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(54) Title: PROCESS FOR PREPARING ELASTIC FIBER HAVING HIGH MODULUS, ALKALI-RESISTANCE AND HEAT-RESISTANCE

(57) Abstract: Disclosed herein is a process for preparing an elastic fiber having a high modulus and superior resistance to alkali and heat. The process comprises the steps of adding 1~20% by weight of a cellulose acetate to a polyurethane or polyurethaneurea solution, based on the total weight of the polyurethane or polyurethaneurea, homogeneously stirring the mixture to obtain a spinning solution, and ripening and spinning the solution. According to the process, a high modulus and highly heat resistant elastic fiber can be prepared without a sudden change in conditions for preparation of the polymer.

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PROCESS FOR PREPARING ELASTIC FIBER HAVING HIGH MODULUS, ALKALI -RESISTANCE AND HEAT-RESISTANCE

Technical Field

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The present invention relates to a process for preparing an elastic fiber having a high modulus and superior resistance to alkali and heat. More specifically, the present invention relates to a process for preparing a high modulus and highly alkali and heat resistant elastic fiber by adding 1~20% by weight of a cellulose acetate to a polyurethane or polyurethaneurea solution, based on the total weight of the polyurethane or polyurethaneurea, to obtain a spinning solution, and ripening and spinning the solution. According to the process, a high modulus and highly heat resistant elastic fiber can be prepared without a sudden change in conditions for preparation of the polymer.

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Background Art

An elastic fiber is used in a wide variety of applications. For example, the elastic fiber acts to tightly hold a polyester yarn of a 3-way warp knitted velvet fabric, together with a polyester fiber. The elastic fiber is required to have a modulus high enough to form and maintain a good raised state, and at the same time, to have heat resistance sufficient to endure dyeing and setting treatments at high temperature in subsequent post-processing steps of a polyester velvet fabric. An embossing technique has been recently in the spotlight as a technique for increasing the added value of a velvet fabric. For embossing of various patterns, raised yarns of the velvet fabric must undergo burning-out and printing into a desired color. For the processes, superior alkali resistance is inevitably necessary for the elastic fiber. Specifically, since a high concentration alkaline solution (e.g., mainly caustic soda solution) is used to dissolve polyester raised yarns at desired sites in the burning-out process, a printing solution is applied thereto, followed by high temperature treatment for dye fixing, the elastic fiber is required to have superior alkali resistance in order to prevent it from being degraded by alkaline solution remaining on the fabric at high temperatures.

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Problems often encountered in manufacturing a velvet fabric using common

elastic fibers are as follows. Since the common elastic fibers have a low modulus, there occurs a phenomenon wherein raised yarns are flattened after shearing, and thus the elastic fibers exhibit poor velvet effects. In addition, since the elastic fibers lose their inherent elastic recovery due to high temperature post-processing, the fabric spreads out, and in extreme cases, breakage of the elastic fibers takes place. Particularly, the elastic fibers are severely broken by a high concentration alkaline solution and a high temperature treatment employed in an embossing technique requiring burning-out and printing, causing occurrence of a number of holes on the velvet fabric.

Due to these problems, manufacturers of velvet fabrics make a demand for high modulus and highly heat resistant elastic fibers from manufacturers of elastic fibers. Furthermore, manufacturers of burnt-out and printed velvet fabrics call for the supply of elastic fibers having superior alkali resistance as well as high modulus and superior heat resistance.

The most common processes adopted by manufacturers of elastic fibers in order to solve the above-mentioned problems are associated with the presence of a high content of hard segment in a polymer solution for the preparation of an elastic fiber, and the use of a chain extender having a high binding force and containing no side chain. The higher the content of the hard segment, the higher the modulus of the elastic fiber. The use of the chain extender improves heat resistance of the elastic fiber. However, since these processes have a difficulty in the management of the viscosity of the polymer solution, they cause non-uniform physical properties of the elastic fiber to be prepared using the polymer solution, and are not suitable where small-scale preparation of the elastic fiber is required. On the other hand, elastic fibers have alkali resistance to some extent due to their inherent characteristics, but are likely to easily degrade when caustic soda at a high concentration of about 25% to about 30% is applied at a temperature as high as 160°C~180°C. Accordingly, burning-out and printing processes have been separately carried out. No technique has been established that can simultaneously proceed burning-out and printing processes to date.

Thus, there is a need in the art for a process for easily preparing an elastic fiber having a high modulus and superior resistance to heat and alkali, without any problem associated with non-uniform physical properties.

