosure relates to a process for forming a synthetic fiber with enhanced stain resistance, said process comprising the steps of: a) forming a polymer melt of a first fiber forming polymer and a masterbatch compound, wherein the masterbatch compound comprises a second polymer having a melting point which is less than the melting point of the first fiber forming polymer, and a stain blocking additive, wherein the first fiber forming polymer is present in a range from about 80 to 98 percent by weight, and the masterbatch compound is present in a range from about 2 to 20 percent by weight; wherein the masterbatch compound is not dried or conditioned prior to forming the polymer melt; and b) forming a synthetic fiber with enhanced stain resistance from the polymer melt.

[0017] In one nonlimiting embodiment, the synthetic fiber with enhanced stain resistance is formed from the polymer melt by extruding the melt through a spinneret to form one or a plurality of filaments, quenching the filament or filaments, drawing the filament or filaments, and winding the filament or filaments onto a tube.

[0018] In one nonlimiting embodiment of this process, the masterbatch compound has a moisture content of less than about 200 ppm.

[0019] In one nonlimiting embodiment of this process, the masterbatch compound is not dried or conditioned prior to forming the polymer melt.

[0020] In another nonlimiting embodiment of this process, no additional step is required to remove volatiles while forming the polymer melt.

[0021] In one nonlimiting embodiment of this process, the first fiber forming polymer is a polyamide, polyester, polyolefin or combination thereof. In another nonlimiting embodiment, the first fiber forming polymer is nylon 6.

[0022] In one nonlimiting embodiment of this process, the second polymer is a polyolefin. In another nonlimiting embodiment the second polymer is an unmodified polyolefin. In one nonlimiting embodiment, the second polymer is polypropylene.

[0023] In one nonlimiting embodiment of this process, the stain blocking additive is an aromatic sulfonate or an alkali metal salt thereof. In another nonlimiting embodiment, the stain blocking additive is 5-sulphoisophthalic acid, sodium salt.

[0024] Another aspect of the present disclosure relates to yarns formed from the synthetic fibers produced by this process.

[0025] Another aspect of the present disclosure relates to fabrics knitted from the synthetic fibers and yarns formed from this process.

[0026] Another aspect of the present disclosure relates to carpet formed from the yarns formed from this process.

[0027] Another aspect of the present disclosure relates to a process for forming a nylon 6 fiber with enhanced stain resistance. The process comprises forming a polymer melt from a nylon 6 polymer and a masterbatch compound. The masterbatch compound comprises a second polymer having a melting point that is lower than the melting point of the first fiber forming polymer, and a stain blocking additive. The nylon 6 polymer is present in a range from about 80 to 98 percent by weight, and the masterbatch compound is present in a range from about 2 to 20 percent by weight. A nylon 6 fiber comprising one or a plurality of filaments having enhanced stain resistance is formed from the polymer melt.

[0028] In one nonlimiting embodiment of this process, the masterbatch compound has a moisture content less than about 200 ppm.

[0029] In one nonlimiting embodiment of this process, the masterbatch compound is not dried or conditioned prior to forming the polymer melt.

[0030] In one nonlimiting embodiment of this process, no additional step is required to remove volatiles while forming the polymer melt.

[0031] Another aspect of the present disclosure relates to yarns formed from the nylon 6 fibers produced by this process.

[0032] Another aspect of the present disclosure relates to fabrics knitted from the nylon 6 fibers and yarns formed from this process.

[0033] Another aspect of the present disclosure relates to carpets and rugs formed from the yarns formed from this process.

[0034] Yet another aspect of the present disclosure relates to a masterbatch compound. The masterbatch compound comprises a thermoplastic carrier and a stain blocking additive, and has a moisture content less than about 200 ppm.

[0035] In one nonlimiting embodiment, the thermoplastic carrier is present in the masterbatch compound in a range from about 20 to 80 percent by weight.

[0036] In one nonlimiting embodiment, the stain blocking additive is present in the masterbatch compound in a range from about 20 to 80 percent by weight.

[0037] In one nonlimiting embodiment of this process, the second polymer is a polyolefin. In another nonlimiting embodiment the second polymer is an unmodified polyolefin. In one nonlimiting embodiment, the second polymer is polypropylene.

[0038] In one nonlimiting embodiment, the stain blocking additive in the masterbatch is an aromatic sulfonate or an alkali metal salt thereof. In another nonlimiting embodiment, the stain blocking additive is 5-sulphoisophthalic acid, sodium salt.

Brief Description of the Figures

[0039] FIGURE 1 shows results of a stain test comparing fabrics knitted from synthetic fibers prepared in accordance with Examples 1, 2, 3 and 4.

