(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
3 August 2006 (03.08.2006)

PCT

(10) International Publication Number
WO 2006/080634 A1

(51) International Patent Classification:
D01F 1/10 (2006.01)

(21) International Application Number:
PCT/KR2005/003159

(22) International Filing Date:
23 September 2005 (23.09.2005)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
10-2004-0084786 22 October 2004 (22.10.2004) KR

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(81) Designated States *(unless otherwise indicated, for every kind of national protection available)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KP, KZ, LC, LK, LR, LS, LT, LU, LV, LY, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States *(unless otherwise indicated, for every kind of regional protection available)*: ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:
— *with international search report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 2006/080634 A1

(54) Title: A POLYURETHANE ELASTIC FIBER HAVING CHLORINE-RESISTANCE ANTISTATIC PROPERTY AND A METHOD FOR PRODUCING THE SAME

(57) Abstract: The present invention relates to a polyurethane elastic fiber having excellent chlorine-resistance and antistatic property without affecting the original physical properties of polyurethane-polymers, and a method for producing the same. According to the present invention, the polyurethane-elastic fiber may have excellent chlorine-resistance and antistatic property by means of containing hydrotalcite coated with aliphatic alocohol.

Description

A POLYURETHANE ELASTIC FIBER HAVING CHLORINE-RESISTANCE ANTISTATIC PROPERTY AND A METHOD FOR PRODUCING THE SAME

Technical Field

[1] The present invention relates to a polyurethane elastic fiber with excellent chlorine-resistance and antistatic property and a method for manufacturing the same, in particular a polyurethane elastic fiber produced by a method that hydrotalcite coated with aliphatic alcohol is added to a polyurethane solution for excellent chlorine-resistance and antistatic property.

[2]

Background Art

[3] In general, polyurethane elastic fiber has higher rubber-like elasticity, and at the same time has excellent physical properties such as high tensile force and restoration force and the like, and hence the fiber has been utilized for underwear, socks and sports wear.

[4] However, if polyurethane contained in the polyurethane fiber as major component is washed with sodium hypochlorite, sodium chlorite or the like, the physical properties of polyurethane may be degraded heavily. And also, if a swimming wear manufactured by warp-knitting polyurethane and other synthetic fiber such as polyamide and polyester contacts chlorine water (the concentration of active chlorine is 0.5 to 3.5 ppm), the physical properties of the polyurethane- elastic fiber would be degraded.

[5] Efforts to improve the bad quality caused by chlorine have been made for a long time, and the results were shown in many documents in which various compounds are utilized for chlorine-resistance, for example zinc oxides in US patent no. 4,340,527; mixture of huntite and hydromagnesite in US patent no. 5,626,960; calcium carbonate and barium carbonate in Korean publication no. 92-3250; MgO/ZnO solid solution in Japan publication no.H6-81215; magnesium oxide, magnesium hydroxide or hydrotalcite in Japan publication no. S59-133248; hydrotalcite coated with stearic acid and inevitably comprising water of crystallization in US patent no. 5,447,969; and hydrotalcite coated with polyorgano siloxane in US patent no. 5,969,028 are used for chlorine-resistance agents.

[6] In general, a hydrotalcite compound can be used to make chlorine be innoxious effectively because the compound of hydrotalcite has a property to capture a halogen compound. Hydrotalcite coated with strearic acid in US patent no. 5,447,969 and

coated with polyorgano siloxane in US patent no. 5,969,028 are used to avoid the absorption of water and to enhance the distribution property, resulting in improving in some degree the problems in respect with the rise of discharge pressure and yarn breakage in spinning process, but other properties of polyurethane elastic fiber except chlorine-resistance cannot be improved.

[7] Polyurethane elastic fiber would generate stastic electricity in post-manufacturing process (for example, circular knit, warp knit) where a large quantity of polyurethane elastic fiber is wound for knitting fabrics because polyurethane elastic fiber has a property of adhersion. The study to improve the problem of static electricity has been developed, and it was found by the inventors of this application that a hydrotalcite coated with aliphatic alcohol is more effective than the conventional hydrotalcites for improving disadvantage relating to static electricity.

[8] Hydrothalcite would have excellent antistatic property, which has been found according to this application, as well as dispersion property, which is advantage of known art, within polyurethane by adding hydrothalcite coated with aplipatic alcohol to polyurethane- polymer.

