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(54) **A process for the treatment of a fibrous structure.**

(57) A process for the treatment of a fibrous structure is provided. The fibrous structure comprises two components or more, at least one component thereof comprises a polyester containing SO_3M groups wherein M represents hydrogen or a metal. The process includes the removal of said SO_3M groups containing polyester preferably by an alkali treatment. Prior to said removal treatment a degrading pre-treatment of the fibrous structure is provided. The pre-treatment uses a degrading agent, which selectively deteriorates the SO_3M groups containing polyester, the removal of which is intended.

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A process for the treatment of a fibrous structure

The present invention relates to a process for the treatment of a fibrous structure containing polyester.

According to the art the treatment of a polyester fibrous structure with an alkaline reagent (in the following termed as "alkali treatment") developes a soft handle (in the meaning of grip and/or feeling) of said polyester fibrous structure by partial dissolution and removal of fiber material. Said alkali treatment is primarily important for the manufacture of polyester fibrous structures comprising essentially one type of polyester. Furthermore, it is known that the treatment of a fiber mixture consisting of alkali easily soluble fibers and of alkali hardly soluble fibers with alkali can lead to an excellent soft handle by dissolution and removal of the easily soluble fibers. However, the latter treatment does not only need

considerably long time in order to completely dissolve the easily soluble fibers, but also may attack the hardly soluble fibers in some extent; especially in case of hardly soluble fibers comprising polyester said alkali treatment may lead to an unexpected deterioration of the physical properties of the hardly soluble polyester fibers. On the other hand, if somebody tries to shorten the period of treatment by making the easily soluble fibers still more soluble in alkali the resulting very easily soluble fibers cannot withstand the conventional conditions during mixing, blending, knitting and weaving processes due to a deterioration of their physical properties. Furthermore, said very easily soluble fibers suffer from certain difficulties during the fiber spinning (extruding) process. At present, this latter method is not practically applied, though an outstanding soft handle can be obtained.

The alkali treatment of conjugated (composite) fibers consisting of an alkali easily soluble component and of an alkali hardly soluble component in order to remove one component thereof and to obtain micro (ultra) fine and/or special shaped fibers rises a still more serious problem, since those fine fibers which are designated to remain within the alkali treated fibrous product are very often easily dissolved.

It is an object of the present invention to provide a process for the treatment of a fibrous structure which structure comprises two components or more at least one component thereof is intended to be removed, which treatment includes the removal of said component(s) , and which process facilitates the removal of said component(s) , avoids any significant deterioration of the physical properties of the remaining component(s) and/or results in a better handle of the final product.

It is a further object of the present invention to provide said respective process for the treatment of a fibrous structure which comprises a mixture of individual fibers consisting of different materials.

It is a further object of the present invention to provide said respective process for the treatment of a fibrous structure which contains multi-component fibers, for example so-called island-in-the-sea type fibers.

It is a still further object of the present invention to provide said respective process for the treatment of a fibrous structure in order to remove at least one component thereof, which represents a polyester.

As a result of intensive studies by the inventors to solve the above-mentioned problems and objects a process has been established which allows to selectively dissolve only the easily soluble component, especially an easily soluble polyester in a short time. Therefore, the foregoing elucidated problems and objects are solved or greatly reduced by the present invention.

In order to give a summary, the present invention provides a process for the treatment of a fibrous structure which structure comprises two components or more, at least one component thereof is a polyester containing SO_3M groups wherein M represents hydrogen or a metal, which process includes a pre-treatment of the fibrous structure with a degrading agent for said certain polyester prior to the further treatment for the removal of said certain polyester preferably by a treatment with an alkaline reagent (alkali treatment).

The process according to the present invention results

not only in a soft and bonny polyester fibrous structure with sufficient resilience but also enhance this the efficiency of the alkali treatment.

Further objects, features and advantages of the present invention will be apparent from the following detailed description of preferred embodiments thereof taken in connection with the accompanying drawing wherein:

Figures 1 to 9 present cross-section views of exemplary conjugated fibers suited for the treatment by the process of the present invention;

Figures 10 to 12 present cross-section views of the product obtained by removal of the sea-component of the conjugated fiber according to figure 3.