[0040] FIGURE 2 shows results of a stain test comparing fabrics knitted from synthetic fibers prepared in accordance with Examples 5, 6, 7, 8 and 9.

Detailed Description of the Invention

[0041] Provided by this disclosure are synthetic filaments and fibers with enhanced stain resistance, yarns, fabrics and carpets prepared from these fibers, and methods and masterbatch compounds for their production.

[0042] The synthetic fiber of the present disclosure comprises a first fiber forming polymer. Examples of first fiber forming polymers which can be used include, but are not limited to, polyamides, polyesters, polyolefins and combinations thereof.

[0043] Suitable polyamides include fiber forming polyamides known in the art to be suitable for the formation of bulked continuous filament fibers, having sufficient viscosity, tenacity, chemical stability and crystallinity to be at least moderately durable in such application. The polyamide may be selected from the group consisting of nylon 5,6; nylon 6/6; nylon 6; nylon 7; nylon 11; nylon 12; nylon 6/10; nylon 6/12; nylon DT; nylon 6T; nylon 6I; and blends or copolymers thereof. In one embodiment the polyamide is nylon 6,6 polymer.

[0044] In one embodiment, the first fiber forming polymer comprises is nylon 6.

[0045] Suitable polyolefins include polypropylene. Suitable polyesters include fiber forming polyesters known in the art. The polyester resin may be selected from the group consisting of polyethylene terephthalate, polytrimethylene terephthalate, polybutylene terephthalate, polyethylene naphthalate, polylactic acid (PLA) and blends or copolymers thereof.

[0046] In one nonlimiting embodiment, the first fiber forming polymer is present in the synthetic fiber in a range from about 80 to 98 percent by weight. In another nonlimiting embodiment, the first fiber forming polymer is present in the synthetic fiber in a range from about 80 to 98.8 percent by weight.

[0047] The synthetic fiber of the present disclosure further comprises a second polymer having a melting point that is less than the melting point of the first fiber forming polymer. It is preferred that the presence of the second polymer does not cause the synthetic fiber to fibrillate. Examples of second polymers useful in the present disclosure include, but are not limited to polyolefins, polylactic acid, polystyrene, or a blend or copolymer thereof. In one nonlimiting embodiment, the polyolefin is an unmodified polyolefin. In another nonlimiting embodiment, the second polymer is polypropylene.

[0048] In one nonlimiting embodiment, the second polymer is present in the synthetic fiber in a range from about 1 to about 10 percent by weight. In another nonlimiting embodiment, the second polymer is present in the synthetic fiber from about 1 to about 5 percent by weight. In another nonlimiting embodiment, the second polymer is present in the synthetic fiber from about 0.1 to about 10 percent by weight.

[0049] The synthetic fiber of the present disclosure further comprises a stain blocking additive. Suitable stain blocking additives include those that disable acid dye sites. For example, in polyamides, such as Nylon 6,6 or Nylon 6, acid dyes sites refer to amine end groups or amide linkages which react or associate with acid dyes which result in staining. Stain blocking additives react or associate with these acid dye sites to prevent the acid dye sites from reacting or associating with acid dyes. Suitable stain blocking additives for use in polyamides are discussed in US Pat. No. 5,155,178, herein incorporated by reference. Suitable stain blocking additives include, but are not limited to aromatic sulfonates and alkali metal salts thereof, such as 5-sulfoisophthalic acid, sodium salt and dimethyl-5-sulfoisophthalate, sodium salt. In one nonlimiting, embodiment, the stain blocking additive is 5-sulfoisophthalic acid, sodium salt (NaSIPA).

[0050] In one nonlimiting embodiment, the stain blocking additive is present in the synthetic fiber in a range from about 1 to about 10 percent by weight. In another nonlimiting embodiment, the stain blocking additive is present in the synthetic fiber in a range from about 1 to about 5 percent by weight. In another nonlimiting embodiment, the stain blocking additive is present in the synthetic fiber in a range from about 0.1 to about 5 percent by weight.

[0051] The present disclosure also relates to yarns prepared from the synthetic fibers, as well as fabrics and carpets prepared from the synthetic fibers and/or yarns.

[0052] Also provided by this disclosure is a process for forming a synthetic fiber with enhanced stain resistance. The process comprises forming a polymer melt from a first fiber forming polymer and a masterbatch compound. The polymer melt can be formed by any known method in the art. In one nonlimiting embodiment, the polymer melt is formed in a melt extruder. A fiber or filament is then formed from the polymer melt.