[9]

Disclosure of Invention

Technical Problem

[10] It is an object of the present invention to provide a polyurethane elastic fiber having excellent chlorine-reisistance and antistatic property by adding a hydrotalcite coated with apliphatic alcohl to polyurethane polymer and a method for producing the same.

[11]

Technical Solution

[12] For achieving the above-mentioned object, a polyurethane elastic fiber may comprise a hydrothalcite coated with aliphatic alcohol.

[13] And also, the content of the added hydrothalcite may be 0.1 to 10 wt% to the weight of polyurethane.

[14] And also, the hydrotalcite may be coated with 0.1 to 20 wt% aliphatic alcohol to the weight of hydrotalcite.

[15] And also, the hydrotalcite may be preferably represented by the following formula (1):

[16] $M^{2+}_{x}Al_2(OH)_y(A^{n-})_zO_k \cdot mH_2O$ ------ (1), wherein M^{2+} represents Mg^{2+}, Ca^{2+}, or Zn^{2+}, A^{n-} represents an any anion with n valences, x and y represent 2 or any positive number equal to or bigger than 2, Z is any positive equal to or less than 3, k is 0 or any positive number equal to or less than 3 and m represent 0 or any positive number. Further, A^{n-} may represent any one among OH^-,F^-,Cl^-,Br^-,NO_3^-,SO_4^{2-},CH_3COO^-,CO_3^{2-}

,HPO_4^{2-}, oxalate ion, salicylate ion and silicate ion.

[17] Preferably the hydrotalcite may be represented as one of the the following formulas from (2) to (13):

[18] $Mg_{4.5}Al_2(OH)_{13}CO_3 \cdot 3.5H_2O$---- (2)

[19] $Mg_6Al_2(OH)_{16}CO_3 \cdot 5H_2O$---- (3)

[20] $Mg_8Al_2(OH)_{20}CO_3 \cdot 6H_2O$---- (4)

[21] $Mg_4Al_2(OH)_{12}CO_3 \cdot 3H_2O$ ---- (5)

[22] $Mg_{4.5}Al_2(OH)_{13}CO_3$ ---- (6)

[23] $Mg_6Al_2(OH)_{16}CO_3$ ---- (7)

[24] $Mg_8Al_2(OH)_{20}CO_3$ ---- (8)

[25] $Mg_4Al_2(OH)_{12}CO_3$ ---- (9)

[26] $Mg_{4.5}Al_2(OH)_{13}(CO_3)_{0.6}O_{0.4}$ ---- (10)

[27] $Mg_6Al_2(OH)_{16}(CO_3)_{0.7}O_{0.3}$ ---- (11)

[28] $Mg_{4.5}Al_2(OH)_{12.2}(CO_3)_{0.8}O_{0.6}$ ---- (12)

[29] $Mg_4Al_2(OH)_{12}(CO_3)_{0.6}O_{0.4}$ ---- (13), wherein formulas (6) to (9) represent hydrotalcites from which water of crystallization is removed, and formulas (10) to (13) represent hydrotalcites from which water of crystallization is removed and at the same time within which a part of CO_3^{2-} or OH^- is decomposed.

[30] Aliphatic alcohol may be an aliphatic alcohol which is mono-valent or polyvalent and is linear or branched with 3 or 40 carbons. Preferably aliphatic alcohol may comprise one or two compounds selected from a group consisting of alkanol, cycloalkanol, n-hexanol, n-heptanol, n-octanol, 2-ethyl hexanol, isooctyl alcohol, octanol-2, methyl heptanol, decyl alcohol, isodecyl alcohol, caprylalcohol, lauryl alcohol, myristyl alcohol, palmityl alcohol, oleyl alcohol, benhenyl alcohol, cetyl alcohol, stearyl alcohol, cyclohanxol, alkandiol, propylene glycol, trimethylene glycol, 1,2-butylene glycol, 2,3-butylene glycol, 1,4-butylene glycol, 1,6-haxanediol, pinacol, 1,2-pentanediol, 2-methyl-2, 4-pentanediol, 1,3-butylene glycol, neopentyl glycol, 2-ethyl-1,3-haxanediol, 2,4-pentanediol, 2,4-heptanediol, 2.2-diethyl-1,3-propanediol, 2-methyl-2-butyl-1,3-propanediol, 2-methyl-2-butyl-1,3-propanediol, pentaerythritol and dipentaerythritol. More preferably, aliphatic alcohol may comprise one or two compounds selected from a group consisting of stearyl alcohol, lauryl alcohol, caprylalcohol and pentaerythritol.