According to the present invention the term "fibrous structure" is intended to include the whole fiber itself and processed goods such as yarn, staple fiber, tow, top, woven fabric, knitted fabric, and non-woven fabric, made from those fibers. Every type of said fibrous structure may contain finishing agents such as silicone resin, melamine resin and urethane resin. According to a distinctive feature of the present invention the fibrous structure comprises two components or more at least one thereof is a polyester containing SO_3M groups wherein M represents hydrogen or a metal. The arrangement of said components within the fibrous structure includes mixtures of these separately spun components obtained by subsequent mixing or blending processes or the like. Another type of arrangement is a fibrous structure formed from conjugated fibers consisting of said two or more components one of which is a SO_3M group containing polyester. A further type of arrangement comprises a fibrous structure obtained by mixing conjugated fibers and ordinary fibers. In other words, there

are several types of the fibrous structure containing as the removable component a SO_3M group containing polyester together with one or more other component(s) and the present invention is not limited to any particular arrangement of said components.

In the fibers having a cross-section according to one of the figures 1 to 9 the component A represents the easily soluble component which should be removed by the alkali treatment. In the special case of conjugated fibers having the typical island-in-the-sea type structure (for example according to figures 1, 2, 5 and 9), the sea component A is generally the easily soluble component.

The easily soluble polyester component which should be removed by the alkali treatment is a polyester containing SO_3M groups wherein M represents a metal, particularly an alkali metal or an alkaline earth metal or the hydrogen atom. Having in mind both, the spinnability of the fibers forming the fibrous structure and the effectiveness of the alkali treatment after the pre-treatment for degrading of the polyester component said removable polyester component should be polyethylene terephthalate copolymerized with preferably 1-15 molar % more preferably with 3-5 molar % of 5-(sodium sulfo)isophthalic acid. The one or more other component(s) forming the fibrous structure can be selected from synthetic fibers such as polyester, polyamide and polyacryl fibers or from semi-synthetic fibers such as acetate fibers, regenerated fibers such as rayon fibers and/or from natural fibers such as cotton, wool and silk fibers, the material of all of said fibers is rather more hardly soluble with respect to the alkaline reagent than the easily soluble polyester component which should be removed by the alkali treatment.

The benefits of the present invention are particularly remarkable in connection with polyester fibers which actually are hardly soluble but not highly resistant with respect to the alkaline reagent and the conditions used in the alkali treatment.

The terms "easily soluble" and "hardly soluble" as used herein describe the solubility of the components forming the fibrous structure at the situation before the removal treatment with alkaline reagent but after the pre-treatment with the degrading agent.

The alkali treatment results in a hydrolysis of the selected polyester with an alkaline reagent. Good results may be obtained with an alkali treatment using the following conditions: (1) maintaining the fibrous structure for 30-120 min within a boiling aqueous solution of an alkaline reagent such as for example sodium hydroxide, or (2) impregnating the fibrous structure with an alkaline reagent and maintaining the impregnated fibrous structure for 10-30 hours at 40-60°C or (3) impregnating the fibrous structure with an alkaline reagent and allowing the action of dry heat or superheated steam for 1-5 min at 130-200°C. Without intending any limitation, the alkaline reagent for the alkali treatment can be selected from typical and known alkaline substances such as alkali metal hydroxides for example sodium hydroxide, potassium hydroxide, and the like or alkaline earth metal hydroxide for example calcium hydroxide, barium hydroxide and the like or other basic salts for example sodium carbonate, potassium carbonate and the like. Besides the above-stated and preferably used conditions for the alkali treatment any other suited method for removal of at least one component of the fibrous structure can be applied, provided stable working conditions are maintained and the remaining fiber(s) of the treated fibrous structure remains essentially uneffected.

A key feature of the process according to the present invention is the pre-treatment of the fibrous structure prior to the mentioned alkali treatment. The intention of said pre-treatment is a degrading of the selected polyester which should be removed afterwards. The degrading may yield in a lower average molecular weight of said certain polyester and/or may in any other way promote the effect of the alkali treatment. The degrading agents suited for the pre-treatment of polyester in the process of the present invention include for example amines such as ethylenediamine, ethylenetriamine and the like, further monoethanolamine and similar compounds, further zinc salts such as zinc chloride, zinc sulfate, zinc nitrate and the like, further oxidizing agents such as hydrogen peroxide, sodium hypochlorit, sodium chlorit, and further typical acidic compounds, for example inorganic acids such as hydrochloric acid, sulfuric acid, nitric acid, phosphoric acid and the like or organic acids, for example formic acid, oxalic acid and the like. Especially acidic compounds have proven to be particularly appropriate, because they selectively degrade the SO_3M groups containing polyester. Therefore said acidic compounds are preferably used as degrading agents in the pre-treatment of the process according to the present invention.