[0053] In one nonlimiting embodiment, fibers may be prepared by using known melt spinning process technology. With such technology, the polymer melt is provided to a spinning machine. The polymer melt is forwarded by a metering pump to a filter pack, and through a spinneret plate

containing capillary orifices of a shape chosen to yield the desired filament cross-section at the spinning temperature. These cross-sectional shapes known in the art can include circular, non-circular, trilobal, and hollow shapes. Typical hollow filaments can be produced as disclosed in US Pat. No. 6,855,425. Spinning temperatures can range from about 270°C to about 300°C. The bundle of filaments emerging from the spinneret plate is cooled by conditioned quench air, treated with spin finish (an oil/water emulsion), and optionally interlaced, e.g. using an interlacing air jet to form a yarn.

[0054] Suitable polymers for use as the first fiber forming polymer include those that can be used to form synthetic fiber. Examples of first fiber forming polymers useful in this process include, but are not limited to, polyamides, polyesters, polyolefins and combinations thereof.

[0055] Suitable polyamides include fiber forming polyamides known in the art to be suitable for the formation of bulked continuous filament fibers, having sufficient viscosity, tenacity, chemical stability and crystallinity to be at least moderately durable in such application. The polyamide may be selected from the group consisting of nylon 5,6; nylon 6/6; nylon 6; nylon 7; nylon 11; nylon 12; nylon 6/10; nylon 6/12; nylon DT; nylon 6T; nylon 6I; and blends or copolymers thereof. In one embodiment the polyamide is nylon 6,6 polymer.

[0056] Suitable polyolefins include polypropylene. Suitable polyesters include fiber forming polyesters known in the art. The polyester resin may be selected from the group consisting of polyethylene terephthalate, polytrimethylene terephthalate, polybutylene terephthalate, polyethylene naphthalate, polylactic acid (PLA) and blends or copolymers thereof.

[0057] In one nonlimiting embodiment, the first fiber forming polymer is nylon 6.

[0058] In one nonlimiting embodiment, the first fiber forming polymer is present in a range from about 80 to 98 percent by weight. In another nonlimiting embodiment, the first fiber forming polymer is present in a range from about 80 to about 98.8 percent by weight.

[0059] The masterbatch compound used in the process comprises a second polymer having a melt point which is lower than the melt point of the first fiber forming polymer. In nonlimiting embodiments, the combination of first polymer and masterbatch does not produce a synthetic fiber that is prone to fibrillation. Examples of second polymers useful in the present disclosure include, but are not limited to polyolefins, polylactic acid, polystyrene, or a blend or copolymer thereof. In one nonlimiting embodiment, the polyolefin is an unmodified polyolefin. In another nonlimiting embodiment, the second polymer is polypropylene.

[0060] The masterbatch compound used in this process further comprises a stain blocking additive. Suitable stain blocking additives include those that disable acid dye sites. For example, in polyamides, such as Nylon 6,6 or Nylon 6, acid dyes sites refer to amine end groups or amide linkages which react or associate with acid dyes which result in staining. Stain blocking additives react or associate with these acid dye sites to prevent the acid dye sites from reacting or associating with acid dyes. Suitable stain blocking additives for use in polyamides are discussed in US Pat. No. 5,155,178, herein incorporated by reference. Suitable stain blocking additives include, but are not limited to aromatic sulfonates and alkali metal salts thereof, such as 5-sulfoisophthalic acid, sodium salt and dimethyl-5-sulfoisophthalate, sodium salt. In one nonlimiting, embodiment, the stain blocking additive is 5-sulfoisophthalic acid, sodium salt (NaSIPA).

[0061] In one nonlimiting embodiment, the masterbatch compound is present in fiber in a range from about 2 to about 20 percent by weight. In another nonlimiting embodiment, the masterbatch compound is present in fiber in a range from about 0.2 to about 20 percent by weight.

[0062] In one nonlimiting embodiment, the moisture content of the masterbatch compound is less than about 200 ppm. In another nonlimiting embodiment, the moisture compound content of the masterbatch is less than about 100 ppm. In yet another nonlimiting embodiment, the moisture content of the masterbatch compound is less than about 50 ppm.

[0063] The absence of moisture or volatiles is important to allow for proper melt processing of the polymer melt to effectively form a synthetic fiber. Drying or conditioning of the feedstocks, including the masterbatch compound, requires additional costs, processing steps and processing time. Conditioning, as is known by those skilled in the art, is a process of allowing the feedstock or masterbatch compound to reach hygroscopic equilibrium with its surrounding atmosphere. Frequently, conditioning is performed using an anhydrous gas, such as nitrogen, to achieve lower moisture content in a material such as pelletized masterbatch, polymer, fiber, or yarn, for example. Through undue experimentation, it was found that the masterbatch compound of the present disclosure does not require drying or conditioning prior to forming the polymer melt to achieve this moisture content. In one nonlimiting embodiment of the current disclosure, no drying or conditioning step is required to lower the moisture content of the masterbatch compound before forming the polymer melt.