Japanese Patent Laid-open No. 2000-303259 issued to Fujibo discloses a polyurethane elastic fiber having improved moisture-absorbing properties and biodegradability. According to this publication, the polyurethane elastic fiber is prepared by adding a cellulose acetate to a polyurethane or polyurethaneurea solution, homogeneously stirring the mixture to obtain a spinning solution, spinning the solution to prepare an acetylcellulose-containing elastic fiber, and treating the acetylcellulose-containing elastic fiber with an alkali. However, the publication does not mention the alkali or heat resistance of the elastic fiber. In addition, the modulus of the elastic fiber is far too low to solve the above-mentioned problems.

Disclosure of the Invention

The present inventors have earnestly and intensively conducted research to solve the above-mentioned problems, and as a result, have found that when 1~20% by weight of a cellulose acetate (diacetate or triacetate) having a degree of acetylation of about 28% to about 72% is added to a polymer solution, based on the solid content (i.e., polymeric components) of the polymer solution, homogeneously stirring the mixture to obtain a spinning solution, ripening the solution for a predetermined period of time, and spinning the ripened solution, an elastic fiber having a high modulus and superior resistance to heat and alkali can be easily prepared without a sudden change in polymerization viscosity or non-uniform physical properties of the fibrous product. The present invention is based on this finding.

Therefore, it is an object of the present invention to provide a process for preparing an elastic fiber having a high modulus and superior resistance to heat and alkali under mild process conditions.

In accordance with one aspect of the present invention, there is provided a process for preparing an elastic fiber comprising the steps of: adding 1~20% by weight of a cellulose acetate to a polyurethane or polyurethaneurea solution, based on the total weight of the polyurethane or polyurethaneurea, to obtain a spinning solution; ripening the solution for a predetermined period of time; and spinning the ripened solution.

In accordance with another aspect of the present invention, there is provided a high modulus and highly alkali and heat resistant elastic fiber, which is prepared by the process.

Hereinafter, the present invention will be explained in more detail.

The polyurethane or polyurethaneurea solution for use in the present invention is obtained by procedures known in the art. For example, an organic diisocyanate is reacted with a polymeric diol to form a polyurethane precursor. After the
5 polyurethane precursor is dissolved in an organic solvent, the resulting precursor solution is reacted with a diamine for chain extension. The chain extension reaction is terminated by using a monoamine to obtain the polyurethane or polyurethaneurea solution.

Examples of organic diisocyanates usable in the present invention include
10 diphenylmethane-4,4'-diisocyanate, hexamethylenediisocyanate, toluenediisocyanate, buthlyenediisocyanate, hydrogenated p,p-methylenediisocyanate, and the like. As the polymeric diol, there may be used, for example, polytetramethyleneether glycol, polypropyleneglycol, or polycarbonatediol, all of which preferably have a number-average molecular weight of 1,750 to 2,050. Meanwhile, the diamine employed as a
15 chain extender may be ethylenediamine, propylenediamine, hydrazine, or the like, and the monoamine employed as a chain terminator may be diethylamine, monoethanolamine, dimethylamine, or the like. Examples of suitable organic solvents that can be used to obtain the polymer solution include, but are not particularly limited to, N,N'-dimethylformamide, N,N'-dimethylacetamide,
20 dimethylsulfoxide, and the like.

If necessary, the polyurethane or polyurethaneurea solution may further contain at least one additive selected from dulling agents, UV stabilizers, antioxidants, NO_x gas anti-yellowing agents, anti-adhesion agents, dyeing promoters, and anti-chlorine agents.

25 According to the process of the present invention, 1~20% by weight of the cellulose acetate is added to the polyurethane or polyurethaneurea solution, based on the total weight of the polymer, and the mixture is homogeneously stirred to obtain a spinning solution. If the amount of the cellulose acetate added is below 1% by weight, the addition effect is negligible. On the other hand, if the amount of the
30 cellulose acetate exceeds 20% by weight, homogeneous mixing is difficult. The cellulose acetate may be cellulose diacetate or cellulose triacetate, and preferably has a degree of acetylation of about 28% to about 72%. The spinning solution is ripened by allowing it to stand at 30°C~70°C for 28~38 hours, and is then spun to prepare the

