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(54) Process for extruding fibers

(57) The present invention provides a process for obtaining a fiber comprising the steps of:

melt extruding a mixture of a thermoplastic polymer and a hydrophilicity imparting compound A to form a plurality of filaments;
applying a spin finish to said filaments;
and spinning said filaments into a fiber;
characterised in that said spin finish comprises a fluorochemical oil and/or water repellent.

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Description

1. Field of the invention.

[0001] The present invention relates to fiber production and fiber treatment rendering a fiber soil resistant, oil repellent, and/or water repellent.

2. Background of the invention.

[0002] In the formation of textile materials from extruded thermoplastic polymers, such as polypropylene, application of a spin oil to the filaments is standard practice. Spin finish including a spin oil is a lubricating composition deposited on the surface of the fiber to reduce the fiber-fiber friction and the friction developed as the yarn passes over the metal machinery surfaces. Spin oil typically contains a large number of chemical components, the major components being lubricant, antistatic agent and emulsifier. The amount of spin finish needed depends on the producer and manufacturing and typically the residual spin finish on the fiber varies between 0.7% and 5%. A major disadvantage of the use of spin finish including a spin oil is that residues on the extruded fiber attract soil. It also decreases the efficiency of a protective treatment and as such reduces the soil resistance of the finished products.

[0003] In the industrial production of textiles, such as carpet and apparel, it is common to treat such substrates with a composition to impart added desirable properties thereto, such as resistance to soiling by particulate or dry soil. Fluorochemical compositions are commercially used for this purpose. They can be applied to various substrates by methods which include, for example, spraying, foaming, padding, and finish bath immersion.

[0004] U.S. Patent No. 4,264,484 discloses a liquid carpet treating composition containing a water-insoluble addition polymer derived from polymerizable ethylenically unsaturated monomer free of nonvinylic fluorine and having at least one major transition temperature higher than about 25°C, and a water-insoluble fluoroaliphatic radical- and aliphatic chlorine-containing ester having at least one major transition temperature higher than about 25°C.

[0005] U.S. Patent No. 4,107,055 discloses a fabric coating composition, including a polymer having a glass transition temperature above room temperature, an ionic fluorinated surfactant and a carrier. The polymer is preferably applied to fabric at a rate giving a dry solids content of about 0.25 to 10%, to give dry soil resistance.

[0006] U.S. Patent No. 4,043,964 discloses a coating which provides a durably soil-resistant carpet which contains (a) at least one phase of a specified water-insoluble addition polymer derived from a polymerizable ethylenically unsaturated monomer free of non-vinylic fluorine and (b) at least one phase of a specified water-insoluble fluorinated component containing a fluoroaliphatic radical of at least 3 carbon atoms. The monomer from which the fluorinated component is formed may contain dicarboxylic acid, glycol, diamine, hydroxyamine, etc.

[0007] A common feature of the treating or coating compositions disclosed in the above mentioned U.S. Patent Nos. 4,264,484, 4,107,055 and 4,043,964 is that they are to be applied to the carpet or fabric after its production in a separate treating step. The application equipment and time required for such treating step add to the cost of the final product.

[0008] Textile fibers and yarns can also be treated by incorporation of a fluorochemical repellent treating agent in the spin finishing bath, such as e.g. disclosed in US 4,190,545 and 4,192,754. A drawback of the addition of fluorochemical treating agent to the spin finishing bath is the formation of deposits on the rolls caused by sedimentation of fluorochemical oil and water repellent agent/spin finish mixture. The deposit on the rolls can cause fiber breaks and therefore must be removed frequently. This is time consuming and expensive and therefore no longer accepted as application method by fiber manufacturers. Typically the more spin finish added together with the fluorochemical treating agent, the more roll build up is observed, and the worse the repellent properties of the finished product are due to the high level of spin oil present.

[0009] Alternatively, treated textile fibers and yams can be obtained by melt extrusion of a blend of a synthetic fiber-forming polymer and a fluorochemical composition. Such melt extrusion is described for example in U.S. Patent No. 3,839,312. This patent discloses that soil and stain repellency of extruded filaments of a synthetic resin can be improved by incorporating in the resin a small amount, about 1 percent, of an amphipathic compound having from one to four fluoroalkyl groups pendent from an organic radical. The repellency is provided by the fluoroalkyl groups, which tend to be concentrated at the surface of the fiber.