[0064] US Pat No. 6,537,475, US Pat No. 6,753,385 and US Pat No. 6,861,480, incorporated herein by reference, require additional steps to remove moisture or volatiles during fiber formation. For example, if an extruder is used to form the polymer melt, one or more vents may be needed to facilitate the removal of moisture or volatiles. In addition, the devolatilization or moisture removal may be assisted through the use of vacuums along with the extruder or with injection of a gas, such as nitrogen, through the extruder vents. Other methods of removing moisture or volatiles include drying or conditioning of the feedstocks, the addition of water-scavenging additives, or a combination of these methods. Through undue experimentation, it was found that with the use of the first fiber forming polymer and the masterbatch compound of current disclosure, no additional steps are required to remove moisture or volatiles while forming the polymer melt. This provides a significant cost and time savings over the processes taught in the prior art. Therefore, in nonlimiting embodiments of the current disclosure, no additional step is required to remove volatiles while forming the polymer melt.

[0065] In one nonlimiting embodiment, the synthetic fibers produced by this process preferably comprise about 1 to about 10% by weight of the second polymer. In another nonlimiting embodiment, the synthetic fibers produced by this process preferably comprise about 1 to about 5% by weight of the second polymer. In another nonlimiting embodiment, the synthetic fibers produced by this process preferably comprise about 0.1 to about 10% by weight of the second polymer.

[0066] In one nonlimiting embodiment, the synthetic fibers produced by this process also preferably comprise about 1 to about 10% by weight of a stain blocking additive. In another nonlimiting embodiment, the synthetic fibers produced by this process also preferably comprise about 1 to about 5% by weight of a stain blocking additive. In another nonlimiting embodiment, the synthetic fibers produced by this process also preferably comprise about 0.1 to about 10% by weight of a stain blocking additive.

[0067] The present disclosure also relates to yarns prepared from synthetic fibers produced by this process, as well as fabrics and carpets prepared from the synthetic fibers and/or yarns.

[0068] Also provided by the present disclosure is a process for forming a nylon 6 fiber with enhanced stain resistance. This process comprises forming a polymer melt of a nylon 6 polymer and a masterbatch compound, then spinning synthetic fiber according to the aforementioned melt spinning processes to form a nylon 6 fiber with enhanced stain resistance from the polymer melt.

[0069] The masterbatch compound used in the process comprises a second polymer having a melt point which is lower than the melt point of the first fiber forming polymer. In nonlimiting embodiments, the combination of first polymer and masterbatch does not produce a synthetic fiber that is prone to fibrillation. Examples of second polymers useful in the present disclosure include, but are not limited to polyolefins, polylactic acid, polystyrene, or a blend or copolymer thereof. In one nonlimiting embodiment, the polyolefin is an unmodified polyolefin. In another nonlimiting embodiment, the second polymer is polypropylene.

[0070] The masterbatch compound used in this process further comprises a stain blocking additive. Suitable stain blocking additives include those that disable acid dye sites. For examples, in polyamides, such as Nylon 6,6 or Nylon 6, acid dyes sites refer to amine end groups or amide linkages which react or associate with acid dyes which result in staining. Stain blocking additives react or associate with these acid dye sites to prevent the acid dye sites from reacting or associating with acid dyes. Suitable stain blocking additives for use in polyamides are discussed in US Pat. No. 5,155,178, herein incorporated by reference. Suitable stain blocking additives include, but are not limited to aromatic sulfonates and alkali metal salts thereof, such as 5-sulfoisophthalic acid, sodium salt and dimethyl-5-sulfoisophthalate, sodium salt. In one nonlimiting, embodiment, the stain blocking additive is 5-sulfoisophthalic acid, sodium salt (NaSIPA).

[0071] In one nonlimiting embodiment, the moisture content of the masterbatch is less than about 200 ppm. In another nonlimiting embodiment, the moisture content of the masterbatch is less than about 100 ppm. In yet another nonlimiting embodiment, the moisture content of the masterbatch is less than about 50 ppm. In one nonlimiting embodiment this process, the masterbatch compound is not dried or conditioned prior to forming the polymer melt. In another nonlimiting embodiment of this process, no additional step is required to remove volatiles while forming of the polymer melt.

[0072] In one nonlimiting embodiment, the nylon 6 polymer is present in a range from about 80 to about 98 percent by weight and the masterbatch compound is present in a range from about 2 to about 20 percent by weight. In another nonlimiting embodiment, the nylon 6 polymer is present in a range from about 80 to about 98.8 percent by weight and the masterbatch compound is present in a range from about 0.2 to about 20 percent by weight.