final elastic fiber having a high modulus and superior resistance to heat and alkali. According to a study undertaken by the present inventors, since the homogeneous stirring of the cellulose acetate as an additive and the polymer solution and the ripening enable formation of urethane, urea and hydrogen bonds between the polymer and the cellulose acetate, the steps have a direct influence on the increase of the modulus and improvement in the resistance to alkali and heat of the electric yarn. Accordingly, it is important to set optimized conditions for the steps. According to the process of the present invention, the cellulose acetate is dissolved in the same organic solvent as that used to obtain the polymer solution, the resulting solution is homogeneously stirred for 7~8 hours, and then the homogeneous solution is added to the polymer solution. Thereafter, the resulting mixture is homogeneously stirred for at least 2 hours. At this time, the stirring time is extended by 30 minutes with increasing percentage of the cellulose acetate added. After stirring, the mixture of the cellulose acetate and the polymer solution is ripened for about 28~38 hours, and is then spun through a spinning nozzle to prepare the final elastic fiber.

The constitution and effects of the present invention will be described in more detail with reference to the following specific examples and comparative examples. However, these examples are given for the purpose of illustration and are not to be construed as limiting the scope of the invention.

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Best Mode for Carrying Out the Invention

Example 1

518g of diphenylmethane-4,4'-diisocyanate, and 2,328g of polytetramethyleneetherglycol having a number-average molecular weight of 1,800 were reacted with each other at 85°C for 90 minutes with stirring to form a polyurethane precursor containing isocyanate groups at both terminal positions. The polyurethane precursor was allowed to cool to room temperature, and was then dissolved in 4,643g of N,N'-dimethylacetamide to obtain a polyurethane precursor solution. Thereafter, 54g of propylenediamine, and 9.1g of diethylamine were dissolved in 1,889g of N,N'-dimethylacetamide, and the resulting solution was added to the polyurethane precursor solution at 10°C or less to produce a segmented polyurethaneurea solution.

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To the polymer solution was added a UV stabilizer, an antioxidant, a NO_x gas anti-yellowing agent, a dyeing promoter, a magnesium-based anti-adhesion agent and a titanium-based dulling agent. The resulting solution was homogeneously stirred. To the homogeneous solution was added a solution of 1 wt% of cellulose diacetate having a degree of acetylation of 45% in N,N'-dimethylacetamide, based on the solid content of the polymer solution. Thereafter, the mixture was defoamed for 2 hours, and ripened at 40°C for 35 hours to obtain a spinning solution. The spinning solution was dry spun at a spinning temperature of 250°C, and drawn at a draw ratio of 1.3 to prepare a polyurethaneurea elastic fiber having a thickness of 40 deniers. The polyurethaneurea elastic fiber thus prepared was wound.

590 polyurethaneurea elastic fibers were warped, knitted with 50-denier polyester yarns, and dyed to manufacture a velvet fabric. The velvet fabric was subjected to burning-out and printing processes. The yarns of the velvet fabric were measured for resistance to heat and alkali, and the velvet fabric was measured for power retention. In addition, the raised state of the fabric before burning-out, and occurrence of holes on the fabric after burning-out were evaluated. The results are shown in Table 1.

Example 2

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that a solution of 5% by weight of cellulose diacetate (degree of acetylation: 45%) in N,N'-dimethylacetamide was added to the polymer solution, based on the solid content of the polymer solution, and homogeneous stirring was carried out for 4 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

Example 3

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that a solution of 10% by weight of cellulose diacetate (degree of acetylation: 45%) in N,N'-dimethylacetamide was added to the polymer solution, based on the solid content of

the polymer solution, and homogeneous stirring was carried out for 6.5 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

5

Example 4

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that a solution of 15% by weight of cellulose diacetate (degree of acetylation: 45%) in N,N'-
10 dimethylacetamide was added to the polymer solution, based on the solid content of the polymer solution, and homogeneous stirring was carried out for 9.5 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

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Example 5

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that a solution of 20% by weight of cellulose diacetate (degree of acetylation: 45%) in N,N'-
20 dimethylacetamide was added to the polymer solution, based on the solid content of the polymer solution, and homogeneous stirring was carried out for 12 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

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Example 6

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that cellulose triacetate (degree of acetylation: 65%) was dissolved in N,N'-dimethylacetamide at
30 110°C for 30 minutes and then the resulting solution was added to the polymer solution so that the amount of the cellulose triacetate was 1% by weight, based on the solid content of the polymer solution, and homogeneous stirring was carried out for 2 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and

was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

Example 7

5 A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that cellulose triacetate (degree of acetylation: 65%) was dissolved in N,N'-dimethylacetamide at 110°C for 30 minutes and then the resulting solution was added to the polymer solution so that the amount of the cellulose triacetate was 5% by weight, based on the solid
10 content of the polymer solution, and homogeneous stirring was carried out for 2 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