[0010] WO 92/18569 and WO 95/01396 disclose permanently soil resistant polymeric compositions such as fibers and yarns that have a fluorochemical dispersed throughout the polymer. These polymer compositions are prepared by melt extrusion of the fluorochemical with the desired polymer. Polymers that can be used with the fluorochemical include polyester, polypropylene, polyethylene and polyamide.

[0011] U.S. Patent No. 5,025,052 discloses certain fluoroaliphatic group-containing oxazolidinone compositions. The patent also discloses fibers, films, and molded articles prepared, for example, by injection molding a blend or mixture of fiber-or film-forming synthetic organic polymers and certain fluorochemical oxazolidinones. The resulting fibers, films, and molded articles are said to have low surface energy, oil and water repellency, and anti-soiling properties.

[0012] European Pat. Pub. No. 0 516 271 discloses durably hydrophilic thermoplastic fibers comprising thermoplastic polymer and fluoroaliphatic group-containing non-ionic compounds.

[0013] While the above mentioned publications, U.S. Patent No. 3,839,312, WO 92/18569 and WO 95/01396, are successful in providing soil and stain repellency to a yarn or fiber and many currently used fluorochemical compositions have demonstrated utility in providing carpet with soil resistance, unfortunately a significant amount of the carpet or fabric manufactured cannot be treated to obtain the desired properties. The reason is that significant and varying amounts of spin oil often remain on the fiber or yarn, lowering the soiling resistance thereof or acting as contaminants which interfere with the fluorochemical treatment and diminish or prevent the desired result thereof.

[0014] WO 97/33019 discloses a carpet yarn that contains a hydrophilicity imparting compound dispersed in the filaments of thermoplastic polymer. Fluorochemical hydrophilicity imparting compounds are disclosed as preferred hydrophilicity imparting compounds. It is taught that as a result of the use of the hydrophilicity imparting compound in the filaments of the yarn, less or no spin oil is needed in the spin finishing bath and as a result, the carpet is less prone to soiling. Although this method is successful, the oil and/or water repellency properties of the obtained fibers is generally poor and there continues to be a desire to improve the soil repellency properties.

3. Summary of the invention.

[0015] The present invention provides a process for obtaining a fiber comprising the steps of:

melt extruding a mixture of a thermoplastic polymer and a hydrophilicity imparting compound A to form a plurality of filaments;
applying a spin finish to said filaments;
and spinning said filaments into a fiber;
characterised in that said spin finish comprises a fluorochemical oil and/or water repellent.

[0016] It was found that the process of the present invention can be run without the occurrence of roll build-up during production of the fibers. Also, the resulting fibers showed good oil and water repellency properties in addition to soil resistance and low levels of spin oil can be used without sacrifice in antistatic behaviour of the fiber.

[0017] Further provided is a fiber containing a plurality of filaments of a thermoplastic polymer having dispersed therein a hydrophilicity imparting compound A characterised in that the filaments comprise on at least part of their surface a fluorochemical oil and/or water repellent.

[0018] Finally, there is also provided a carpet or textile including such fibers.

4. Detailed description of the invention.

[0019] Thermoplastic polymers useful in the invention include fiber-forming poly(alpha)olefins, polyesters and polyamides. Preferred thermoplastic polymers are poly (alpha)olefins. The poly(alpha)olefins of the present invention include the normally solid homo-, co- and terpolymers of aliphatic mono-1-olefins (alpha olefins) as they are generally recognized in the art. Usually the monomers employed in making such poly(alpha)olefins contain 2 to 10 carbon atoms per molecule, though higher molecular weight monomers sometimes are used as comonomers. The invention is applicable also to blends of the polymers and copolymers prepared mechanically or in situ. The monomers employed include ethylene, propylene, butene-1, pentene-1, 4-methyl-pentene-1, hexene-1, and octene-1, alone, or in admixture, or in sequential polymerization systems.