[31] The method for coating aliphatic alcohol may be performed with water or solvents, and alternatively a dried coating method may be utilized.

[32] The method for coating with water or solvents may be performed in a manner that hydrotalcite is dissolved into water or a solvent and aliphatic alocohl is added to the solution with stir in the amount of 0.1 to 20 wt% compared to the weight of hydrotalcite to heat up to the melting point of the aliphatic alocohl with mixture. After

stirring for more than 30 minutes, the temperature of the solution is lowered to the room temperature to filter and dry, and then the resultant solution is coated on the surface of hydrotalcite with electro-magnetic wave.

[33] On the other hand, the dryed coating method may be performed in a manner that hydrotalcite and 0.1 to 20 wt% aliphatic alcohol to the weight of hydrotalcite is input into a high speed stirrer and then heats up to the melting point of aliphatic alcohol with mixture.

[34] Alternatively, only hydrotalcite may be input into the stirrer for mixing and the pre-melt apliphatic alocohol may be sprayed into hyrotalcite for mixing, and then the mixture may be coated on the surface of hydrotalcite with electro-magnetic wave. The electro-magnetic wave may comprise ultra-violet rays, gamma-rays, X-rays, microwaves or infrared rays, preferably microwaves or infrared rays.

[35] If aliphatic alocohol is not coated using electro-magnetic waves such as ultraviolet rays, gamma-rays, X-rays, microwaves or infrared rays, the coating efficiency may be poor, resulting in bad dispersion within polymer. It was found that the method for coating with elecro-magnetic waves improves the efficiency. And a polyurethane elastic fiber having excellent chlorine-resistance and antistatic property can be obtained by adding hydrotalcite coated with aliphatic alcohols by using electro-magnetic waves.

[36] The particle size of hydrotalcite coated with aliphatic alocohol may be less than 10 um, preferably less than 5 um.

[37] The amount of hydrotalcite coated with aliphatic alocohol may be 0.1 to 10 wt% to the weight of the polyurethane elastic fiber. If the amount of hydrotalcite is less than 0.1 wt%, then chlorine-resistance and antistatic property of polyurethane fiber is too weak to use for industrial material, while if more than 10 wt%, then strength, elongation and modulus are poorer owing to the excessive content of inorganic component despite the improvement of chlorine-resistance and antistatic property.

[38] And also, hydrotalcite coated with aliphatic alcohol may be added at any step for manufacturing the polyurethane elastic fiber. For example, hydrotalcite coated with aliphatic alcohol may be used being added to any other additive solution or dispersion solution, and alternatively hydrotalcite may be separately added to a polymer solution.

[39] Polyurethane polymer used for manufacturing a polyurethane elastic fiber according to the present invention may be prepared in a known method, for example organic di-isocynnate may be reacted with high molecular diol to produce a precursor of polyurethane, and then the resultant is dissolved into an organic solution to react diamine and monoamne for producing a polyurethane- polymer.

[40] Diisocynnate used for producing polyurethane may comprise diphenylmethane-4, 4'diisocynnate, hexamethylene diisocynnate, toluene diisocynnate, butyl diisocyannte,

hydride P,P-methyl diisocynnate and the like. High molecular diol may comprise poly-tetramethylene ether glycol, polypropylene glycol, polycarbonate diol and the like.

[41] And also, Amines may be used as terminating-chain agent, including diethylamine, monoethanol amine, dimethylamine and the like.

[42] According to the present invention, in order to avoid discoloration of a polyurethane elastic fiber and degradation of physical property owing to ultra-violet rays, atmosphere smog and heat-treatment in manufacturing process of the polyurethane elastic fiber, some compounds such as hindered phenols, benzofuran-ons, semi-cabizids, benzo triazoles, hindered amines, polymeric tertiary amine stabilizers(for example, polyurethane , polydialkyl aminoalkyl methacrylate having tertiary nitrogen elements) and the like.