15 Example 8

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that cellulose triacetate (degree of acetylation: 65%) was dissolved in N,N'-dimethylacetamide at 110°C for 30 minutes and then the resulting solution was added to the polymer solution
20 so that the amount of the cellulose triacetate was 10% by weight, based on the solid content of the polymer solution, and homogeneous stirring was carried out for 2 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

25 Example 9

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that cellulose triacetate (degree of acetylation: 65%) was dissolved in N,N'-dimethylacetamide at
30 110°C for 30 minutes and then the resulting solution was added to the polymer solution so that the amount of the cellulose triacetate was 15% by weight, based on the solid content of the polymer solution, and homogeneous stirring was carried out for 2 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and

was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

Example 10

5 A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that cellulose triacetate (degree of acetylation: 65%) was dissolved in N,N'-dimethylacetamide at 110°C for 30 minutes and then the resulting solution was added to the polymer solution so that the amount of the cellulose triacetate was 20% by weight, based on the solid
10 content of the polymer solution, and homogeneous stirring was carried out for 2 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

15 Comparative Example 1

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that a solution of 25% by weight of cellulose diacetate (degree of acetylation: 45%) in N,N'-
20 dimethylacetamide was added to the polymer solution, based on the solid content of the polymer solution, and homogeneous stirring was carried out for 9.5 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

25 Comparative Example 2

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that cellulose triacetate (degree of acetylation: 65%) was dissolved in N,N'-dimethylacetamide at 110°C for 30 minutes and then the resulting solution was added to the polymer solution so that the amount of the cellulose triacetate was 25% by weight, based on the solid
30 content of the polymer solution, and homogeneous stirring was carried out for 2 hours. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric

were measured and evaluated, and the results are shown in Table 1.

Comparative Example 3

A polyurethaneurea elastic fiber having a thickness of 40 deniers was prepared and wound in the same manner as in Example 1, except that cellulose diacetate was not added. Thereafter, a velvet fabric was manufactured in the same manner as in Example 1, and was then subjected to burning-out and printing. The properties of the velvet fabric were measured and evaluated, and the results are shown in Table 1.

Table 1

Exa. No.	Evaluation of Yarns			Evaluation of Fabrics			Remarks
	Modulus (g)	Heat resistance ₁₎	Alkali resistance ₂₎	Power retention ₃₎	Raised state before burning-out ₄₎	Occurrence of holes on fabric after burning-out ₅₎	
Ex. 1	5.4	47%	117 min.	39.7%	Δ	Occurred	
Ex. 2	6.2	65%	130 min.	47.3%	O	Few occurred	
Ex. 3	5.9	63%	133 min.	44.5%	O	Few occurred	
Ex. 4	5.7	54%	125 min.	42.7%	Δ	Few occurred	
Ex. 5	5.4	48%	121 min.	40.4%	Δ	Occurred	
Ex. 6	5.5	50%	129 min.	41.6%	Δ	Few occurred	
Ex. 7	6.5	69%	145 min.	48.8%	O	Few occurred	
Ex. 8	6.1	67%	152 min.	46.0%	O	Few occurred	
Ex. 9	5.8	58%	136 min.	42.9%	Δ	Few occurred	
Ex. 10	5.3	53%	135 min.	41.1%	Δ	Few occurred	
Comp. Ex. 1	4.9	42%	115 min.	38.5%	Δ	Many occurred	Homogeneous mixing was difficult
Comp. Ex. 2	4.6	45%	127 min.	39.0%	Δ	Few occurred	Homogeneous mixing was difficult
Comp. Ex. 3	5.1	45%	113 min.	39.2%	Δ	Many Occurred	

Notes:

¹⁾ The heat resistance of the yarns was evaluated by the following procedure: A yarn sample is stretched by 100%, and wet-heat treated at 130°C for 1 hour. The heat treatment is repeated five times (5 cycles). The heat resistance of the yarn sample is expressed as percentage of the difference between the length ("initial length") of the yarn sample before the heat treatment and that ("download value") after the fifth cycle.

²⁾ The alkali resistance of the yarns was evaluated by the following procedure: A yarn sample is dipped in an 25% aqueous NaOH (25wt%) solution, and heated to 150°C. The alkali resistance of the yarn sample is expressed as the time taken for the yarn sample to decompose.