[0020] Examples include polyethylene, the presently preferred polypropylene, propylene/ethylene copolymers, polybutylene and blends thereof. Processes for preparing various polymers are well known, and the invention is not limited to a polymer made with a particular catalyst or process.

[0021] Hydrophilicity imparting compounds A suitable for use in this invention can be fluoro-chemical or non-fluorochemical or a mixture of such compounds can be employed. Non-fluorochemical hydrophilicity imparting compounds A are substantially free of fluorine (preferably containing less than 10 % by weight and more preferably less than 5% by weight of fluorine) and are generally hydrophilic in nature or contain at least a hydrophilic moiety such that hydrophilicity or wettability can be provided to the surface of the thermoplastic polymer filaments. Included are low molecular weight compounds, oligomers as well as polymers. Suitable non-fluorochemical hydrophilicity imparting compounds A are preferably incompatible with the thermoplastic polymer melt and are preferably sufficiently stable at the required extrusion temperatures.

[0022] Suitable non-fluorochemical hydrophilicity imparting compounds A can be anionic, cationic, non-ionic or amphoteric. Preferred compounds are surfactants. Particularly preferred non-fluorochemical hydrophilicity imparting compounds are those that contain a poly(oxyalkylene) group.

[0023] Fluorochemical hydrophilicity imparting compounds A which are useful in the techniques of this invention are

hydrophilic in nature and include compounds, oligomers, and polymers. Such materials will contain at least about 10% by weight of fluorine, i.e. carbon-bonded fluorine. They contain one or more fluorochemical radicals (R_f), and one or more water solubilizing polar groups (Z), which radicals and groups are usually connected together by suitable linking groups (Q).

[0024] The fluorochemical radical, R_f, in compound A can be generally described as a fluorinated, preferably saturated, monovalent radical of at least 4 carbon atoms. Preferably the fluorochemical radical is a fluoroaliphatic, non-aromatic radical. The aliphatic chain may be straight, branched, or, if sufficiently large, cyclic and may include oxygen, di- or hexavalent sulfur, or trivalent nitrogen atoms bonded only to carbon atoms. A fully fluorinated radical is preferred, but hydrogen or chlorine atoms may be present as substituents provided that no more than one atom of either is present for every two carbon atoms. Fluoroaliphatic radicals containing about 5 to about 12 carbon atoms are most preferred.

[0025] The water solubilizing polar group or moiety, Z, of fluorochemical compound A can be a non-ionic, anionic, cationic, or amphoteric moiety, or combinations of said groups or moieties which may be the same or different. Preferably, the water solubilizing group comprises a poly(oxyalkylene) group, (OR')x, where R' is an alkylene group having 2 to 4 carbon atoms, such as $-CH_2CH_2-$, $-CH_2CH_2CH_2-$, $-CH(CH_3)CH_2-$, and $-CH(CH_3)CH(CH_3)-$ or mixtures thereof, and x is an integer from about 6 to about 20. The oxyalkylene units in said poly(oxyalkylene) may be the same, as in poly(oxypropylene), or present as a mixture, such as in a heteric straight or branched chain of randomly distributed oxyethylene and oxypropylene units poly(oxyethylene-co-oxypropylene), or as in a straight or branched chain of blocks of oxyethylene units and blocks of oxypropylene units. The poly(oxyalkylene) chain can be interrupted by or include one or more catenary linkages, providing such linkages do not substantially alter the water-solubilizing character of the poly(oxyalkylene) chain. The Z group is terminated with hydroxyl or lower alkyl ether for example, $-OCH_3$ or $-OCH_2CH_3$. Typical anionic groups include CO_2H, CO_2M, SO_3H, SO_3M, OSO_3H, OSO_3M, $OPO(OH)_2$, and $OPO(OM)_2$, where M is a metallic ion (such as sodium or potassium), or ammonium ion, or other amine cation. Typical cationic groups include $NR_3^+A^-$, where R is a lower alkyl group such as methyl, ethyl, butyl, hydroxyethyl or hydrogen and A is a anion such as chloride, sulfate, phosphate, hydroxide or iodide. Typical mixed or amphoteric groups would include $N^+(CH_3)_2C_2H_4COO^-$, $N^+(CH_3)_2C_3H_6SO_3^-$ or an amine-oxide.