[0073] The present disclosure also relates to yarns prepared from the nylon 6 fibers produced by this process, as well as fabrics and carpets prepared from the nylon 6 fibers and/or yarns.

[0074] Also provided by the present disclosure is a masterbatch compound. The masterbatch compound comprises a thermoplastic carrier. Examples of thermoplastic carrier useful in the masterbatch include, but are not limited to polyolefins, polylactic acid, polystyrene, or a blend or copolymer thereof. In one nonlimiting embodiment, the thermoplastic carrier is present in the masterbatch compound in a range from about 20 to about 80 percent by weight. In one nonlimiting embodiment, the polyolefin is an unmodified polyolefin. In another nonlimiting embodiment, the second polymer is polypropylene.

[0075] The masterbatch compound further comprises a stain blocking additive. Suitable stain blocking additives include those that disable acid dye sites. For example, in polyamides, such as Nylon 6,6 or Nylon 6, acid dyes sites refer to amine end groups or amide linkages which react or associate with acid dyes which result in staining. Stain blocking additives react or associate with these acid dye sites to prevent the acid dye sites from reacting or associating with acid dyes.

Suitable stain blocking additives for use in polyamides are discussed in US Pat. No. 5,155,178, herein incorporated by reference. Suitable stain blocking additives include, but are not limited to aromatic sulfonates and alkali metal salts thereof, such as 5-sulfoisophthalic acid, sodium salt and dimethyl-5-sulfoisophthalate, sodium salt. In one nonlimiting, embodiment, the stain blocking additive is 5-sulfoisophthalic acid, sodium salt (NaSIPA).

[0076] The masterbatch compound may further comprise other additives, to be used to confer additional benefits to articles upon polymer melt extrusion and melt spinning. Examples of such additives are inorganic pigments, and ultraviolet (UV) light absorbers or optical brightening agents. Examples of inorganic pigments are titanium dioxide, barium sulfate, carbon black, manganese dioxide, and zinc oxide. Examples of UV light absorbers or optical brightening agents are 2,2'-(1,2-ethenediyl)-4,4'-biphenylenebisbenzoxazole, available commercially by Eastman Chemical Company under the tradename Eastobrite® OB-1, and 2,2'-(2,5-thiophenediyl)bis(5-tert-butylbenzoxazole, available commercially by Mayzo, Inc. under the tradename Benetex® OB.

[0077] In one nonlimiting embodiment, the moisture content of the masterbatch compound is less than about 200 ppm. In another nonlimiting embodiment, the moisture content of the

masterbatch compound is less than about 100 ppm. In yet another nonlimiting embodiment, the moisture content of the masterbatch compound is less than about 50 ppm.

[0078] The following section provides further illustration of the synthetic fiber and fabrics knitted of this invention as well as comparative fibers and fabrics knitted therefrom. These working examples are illustrative only and are not intended to limit the scope of the invention in any way.

TEST METHOD

ACID DYE STAIN TEST

Acid dye stain resistance is evaluated using a procedure adapted from the American Association of Textile Chemists and Colorists (AATCC) Method 175-2003, "Stain Resistance: Pile Floor Coverings." 9 wt % of aqueous staining solution is prepared, according to the manufacturer's directions, by mixing cherry-flavored KOOL-AID® powder (Kraft/General Foods, Northfield, IL White Plains, N.Y., a powdered drink mix containing, inter alia, FD&C Red No. 40). A knitted sock (4x6-inch) is placed on a flat non-absorbent surface. A hollow plastic 2-inch (5.1 cm) diameter cup is placed tightly over the test sample, which can be a knitted sock, or a tufted carpet, for example. Twenty ml. of the KOOL-AID® staining solution is poured into the cup and the solution is allowed to absorb completely into the test sample. The cup is removed and the stained sample is allowed to sit undisturbed for 24 hours. Following incubation, the stained sample is rinsed thoroughly under cold tap water, excess water is removed by centrifugation, and the sample is dried in air. The sample was visually inspected and rated for staining according to the FD&C Red No. 40 Stain Scale described in AATCC Method 175-2003. Stain resistance is measured using a 1-10 scale. An undetectable test staining is accorded a value of 10.

EXAMPLES

[0079] Comparative Examples 1 and 2 and Examples 3 and 4 were produced using pilot scale machine. The pilot equipment included a 12" single screw extruder having five heating zones, a filter screen pack, any of a selection of desired spinnerets, a fiber quenching zone, godet rolls, and winders.

Example 1: Comparative Example – No Masterbatch

[0080] A 920 denier, 64 filaments N6 bulked continuous filament (BCF) was made on the pilot scale machine. Pigments of various colors were mixed with a N6 polymer product made by BASF, Ultramid® B27 E 01. The pigments and N6 were mixed at the screw feeder. Fibers were spun with no process breaks. This BCF yarn had a light earth tone color. It was knitted into a sock and tested for stain resistance according to the Acid Dye Stain Test. This sock rated 1 out of 10, and so failed the Acid Dye Stain Test.