[43] The polyurethane elastic fiber according to the present invention may further comprise inorganic additives such as TiO_2, magnesium stearate and the like. The amount of TiO_2 may be 0.1 to 5 wt% depending to the whiteness of the polyurethane elastic fiber and compared to the total weight of the polyurethane elastic fiber. And also, magnesium stearate may be added to in amount of 0.1 to 2 wt% to the total weight of the polyurethane elastic fiber in order to improve the winding property of the polyurethane elastic fiber.

[44]

Advantageous Effects

[45] As foregoing described in detail, the present invention makes the advantage of prior art, excellent dispersion property of hydrotalcite within polyurethane, be kept in the produced polyurethane-elastic fiber. And in addition the present invetn imparts new characteristic, excellent antistatic property, to the procduced polyurethane elastic fiber by adding hydrotalcite coated with aliphatic alocohol to the polyurethane- polymer.

[46]

Best Mode for Carrying Out the Invention

[47] The present invention will be clearly understood with examples in the following, not limiting the scope of the present invention.

[48]

[49] **<Chlorine Resistance Test>**

[50] Strength preservation rate test within chlorine water:

[51] A polyurethane elastic fiber under elongation of 50% was treated within water of pH 4.2 at the temperature of 97 to 98 ℃ for 2 hours (wet-heat treatment), and then the fiber is deposited into 45 L chlorine water of 3.5 ppm active chlorine concentration and pH 7.5 for 24 hours after cooling the fiber to the room temperature, and then the strength preservation rate was tested for the polyurethane elastic fiber according to the

following equation:

[52] - Strength preservation rate (%) = S/S_0 100, wherein S and S_0 represents strength before treatment and strength after treatment, respectively.

[53] For estimating strength preservation rate, Instron 4301 available from Instron LTD., USA was used and the length of sample was 5 cm and cross head speed was 300 mm/ min with 1 kg cell.

[54]

[55] **<Static Electricity Test>**

[56] The bobbine for which static electricity should be tested kept under the condition of 20 ℃ temperature and 42 % retive humidity for 24 hours and then static electricity was tested under the same condition as the above.

[57] The test was performed according to the rolling take-up method that the bobbine was fixed at a creel and an unwinding roller contacting the bobbine was rotated to unwind. In the test the unwinding speed was 150 m/min and a take-up roller speed was 200 m/min. The diameter of winding roller is the same as that of the take-up roller, and the space between the winding roller and the take-up roller was 1 m, placed on the same height from the bottom.

[58] A steel bar was placed between the winding roller and the take-up roller for raw yarns to pass above the steel bar. The location for measuring static electricity was between the steel bar and the take-up roller, and the measurement was done at 2 minites after unwinding start. KSD-0103 static electricity device available from KASUGA LTD., Japan was used for measuring.

[59]

[60] **Preparation of Segment Polyurethane polymer**

[61] A Polyurethane prepolymer having isocynnates at both terminals was prepared by reacting 518 g diphenylmethane-4, 4'diisocynnate with 2328 g polytetramethyl ether glycol (molecular weight 1800) at 80 ℃ for 90 minutes with stirring in nitrogen atmosphere. The prepolymer was cooled to the room temperature, and then 4269 g dimethylacetamide was added to for obtaining a prepolymer solution. Subsequently, 34.4 g ethylene diamine, 10.6 g propylene diamine and 9.1 g diethylamine were dissolved into 1117g dimethylacetamide to obtain a mixed solution, and the the mixed solution was added to the prepolymer solution to obtain a polyurethaneurea solution. In order to obtain spinning solution, additives such as 1.5 wt% ethylene bis(oxyethylene)bis-(3-(5-t-butyl-4-hydroxy-m-toil)-propionate), 0.5 wt% 5,7-di-t-butyl-3-(3,4-dimethylphenyl)-3H-benzofuran-2-on, 1 wt% 1, 1,1'1'-tetramethyl-4,4'-(methylene-di-phenylene)disemicabazide, 1 wt% poly(N,N-diethyl-2-aminoethyl metacrylate) and 0.1 wt% TiO_2 were added to the prepolymer for obtaining a spinning solution, wherein the weight percentage of the

above additives represents the amount compared to the total solids weight of the polymer.