[0026] The linking group, Q, is a multivalent, generally divalent, linking group such as alkylene, arylene, sulfonamidoalkylene, carbonamidoalkylene, and other heteroatom-containing groups such as siloxane, including combinations of such groups. In some instances more than one fluoroaliphatic radical may be attached to a single linking group and in other instances a single fluoroaliphatic radical may be linked by a single linking group to more than one polar solubilizing group. Q can also be a covalent bond.

[0027] A particularly useful class of fluorochemical hydrophilicity imparting compounds A are those of the formula

$$R_f\text{-}Q\text{-}Z$$

where R_f is said fluoroaliphatic radical, Q is said linking group and Z is said water solubilizing group.

[0028] Hydrophilicity imparting fluorochemical non-ionic compounds A useful in the present invention can be prepared using known methods such as those described in U.S. Patent No. 2,915,554 (Albrecht et al.). The Albrecht patent discloses the preparation of fluoroaliphatic group-containing non-ionic compounds from active hydrogen containing fluorochemical intermediates, such as fluoroaliphatic alcohols, e.g. $R_fC_2H_4OH$, acids e.g. $R_fSO_2N(R'')CH_2COOH$, and sulphonamides, e.g., $R_fSO_2N(R'')H$, by reaction of the intermediates with, for example, ethylene oxide to yield, respectively, $R_fC_2H_4O(C_2H_4O)_nH$, $R_fSO_2N(R'')CH_2CO_2(C_2H_4O)_nH$, and $R_fSO_2N(R'')(C_2H_4O)_nH$, where n is a number greater than about 3, and R'' is hydrogen or lower alkyl (e.g., 1 to 6 carbons). Analogous compounds can be prepared by treating the intermediates with propylene oxide or a mixture of ethylene oxide and propylene oxide. See also the fluoroaliphatic oligomers disclosed in U.S. Pat. No. 3,787,351 (Olson), and certain fluorinated alcohol-ethylene oxide condensates described in U.S. Pat. No. 2,723,999 (Cowen et al.).

[0029] The hydrophilicity imparting compound A is typically added in amounts between about 0.05 and 5% by weight, preferably between about 0.5 and 1.5% by weight, based on the total weight of thermoplastic polymer.

[0030] Examples of hydrophilicity imparting compounds A include:

$C_8F_{17}SO_2N(C_2H_5)CH_2CH_2O(CH_2CH_2O)_7CH_3$

2 $C_8F_{17}SO_3H$. Jeffamine™ ED-600 di-salt (Jeffamine™ ED 600 is an amino terminated ethylene oxide-propylene oxide polymer)

Copolymers of fluorochemical acrylates or methacrylates and mono- acrylate or mono-methacrylates of polyethylene-oxide

$C_8H_{17}SO_2N(C_2H_5)(CH_2CH_2O)_2\text{-}(CH(CH_3)CH_2O)_6H$

Polyethylene oxide

Copolymers of ethylene oxide and propylene oxide

Ethoxylated alkyl phenols (such as Triton™ X-100, available from Union Carbide, Danbury, Connecticut)

[0031] In accordance with the present invention a fiber or yarn can be produced by providing a mixture comprising a thermoplastic polymer and one or more hydrophilicity imparting compounds A which can be fluorochemical or non-fluorochemical as described above. This mixture can be extruded to form filaments which are then preferably cooled. The bundle of filaments is then typically treated in a spin finish bath. Alter receiving the spin finish treatment, the filaments are generally stretched. Stretching may be accomplished over a number of rolls that are at elevated temperature sufficient to soften the thermoplastic polymer. By rotating the rolls at different speeds, stretching of the filaments can be obtained. While stretching can be accomplished in one step, it may be desirable to stretch the filaments in two steps. Typically, the filaments will be stretched 3 to 4 times the extruded length. Subsequent to stretching and to obtain a carpet yarn, it will often be desirable to texture the yarn with pressured air at an elevated temperature or steam jet and to subject it to an entanglement.