Example 2: Comparative Example –NaSIPA/N6 Masterbatch

[0081] This example was similar to Example 1, except a masterbatch was added to the flake feeder in addition to the pigments. This masterbatch was a melt blend of NaSIPA with N6 (Ultramid® B27 E 01) at a 50/50 ratio by weight. The masterbatch was not dried or conditioned prior to use. The rate of masterbatch addition at the screw feeder was 6 weight percent. The addition of the NaSIPA/N6 masterbatch drastically reduced the polymer melt viscosity, and made the spinning process inoperable.

Example 3: NaSIPA/Polypropylene Masterbatch

[0082] Instead of the masterbatch as used in Example 2, a NaSIPA/polypropylene masterbatch (50/50 weight % blend) was used in this example in accordance with the present invention. The masterbatch was not dried or conditioned prior to use. Although some reduction in polymer viscosity was observed at 6 wt. % masterbatch addition rate, the spinning process was manageable. Light earth tone color solution dyed N6 BCF was successfully produced. A sock was knitted from the BCF and tested for stain resistance according to the Acid Dye Stain Test. The sock passed the stain test (rating 9 out of 10).

Example 4: Dimethyl-5-sulfoisophthalate, sodium salt/Polypropylene Masterbatch

[0083] Instead of the masterbatch as used in Example 2, a dimethyl-5-sulfoisophthalate, sodium salt/polypropylene masterbatch (50/50 weight % blend) was used in this example in accordance with the present invention. The dimethyl-5-sulfoisophthalate, sodium salt is sold by Mytech Inc. The masterbatch was not dried or conditioned prior to use. The rate of addition of the masterbatch to the N6 polymer was 6 weight percent. The viscosity drop was significantly less

than example 3. The spinning process was good. Light earth tone color solution dyed N6 BCF was successfully produced. A sock was knitted from the BCF and tested for stain resistance according to the Acid Dye Stain Test. The stain rating was 7.5 which was significantly better than example 1 with no masterbatch addition.

Example 5: NaSIPA/Polypropylene Masterbatch

[0084] This example was similar to Example 3 except the masterbatch addition rate was raised from 6 to 10 percent. It was also knitted into a sock, and tested for stain resistance according to the Acid Dye Stain Test. The sock passed the stain test (10 out of 10).

Example 6: Dimethyl-5-sulfoisophthalate, sodium salt /Polypropylene Masterbatch

[0085] This example was similar to Example 4 except the masterbatch addition rate was raised from 6 to 10 percent. It was also knitted into a sock, and tested for stain resistance according to the Acid Dye Stain Test. The stain rating was 8.5, which was significantly better than example 1.

[0086] Comparative Examples 7 and 11 and Examples 8, 9 and 10 were produced on a prototypic single position machine with roll speeds and productivity similar to commercial spinning machines. The nylon 6 polymer used for the Examples 7 through 11 was a bright polymer produced by BASF (Ultramid® B27 E 01). The resin had 2.7 +/- 0.3 relative viscosity (ISO 307) and was conditioned to have a moisture content of approximately 500 ppm. The polymer temperature before the spinning pack was controlled at about 265 +/- 1 °C). The spinning throughput was eighty pounds (80 lbs.) per hour.

[0087] The polymer was extruded through the spinnerets and divided into two (2) sixty-eight filament (68) segments. The molten fibers were then rapidly quenched in a chimney, where cooling air at about 10 °C was blown past the filaments at four hundred and fifty cubic feet per minute [450 cfm] through the quench zone. The filaments were then coated with a lubricant for drawing and crimping. The coated yarns were drawn at 2422 yards per minute (2.9x draw ratio) using a pair of heated draw rolls. The draw roll temperature was 160°C. The filaments were then forwarded into a dual-impingement hot air bulking jet, similar to that described in Coon, U.S. Patent 3,525,134, teachings of which are herein incorporated by reference, to form two

1350 denier, 20 denier per filament (dpf) BCF yarns. The temperature of the air in the bulking jet was 180 °C.

[0088] The spun, drawn, and crimped BCF yarns were knitted into socks and heat-set on a Suessen heat-setting machine at setting temperature of 185°C. The holdup time in the setting zone was about 60 seconds. The heatset socks were tested for stain resistance according to the Acid Dye Stain Test.