[62] The above additives were dispersed into a dimetylacetamide solvent and ground by using Advantis V3 device available from Drais Mannheim Cooperate, German before being added to the polymer, and then the milled additives were added to the polymer.

[63]

Mode for the Invention

[64] **Example 1-5 and comparison 1-4**

[65] Hydrotalcite (example 1-5 and comparison 1-4) was added to a polyurethaneurea solution, to be mixed for obtaining a spinning solution of polyurethaneurea, as showin in the following Table 1 and 2.

[66] After degassing the spinning solution, the spinning solution was spun into 250 ℃ hot air of the upper spinning channel to produce a 3-filament-40-deniers polyurethaneurea elastic fiber, and the fiber was wound after treated using 3.5 % finish oil TNU-604 available from Sanyo Cooperate, Japan.

[67] The physical properties of the produced fiber were estimated and shown in Table 1 and 2.

[68]

[69] Table 1

	Ex.1	Ex.2	Ex.3	Ex.4	Ex.5
Co.; Am.	Stearyl; 3 wt%	Pentaerythritol;3 wt%	Lauryl; 3 wt%	Caprylalcohol;3 wt%	Oleil;3 wt%
FFor.; Am.	$Mg_4Al_2(OH)_{12}CO_3\cdot 3H_2O$; 4 wt%	$Mg_4Al_2(OH)_{12}CO_3\cdot 3H_2O$;4 wt%	$Mg_4Al_2(OH)_{12}CO_3\cdot 3H_2O$; 4 wt%	$Mg_4Al_2(OH)_{12}CO_3$;4 wt%	$Mg_4Al_2(OH)_{12}(CO_3)_{0.8}O_{0.2}$; 4 wt%
S.P.R	88 %	89 %	87 %	95 %	86 %
S.E	0.2	0.2	0.3	0.2	0.1

[70]

[71] Note) "Co." and "Am." represent "coating material" and "the weight % of the coating material compared to the weight of the hydrotalcite" respectively and the coating material is aliphatic alocohols.

[72] "For." and "Am." represent "chemical formula for hydrotalcite" and "the weight % compared to the total weight of the produced fiber" respectively.

[73] "S.P.R" represents "strength preservation rate (%)" after chrorine resistant test.

[74] "S.E" represents "Static electricity" in kV unit.

[75]

[76] Table 2

	Com.1	Com.2	Com.3	Com.4
Co.; Am.	Stearic acid 3 wt%	Parmitic acid; 3 wt%	No hydrotalcite	No hydrotalcite
For.; Am.	$Mg_{4.5}Al_2(OH)_{13}CO_3 \cdot 3.5H_2O$;4 wt%	$Mg_6Al_2(OH)_{16}CO_3 \cdot 5H_2O$;4 wt%	$Mg_4Al_2(OH)_{12}CO_3$;4 wt%	$Mg_4Al_2(OH)_{12}(CO_3)_{0.5}O_{0.5}$;4 wt%
S.P.R.	85 %	83 %	80 %	72 %
S.E	0.5	0.6	0.5	0.7

[77] Note) "Co." and "Am." represent "coating material" and "the weight % of the coating material compared to the weight of the hydrotalcite" respectively and the coating material is aliphatic alocohols.

[78] "For." and "Am." represent "chemical formula for hydrotalcite" and "the weight % compared to the total weight of the produced fiber" respectively.

[79] "S.P.R." represents "strength preservation rate (%)" after chrorine resistant test.

[80] "S.E" represents "Static electricity" in kV unit.

[81]

[82] Referring to Table 1 and 2, yarns producd according to comparison 3 and 4 were broken frequently in spinning process of the polymer, and also the physical properties of the produced yarns were poorer than these of yarns produced by other examples.

[83] As shown in Table 1 and 2, the polyurethane elastic fibers (example 1 to 5) according to the present invention, which were produced by adding hydrotalcite coated with aliphatic alcohol to the polyurethane solution, has more excellent chlorine-resistance by at least 85 % in strength preservation rate(%) than the fibers produced according to the prior art (comparison 1 to 4), and also the polyurethane elastic fiber according to the present invention (example 1 to 5) has excellent antistatic property.

[84]

Claims

[1] A polyurethane elastic fiber with excellent chlorine-resistance and antistatic property comprising hydrotalcite, whererin the hydrotalcite is coated with aliphatic alcohols and the amount of hydrotalcite is 0.1 to 10 wt% to the weight of the the polyurethane elastic fiber.