[0032] In accordance with the present invention, the spin finish comprises a fluorochemical oil and/or water repellent. Typically, the amount of fluorochemical oil and/or water repellent in the spin finish will be between 1% by weight and 20% by weight, preferably between 7% by weight and 13% by weight. In general, fluorochemical oil and/or water repellents useful in the invention include any of the known fluoroaliphatic radical-containing agents useful for the treatment of fabrics to obtain oil and water repellency. Fluorochemical radical-containing agents include condensation polymers such as polyesters, polyamides or polyepoxides and vinyl polymers such as polyacrylates, polymethacrylates or polyvinyl ethers. Such known agents include, for example, those described in U.S. Pat. No. 3,546,187; U.S. Pat. No. 3,544,537; U.S. Pat. No. 3,470,124; U.S. Pat. No. 3,445,491; U.S. Pat. No. 3,341,497 and U.S. Pat. No. 3,420,697.

[0033] Further examples of such fluoroaliphatic radical-containing water and oil repellency imparting agents include those formed by the reaction of perfluoroaliphatic glycols or thioglycols with diisocyanates to provide perfluoroaliphatic group-bearing polyurethanes. These products are normally applied as aqueous dispersions for fiber treatment. Such reaction products are described, for example, in U.S. Patent No. 4,054,592. Another group of compounds which can be used are fluoroaliphatic radical-containing N-methylol condensation products. These compounds are described in U.S. Patent No. 4,477,498. Further examples include fluoroaliphatic radical containing polycarbodiimides which can be obtained by, for example, reaction of perfluoroaliphatic sulfonamido alkanols with polyisocyanates in the presence of suitable catalysts.

[0034] The fluorochemical oil and/or water repellent can be a copolymer of one or more fluoroaliphatic radical-containing acrylate or methacrylate monomers, and one or more fluorine-free (or hydrocarbon) terminally ethylenically-unsaturated comonomers.

[0035] Representative examples of fluorochemical monomers include:

$CF_3(CF_2)_4CH_2OCOC(CH_3)=CH_2$

$CF_3(CF_2)_6(CH_2)_2OCOC(CH_3)=CH_2$

$CF_3(CF_2)_6(CH_2)_2OCOCH=CH_2$

$CF_3(CF_2)_7(CH_2)_2OCOCH=CH_2$

$$CF_3(CF_2)_7SO_2N(C_2H_5)(CH_2)_2OCOCH=CH_2$$

$CF_3CF_2(CF_2CF_2)_{2\text{-}8}CH_2CH_2OCOCH=CH_2$

$$CF_3(CF_2)_7SO_2N(C_2H_5)(CH_2)_2OCOC(CH_3)=CH_2$$

$$CF_3(CF_2)_7CH_2CH_2SO_2NCH_2CH_2OCOC(CH_3)=CH_2$$
$$|$$
$$CH_3$$

[0036] Preferred co-monomers which can be copolymerized with the above-described fluoroaliphatic radical-containing monomers include those selected from the group consisting of octadecylmethacryate, 1,4-butanediol diacrylate, laurylmethacrylate, butylacrylate, N-methylol acrylamide, isobutylmethacrylate, ethylhexyl acrylate, ethylhexyl methacrylate, vinylchloride and vinylidene chloride. The relative weight ratio of the fluoroaliphatic monomer(s) to the hydrocarbon co-monomer(s) can vary as is known in the art.

[0037] Further examples of useful fluorochemical oil and/or water repellents include, for example, fluorinated group containing urethanes, ureas, esters, amines (and salts thereof), amides, acids (and salts thereof), carbodiimides, guanidines, allophanates, biurets oxazolidinones, and other substances containing one or more fluorinated groups, as well as mixtures and blends thereof. Such agents are well known to those skilled in the art, see e.g., Kirk-Othmer, Encyclopedia of Chemical Technology, 3rd Ed., Vol. 24, pp. 448-451 and many (e.g., SCOTCHGARD®. Fabric Protector, 3M) are commercially available as ready-made formulations

[0038] The spin finish typically also includes any conventional spin oil used for the extrusion of thermoplastic polymers. However, the spin finish can also be water without any spin oil added to it. The spin finish can be applied using methods known in the art. One example includes kiss roll application. The lower part of the kiss roll dips in the finish bath, while the yarn tangentially moves over the top part. The add-on level of spin oil can be varied by changing several parameters, such as the geometry between yarn and roll, roll speed and the concentration of spin oil in the spin finishing bath. When spin oil is used, the parameters will be adjusted so that the residual amount of spin oil on the filament will be between about 0.01% and 1.2% by weight, preferably between about 0.01% ad 0.6% by weight, based on the total weight of filaments and spin oil. Most preferably, the residual amount of spin oil will be less than 0.4%.