Example 7: Comparative Example – No Masterbatch

[0089] For this example, color pigments were mixed with N6 polymer at the screw feeder. The spinning process was good, with no process breaks. This BCF yarn had a light earth tone color. The test sample was knitted into sock, heatset in a Suessen chamber and tested for stain resistance according to the Acid Dye Stain Test. It failed the stain test with the rating of 5 in a 1 to 10 rating scale.

Example 8: Example – NaSIPA/Polypropylene Masterbatch

[0090] A masterbatch that consisted of color pigments, NaSIPA and polypropylene carrier was used to make this example. The NaSIPA loading in this masterbatch was approximately 50 weight percent. About 2 weight percent of the masterbatch was mixed with Nylon 6 polymer at the screw feeder. The spinning process was similar to Example 7. This BCF yarn also had a light earth tone color identical to Example 7. The test sample was knitted into sock, heatset in a Suessen chamber, and tested for stain resistance according to the Acid Dye Stain Test. It was rated 7.5. In terms of stain resistance, the BCF yarn was better than the yarn made in Example 7, but this yarn still underperformed.

Example 9: Example – NaSIPA/Polypropylene Masterbatch

[0091] This example was similar to Example 8, except the masterbatch loading was increased from 2 to 3 weight percent. The Suessen heat-set sock was tested for resistance according to the Acid Dye Stain Test. It was rated 9.0, which is considered as passing the Acid Dye Stain Test.

Example 10: Example – NaSIPA/Polypropylene Masterbatch

[0092] This example was similar to Example 8, except the masterbatch loading was increased from 3 to 4 weight percent. This example had a medium earth tone color. It was knitted into sock, heat set in a Suessen chamber, and tested for stain resistance according to the Acid Dye Stain Test. It was rated 9.5, which is considered as passing the Acid Dye Stain Test.

Example 11: Comparative Example with no Masterbatch

[0093] This example was similar to Example 7, except the pigment loading was increased to match the color of Example 10. The finished carpet was tested for stain resistance according to the Acid Dye Stain Test. It was rated 5.0, which is considered as not passing the Acid Dye Stain Test.

[0094] The reference in this specification to any prior publication (or information derived from it), or to any matter which is known, is not, and should not be taken as an acknowledgment or admission or any form of suggestion that that prior publication (or information derived from it) or known matter forms part of the common general knowledge in the field of endeavour to which this specification relates.

[0095] Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A process for forming a synthetic fiber with enhanced stain resistance, said process comprising the steps of:
 - a) forming a polymer melt of a first fiber forming polymer and a masterbatch compound, wherein the masterbatch compound comprises a second polymer having a melting point which is less than the melting point of the first fiber forming polymer, and a stain blocking additive, wherein the first fiber forming polymer is present in a range from about 80 to 98 percent by weight, and the masterbatch compound is present in a range from about 2 to 20 percent by weight; wherein the masterbatch compound is not dried or conditioned prior to forming the polymer melt; and
 - b) forming a synthetic fiber with enhanced stain resistance from the polymer melt.
2. The process of claim 1, wherein the masterbatch compound has a moisture content less than about 200 ppm, or less than about 50 ppm.
3. The process of claim 1 or 2, wherein no additional step is required to remove volatiles while forming the polymer melt.
4. The process of any one of claims 1 to 3, wherein the presence of the second polymer does not cause the synthetic fiber to fibrillate.
5. The process of any one of claims 1 to 4, wherein the first fiber forming polymer is selected from the group consisting of a polyamide, polyester, polyolefin and combinations thereof, or the first fiber forming polymer is nylon 6.
6. The process of any one of claims 1 to 5, wherein the second polymer is present in the masterbatch compound in a range from about 20 to 80 percent by weight.

7. The process of any one of claims 1 to 6, wherein the stain blocking additive is present in the masterbatch compound in a range from about 20 to 80 percent by weight.
8. The process of any one of claims 1 to 7, wherein the second polymer is present in the synthetic fiber in a range from about 1 to 10 percent by weight
.
9. The process of claim 8, wherein the second polymer is present in the synthetic fiber in a range from about 1 to 5 percent by weight.
10. The process of any one of claims 1 to 9, wherein the second polymer is a polyolefin.
11. The process of claim 10, wherein the second polymer is an unmodified polyolefin.
12. The process of any one of claims 1 to 9, wherein the second polymer is polypropylene.
13. The process of any one of claims 1 to 12, wherein the stain blocking additive is present in the synthetic fiber in a range from about 1 to 10 percent by weight.
14. The process of claim 13, wherein the stain blocking additive is present in the synthetic fiber in a range from about 1 to 5 percent by weight.
15. The process of any one of claims 1 to 14, wherein the stain blocking additive is an aromatic sulfonate or an alkali metal salt thereof, optionally wherein the stain blocking additive is 5-sulphoisophthalate, sodium salt.
16. The process of any one of claims 1 to 15, wherein forming a synthetic fiber further comprises:
 - a) extruding said polymer melt through a spinneret to form one or a plurality of filaments, and

- b) drawing the fiber.
- 17. A yarn formed from the process of any of claims 1 to 16.
- 18. A fabric knitted from the synthetic fiber produced from any of the process claims of claims 1 to 16 or the yarn of claim 17.
- 19. A carpet formed from the yarn of claim 17.
- 20. A rug formed from the yarn of claim 17.