[2] The polyurethane elastic fiber according to claim 1, wherein the aliphatic alcohol is at least one selected from mono or polyvalent aliphatic alocohols containg linear or branched hydrogencarbon with 3 to 40 carbons.

[3] The polyurethane elastic fiber according to claim 1, wherein the aliphatic alocohol is seleceted from a group consisting of stearyl alcohol, lauryl alcohol, caprylalcohol and pentaerythritol.

[4] The polyurethane elastic fiber according to claim 1, wherein the hydrotalcite is represented in the following formula:

$M^{2+}_{x}Al_2(OH)_y(A^{n-})_z O_k \cdot mH_2O$, wherein M^{2+} represents Mg^{2+}, Ca_2^{+}, or Zn^{2+}, A^{n-} represents an any anion with n valences, x and y represent 2 or any positive number equal to or bigger than 2, Z is any positive equal to or less than 3, k is 0 or any positive number equal to or less than 3 and m represent 0 or any positive number, and further, A^{n-} may represent any one among OH^-,F^-,Cl^-,Br^-,NO_3^-,SO_4^{2-},CH_3COO^-,CO_3^{2-},HPO_4^{2-}, oxalate ion, salicylate ion and silicate ion.

[5] The polyurethane elastic fiber according to claim 1, wherein the hydrotalcite is at least one selected from compounds represented in the following formulae (2) to (13):

$Mg_{4.5}Al_2(OH)_{13}CO_3 \cdot 3.5H_2O$---- (2)

$Mg_6Al_2(OH)_{16}CO_3 \cdot 5H_2O$---- (3)

$Mg_8Al_2(OH)_{20}CO_3 \cdot 6H_2O$---- (4)

$Mg_4Al_2(OH)_{12}CO_3 \cdot 3H_2O$ ---- (5)

$Mg_{4.5}Al_2(OH)_{13}CO_3$ ---- (6)

$Mg_6Al_2(OH)_{16}CO_3$ ---- (7)

$Mg_8Al_2(OH)_{20}CO_3$ ---- (8)

$Mg_4Al_2(OH)_{12}CO_3$ ---- (9)

$Mg_{4.5}Al_2(OH)_{13}(CO_3)_{0.6}O_{0.4}$ ---- (10)

$Mg_6Al_2(OH)_{13}(CO_3)_{0.7}O_{0.3}$ ---- (11)

$Mg_{4.5}Al_2(OH)_{12.2}(CO_3)_{0.8}O_{0.6}$ ---- (12)

$Mg_4Al_2(OH)_{12}(CO_3)_{0.6}O_{0.4}$ ---- (13), wherein formula (6) to (9) represents hydrotalcites from which the water of crystallization is removed, and formula (10) to (13) represents hydrotalcites from which the water of crystallization is removed and at the same time within which a part of CO_3^{2-} or OH^- is

decomposed.

[6] A method for producing a polyurethane elastic fiber containing hydrotalcite comprising the steps of:

(A) preparing a polyurethane prepolymer by reacting organic diisocynate with diol;

(B) preparing a polyurethane solution by dissolving the polyurethane prepolymer into a organic solvent and then reacting diamines and monoamines; and

(C) adding the hydrotalcite coated with alipathic alocohols to the polyurethane solution together with additives or seperatly and then spinning the polyurethane solution.

[7] The method according to claim 6, wherein the hydrotalcite coated with aliphatic alocohols is treated with ultraviolet rays, gamma rays, X-rays, microwaves or infrared rays in finishing step of the coating process.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2005/003159

A. CLASSIFICATION OF SUBJECT MATTER

IPC7 D01F 1/10

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7 D01F

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

KR, JP : IPC as above

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 12-198979 A (KYOWA CHEM. IND. CO. LTD.) 18 July 2000 (18.07.2000) see the whole documents	1-7
A	US 6692828 B2 BYUNG SU SONG wt als) 17 February 2004 (17.02.2004) see the whole documents	1-7
A	US 6716523 B2 (THOMAS EDWARD CARNEY et als) 06 April 2004 (06.04.2004) see the whole documents	1-7

☐ Further documents are listed in the continuation of Box C.

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