[0039] The following examples further illustrate the invention without however the intention to limit the invention thereto.

EXAMPLES

[0040] All parts, percentages, etc in the Examples and Comparative Examples are by weight unless otherwise noted.

[0041] Respective data of oil and water repellency and soil resistance shown in the Examples and Comparative Examples were based on the following methods of measurement: Water repellency (WR) : The water repellency of a substrate was measured using a series of water-isopropyl alcohol test liquids and was expressed in terms of the "WR" rating of the treated substrate. The WR rating corresponded to the most penetrating test liquid which does not penetrate or wet the substrate surface after 15 seconds exposure. Substrates which were penetrated by or were resistant only to a 100% water (0% isopropyl alcohol), the least penetrating test liquid, were given a rating of 0, whereas treated substrates resistant to 100% isopropyl alcohol (0% water), the most penetrating test liquid, were given a rating of 10. Other intermediate ratings were calculated by dividing the percent isopropylalcohol in the test liquid by 10, e.g., a treated substrate resistant to a 70%/30% isopropyl alcohol/water blend, but not to an 80%/20% blend, would be given a rating of 7.

Oil repellency (OR) : measured according to AATCC standard test method No 118-1983.

Dry soil resistance : method described in US 5,370,919. A value of 5 indicates that there is no increase in soiling versus an unsoiled sample ; a dry soil rating of 1 refers to severe soiling.

Determination of fluorine in the fiber

[0042] In order to determine the amount of fluorine in the extruded fiber, following method is used: a known weight of the sample is placed in an ignition basket made from platinum wires. The sample is then decomposed in a sealed polycarbonate flask in the presence of oxygen and a known volume of buffer solution, TISAB III (available from Orion). After absorption in the buffer solution, the fluoride is measured with an Orion 9409 (fluoride sensitive) electrode connected to a pH meter using the mV-mode of operation. The amount of fluoride is then calculated from the mV reading using a graph plotted from standard fluoride solutions. All samples are analyzed in duplicate and the results should show less than 10% variation to be considered uniform. When properly calibrated, the electrode measurement will be reproducible with a deviation of about 2%.

Abbreviations

[0043] The following abbreviations and trade names are used in the examples :

PP:	polypropylene HG 235 J : polypropylene with MFI 25, available from Borealis
FC-1 :	$C_8F_{17}SO_2N(C_2H_5)CH_2CH_2O(CH_2CH_2O)_7CH_3$
FC :	50/50 blend of FC 398/399 : Scotchgard™ Carpet Protector
SF :	spin finish
FA 2820 :	spin finish available from Zschimmer und Schwartz (Germany)
FA 2825 :	spin finish available from Zschimmer und Schwartz (Germany)
Lurol™ NF-6239-20 :	spin finish available from Deva NV (Belgium)
Lurol™ PP-3919 :	spin finish available from Devan NV (Belgium)
rpm :	rates per minute

General procedure for producing yarn

[0044] In a first step, masterbatches of polypropylene containing 25% FC-1 were prepared and extruded into granules. These granules were further blended with polypropylene in order to obtain polypropylene comprising 0.4-0.45% FC-1. The so formed blends were extruded with a Thermo Alfa single screw extruder (Triply extruder-one end) at about 230°C over spin plates with a trilobal cross section.
Alter leaving the extruder, the filaments passed through a cooling zone over a kiss roll where a solution of water with a conventional spin oil and a conventional fluorochemical oil and water repellent treating agent was applied. The amount of spin oil and oil and water repellent treating agent applied to the yarn was varied by adjusting the kiss roll speed and the concentration of the compounds in the spin finishing bath. The carpet yarn was then drawn at about 3-4 times the extruded length to obtain a yarn with a tex (weight (g)/1000 m) of about 165. During the production of the yarn there was no roll build up when FC-1 was added to the PP melt. Without the use of FC-1, deposition of a sticky layer on the guidance rolls (of the cooling unit) caused major problems and delays in the production of treated yarn. The yarn was textured at a temperature of 140°C to 180°C to produce a bulked yarn that is particularly useful for production of carpets. The bulked yarn was visually inspected for mechanical quality after spinning and texturing. Bulked yarn made in