5 ³⁾ The power retention of the fabrics was measured by the following procedure: A finally processed fabric is cut into a fabric sample (1 inch x 30cm). The fabric sample is held by grips from Instron Co., so that the length of the fabric sample for measurement is 20cm. The holding is repeated five times (5 cycles). The power retention of the fabric sample is expressed as percentage of the difference between the
10 length (“upload value”) of the fabric sample after the first cycle and that (“download value”) after the fifth cycle.

⁴⁾ The raised state before burning-out was evaluated by visually examining the degree of uprightness of raised yarns after the velvet fabrics were subjected to shearing and background dyeing. When the raised yarns were straight and upright, the raised
15 state was judged as “O”. On the other hand, when some of the raised yarns were flattened, the state was judged as “Δ”.

⁵⁾ The occurrence of holes on the fabrics after burning-out was evaluated by visual examination.

As shown in Table 1, since the process of the present invention enables
20 uniform management of polymerization and spinning viscosity, the elastic fibers show uniform physical properties, a high modulus, and improved resistance to heat and alkali. Accordingly, when the process of the present invention is applied to general velvet fabrics or special velvet fabrics requiring continuous burning-out and printing, it has advantages that no degradation of elastic fibers arises, and the state of raised yarns
25 and fabrics is stably maintained.

Although the preferred embodiments of the present invention have been disclosed for illustrative purposes, those skilled in the art will appreciate that various modifications, additions and substitutions are possible, without departing from the scope and spirit of the invention as disclosed in the accompanying claims.

Claims:

1. A process for preparing an elastic fiber, comprising the steps of:
adding 1~20% by weight of a cellulose acetate to a polyurethane or
5 polyurethaneurea solution, based on the total weight of the polyurethane or
polyurethaneurea, and homogeneously stirring the mixture to obtain a spinning
solution;
ripening the spinning solution; and
spinning the ripened solution.
- 10
2. The process according to claim 1, wherein the cellulose acetate is cellulose
diacetate or cellulose triacetate having a degree of acetylation of 28%~72%.
3. The process according to claim 1 or 2, wherein the polyurethane or
15 polyurethaneurea solution is obtained by reacting an organic diisocyanate with a
polymeric diol to form a polyurethane precursor, dissolving the polyurethane precursor
in an organic solvent, and reacting the precursor solution with a diamine and a
monoamine sequentially.
- 20
4. The process according to claim 3, wherein the organic diisocyanate is
selected from the group consisting of diphenylmethane-4,4'-diisocyanate,
hexamethylenediisocyanate, toluenediisocyanate, buthlyenediisocyanate, and
hydrogenated p,p-methylenediisocyanate; the polymeric diol is selected from the group
25 consisting of polytetramethyleneether glycol, polypropyleneglycol, and
polycarbonatediol; the diamine is selected from the group consisting of
ethylenediamine, propylenediamine, and hydrazine; and the monoamine is selected
from the group consisting of diethylamine, monoethanolamine, and dimethylamine;
and the organic solvent is selected from the group consisting of N,N'-
30 dimethylformamide, N,N'-dimethylacetamide, and dimethylsulfoxide.
5. The process according to claim 1 or 2, wherein the spinning solution further
contains at least one additive selected from dulling agents, UV stabilizers,
antioxidants, NO_x gas anti-yellowing agents, anti-adhesion agents, dyeing promoters,

and anti-chlorine agents.

5 6. The process according to claim 1 or 2, wherein after the addition of the cellulose acetate, the homogeneous stirring is carried out for at least 2 hours, and the spinning solution is ripened by allowing it to stand at 30°C~70°C for 28~38 hours,


7. An elastic fiber prepared by the process according to claim 1 or 2.

8. A velvet fabric manufactured using the elastic fiber according to claim 7.

INTERNATIONAL SEARCH REPORT

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Electronic data base consulted during the international 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	US 4342847 A (BAYER AKTIENGESELLSCHAFT) 03 August 1982(03.08.1982) (Family None) see the whole document	1-8
A	US 6156842 A (DOW CHEMICAL CO.) 05 December 2000 (05.12.2000) (Family None) see the whole document	1-8
A	US 6207274 A (INTERNATIONAL FLAVORS & FRAGRANCES INC.) 27 March 2001 (27..3.2001) (Family None) see the whole document	1-8
A	US 5911930 A (MONSANTO CO.) 15 June 1999 (15.06.1999) (Family None) see the whole document	1-8
<input type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.		
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