1/2

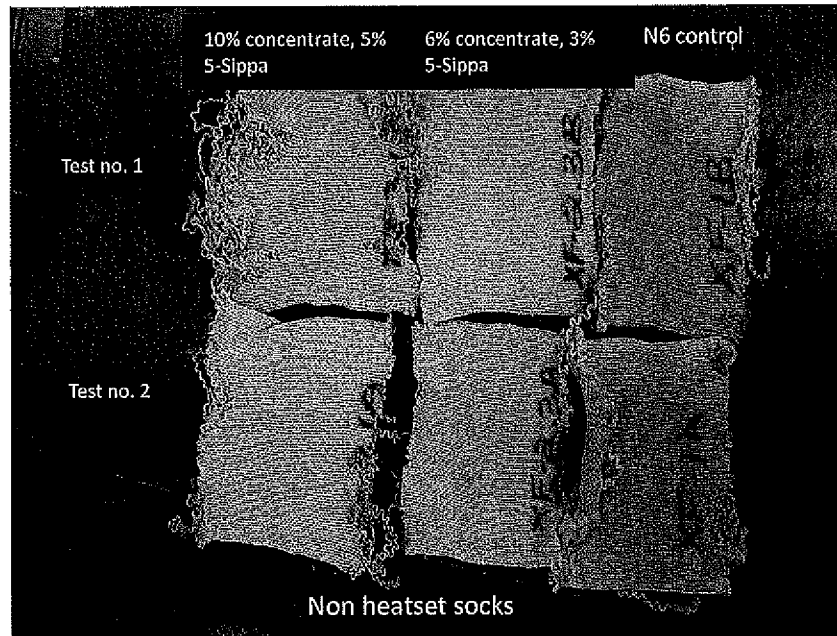


FIGURE 1

2/2

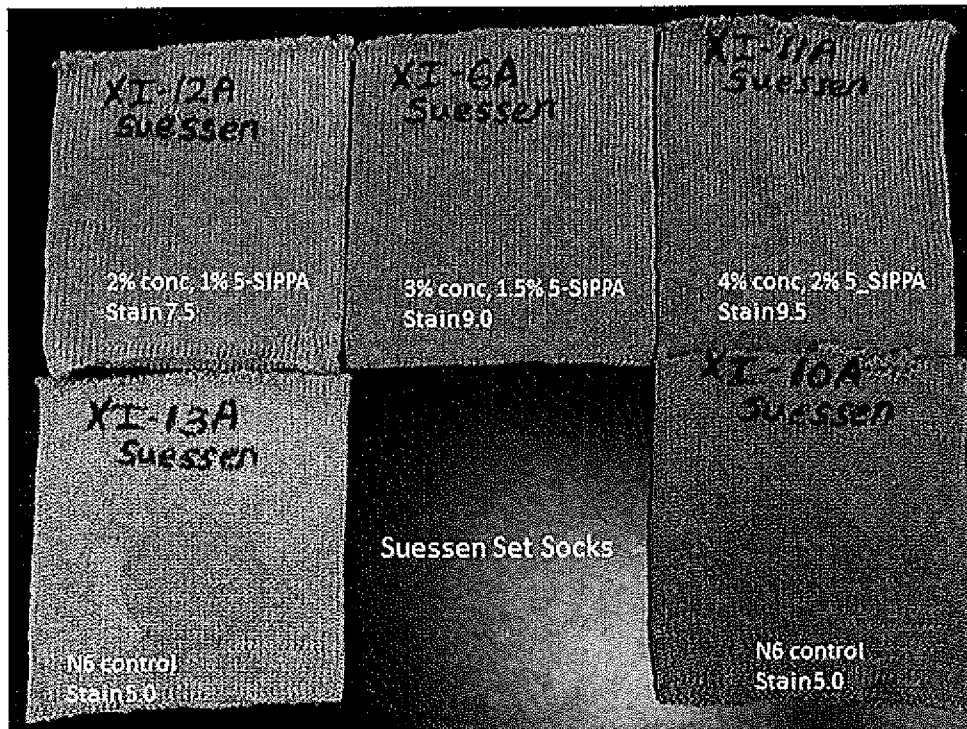


FIGURE 2