accordance with the present invention had no visible broken filaments. In order to test oil and water repellency and soil resistance yarn was wrapped closely together around a piece of cardboard, so that there were no gaps in between the yarn and a flat surface of one layer of yarn was obtained.

Examples 1 to 3 and comparative example C-1.

[0045] Examples 1 to 3 were made by using 0.4% FC-1 as polypropylene melt additive. The spin finishing bath contained different amounts of spin finish FA 2820 and fluorochemical oil and water repellent treating agent FC 398/399. Comparative example C-1 was made without the use of FC-1 as polypropylene melt additive. In this case, it will be noted that higher level of spin finish was needed in order to avoid too much static build up. Composition and extrusion parameters of polypropylene fibers are recorded in table 1.

Table 1

Composition and extrusion parameters of polypropylene fibers				
Ex No	% SF (*)	% FC	Speed extruder screw (rpm)	roll build up
1	7	8	85	initially some at edges of yarn trail
2	7	12	85	no
3	5	8	85	negligible
C-1	10	8	52	yes

Note : (*) % Spin finish used in the spin finishing bath, not the residual amount of spin finish on the fiber

[0046] As can be seen from the results, the addition of fluorochemical hydrophilicity imparting compound to the melt prevents roll build up despite the use of a fairly low concentration (0.4%) of fluorochemical hydrophilicity imparting compound in the polypropylene melt. Comparative example C-1, without FC-1 in the melt showed severe roll build up right from the start.

[0047] The fibers were tested for their oil and water repellency properties and dry soil resistance. The results are recorded in table 2.

Table 2

Properties of polypropylene fibers				
Ex No	OR	WR	Dry soil	F analysis (ppm)
1	3	5	3	1652
2	3	3	3.5	2663
3	1	0	3.5	1878
C-1	3	5	3	707

[0048] Although the repellent properties of C-1 were good, production was not acceptable, because of severe roll build up. Good oil and water repellent properties could be obtained with the samples according to the invention and excellent Dry soil values were obtained therewith.

Examples 4 to 11 and comparative examples C-2 to C-6.

[0049] In examples 4 to 11 polypropylene containing 0.45 % FC-1 was extruded. Different spin finish agents together with fluorochemical oil and water repellent treating agent (FC398/399) were added to the spin finishing bath. Comparative examples C-2 to C-6 were made with 0.45% FC-1 in the melt, but without the use of fluorochemical oil and water repellent treating agent in the spin finishing bath. Extrusion behaviour and fiber properties are given in tables 3 and 4.

Table 3

Composition and extrusion of polypropylene fibers			
Ex No	% Spin finish	% FC	Roll build up
4	7% FA 2825	8	no
5	7% FA 2820	8	no
6	7% FA 2820	12	no
7	7% Lurol™ NF-6239-20	12	no
8	12% Lurol™ NF-6239-20	12	no
9	20% Lurol™ NF-6239-20	12	very small, at yarn edges
10**	7%Lurol™ PP-3919	12	no
11	7% Lurol™ PP-3919	12	no
C-2	7% FA 2825	/	no
C-3	7% FA 2820	/	no
C-4	7% Lurol™ NF-6239-20	/	no
C-5	20% Lurol™ NF-6239-20	/	no
C-6	7% Lurol™ PP-3919	/	no

Note : ** : this trial was run with a kiss roll speed of 18 rpm instead of the standard 13.5 rpm. This did not cause any problems, indicating that an increased add-on level does not result into unexpected roll build up as would be the case if no FC-1 was used.

Table 4

Evaluation of oil and water repellency and dry soil resistance				
Ex No	OW	WR	Soil	F analysis
4	4	2	3.5	2243
5	3	4	2	2512
6	2	2	2.5	2910
7	3	7	3.5	2442
8	3	6	4	2258
9	3	7	3.5	2613
10	1	0	1.5	3122
11	2	1	2.5	2524
C-2	0	0	3.5	1574
C-3	0	0	3	1474
C-4	0	0	3.5	1898
C-5	0	0	4	1827

Table 4 (continued)

Evaluation of oil and water repellency and dry soil resistance				
Ex No	OW	WR	Soil	F analysis
C-6	0	0	2.5	1871

[0050] As can be seen from the results, the fibers comprising fluorochemical hydrophilicity imparting compound dispersed in their filaments and oil and water repellent treating agent on their surface showed high repellency properties combined with no sedimentation or roll build up. Since FC-1 was used in the comparative examples, low level of spin finish could be used and the comparative examples also showed good soil resistance. However, they did not have any oil/water repellent properties.

Claims

1. Process for obtaining a fiber comprising the steps of:

   melt extruding a mixture of a thermoplastic polymer and a hydrophilicity imparting compound A to form a plurality of filaments;
   applying a spin finish to said filaments;
   and spinning said filaments into a fiber;
   characterised in that said spin finish comprises a fluorochemical oil and/or water repellent.

2. Process according to claim 1 wherein said hydrophilicity imparting compound A is a fluorochemical hydrophilicity imparting compound.

3. Process according to claim 1 or 2 wherein said hydrophilicity imparting compound A is a non-ionic hydrophilicity imparting compound.

4. Process according to claim 1 wherein said hydrophilicity imparting compound A corresponds to the following formula:

$$R_f\text{-}Q\text{-}Z$$

   wherein R_f is a fluorinated, monovalent residue having at least 4 carbon atoms, Q is a linking group or a covalent bond and Z is a water solubilizing group.

5. Process according to claim 4 wherein Z comprises a poly(oxyalkylene) group.

6. Process according to claim 1 wherein the amount of hydrophilicity imparting compound A in said mixture is between 0.05% by weight and 5% by weight relative to the weight of thermoplastic polymer.

7. Process according to any of the previous claims wherein said thermoplastic polymer is a poly(alpha)olefin.

8. Process according to claim 7 wherein the poly(alpha)olefin is a polypropylene.

9. Process according to any of the previous claim wherein said spin finish further includes a spin oil.

10. Fiber containing a plurality of filaments of a thermoplastic polymer having dispersed therein a hydrophilicity imparting compound A characterised in that the filaments comprise on at least part of their surface a fluorochemical oil and/or water repellent.

11. Fiber according to claim 10 wherein said hydrophilicity imparting compound A is a fluorochemical hydrophilicity imparting compound.

12. Fiber according to claim 10 or 11 wherein said hydrophilicity imparting compound A is non-ionic hydrophilicity imparting compound.

13. Fiber according to claim 10 wherein said hydrophilicity imparting compound A corresponds to the following formula:

$$R_f\text{-}Q\text{-}Z$$

wherein R_f is a fluorinated, monovalent residue having at least 4 carbon atoms, Q is a linking group or a covalent bond and Z is a water solubilizing group.

14. Fiber according to claim 13 wherein Z comprises a poly(oxyalkylene) group.

15. Fiber according to claim 10 wherein the amount of hydrophilicity imparting compound A in said fiber is between 0.05% by weight and 5% by weight relative to the weight of thermoplastic polymer.

16. Fiber according to any of claims 10 to 15 wherein said thermoplastic polymer is a poly(alpha)olefin.

17. Fiber according to claim 16 wherein the poly(alpha)olefin is a polypropylene.

18. A textile or carpet comprising fibers as defined in any of claims 10 to 17.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number
EP 97 20 3812

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.Cl.6)
A	WO 97 44508 A (MINNESOTA MINING & MFG)		D01F1/10 D01F6/04 D01F6/06 D06M13/213 D06M15/277 D06M15/576 //D06M101:20
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TECHNICAL FIELDS SEARCHED (Int.Cl.6)

D01F
D06M

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	29 April 1998	TARRIDA TORRELL, J

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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