The degrading pre-treatment can be effected in several ways, for example by the following processes:

(1) By immersing the fibrous structure for about 10-120 min into a boiling aqueous solution containing the degrading agent;

(2) by adding the degrading agent to the fibrous structure and the subsequent action of saturated vapor for about 1 to 30 min at 100-130°C;

(3) by adding the degrading agent to the fibrous structure and the subsequent action of dry heat or

superheated steam for about 1-10 min at 130-220°C;

(4) or by adding the degrading agent to the fibrous structure followed by the continued reaction for 10-30 hours at 40-60°C.

The degrading pre-treatment is by no way limited to the above-listed methods. Indeed any suited process can be applied which results in a lowering of the average molecular weight of the selected polyester by the action of the degrading agent(s). Regarding the treatment with acid in a boiling aqueous solution preferred conditions look for a treatment at a pH-value below 2 for about 30 min at 110-140°C or at a pH-value of about 3 for 60 min at 110-140°C. The addition of selected additives to the treating bath for example such as carrier agent, surfactant agent or quaternary ammonium salt can even improve the results of the action of the degrading agent.

In the following there are stated some results of the characteristic features of the process according to the present invention in comparison with conventional processes:

(1) According to a conventional process the treatment of a fibrous structure comprising blended yarn or mixed filament yarn in order to remove the desired component needs a rather long treatment period which often yields to an substantial deterioration of the remaining fibers. To the contrary the process according to the present invention requires a significant shorter treatment period and provides a fibrous structure with a better handle without loss of physical properties of the remaining fibers.

This highly desired result is obtained without any impairing of spinning and/or weaving conditions of the fibers. As stated earlier some very easily soluble fibers can be rapidly removed by an alkali treatment

however the manufacturing conditions of those very easily soluble fibers provides difficulties with respect to stable spinning or weaving conditions. To the contrary, the process according to the present invention does not impair the manufacturing of the fibers, because the distinctive pre-treatment with a selected degrading agent for the polyester promotes the hydrolysis rate in an alkaline environment.

(2) The process according to the present invention proves to be particularly effective for the treatment of island-in-the-sea-type conjugated fiber both components thereof comprises polyester wherein one polyester component is more easily soluble in the alkaline reagent used for alkali treatment. If somebody tries to remove the easily soluble component (A) of island-in-the-sea-type fibers according to figure 3 only the fibers shown in figure 11 can be obtained, because in addition to the component (A) a part of the hardly soluble component (B) is also dissolved during the alkali treatment. To the contrary the process according to the present invention allows a complete removal of the component (A) prior to the hydrolysis of component (B) starts and therefore the inventive process finally leads to fibers having a cross-section according to figure 10. In fact the process according to the present invention allows to maintain the original shape of the island components during the removal of the sea component. Therefore, if an island-in-the-sea-type fiber as shown in figure 1 is treated along the process according to the present invention, the respective independent islands can be obtained separately with minimum damage of the island component in the outside portion thereof. To the contrary, the conventional process sometimes dissolve the outside portion of the island component before the island component located within the inner section of the fiber have

been separated.

(3) The degrading pre-treatment according to the present invention increases the effectiveness of the subsequent alkali treatment, this means the so-called alkali reduction rate of the selected polyester. It is known in the art, that said alkali reduction rate can also be increased by the conventional process, if a quaternary ammonium salt is used during the alkali treatment. The presence of the ammonium salt increases the alkali reduction rates of both components, the polyester intended to be removed and the other component which should remain in the treated fibrous structure. To the contrary the degrading pre-treatment according to the present invention allows to increase selectively the alkali reduction rate of only the one component, which should be removed afterwards. Therefore, the conventional process does not attain the same effect as the process according to the present invention, even if the conventional process uses a quaternary ammonium salt during the alkali treatment.

The following examples are provided for a further illustration of the process according to the present invention. These examples serve only for illustration purposes and are not to be construed as limiting the scope of the present invention.

Example 1.

A fabric in taffeta weave has been woven from island-in-the-sea-type fibers (75 denier, 36 filaments) as shown in figure 3, using said fibers for both warp and weft yarn. The specifications of the used fibers are the following:

Component A: Polyethylene terephthalate copolymerized with 4 molar % of 5-(sodium sulfo) isophthalic acid;

Component B: Polyethylene terephthalate;
Ratio A:B: 30:70

For effecting the degrading pre-treatment said fabric was immersed in a boiling 1 % aqueous sulfuric acid solution for 60 min. Subsequently the alkali treatment was effected in order to completely remove the component A by immersing said pre-treated fabric in a boiling 1.5 % aqueous sodium hydroxide solution for 4 min. After removal from the alkaline bath and drying the fabric shows a weight reduction in the amount of 30.5 %. Thereafter the fabric was dyed using an conventional method. The dyed fabric provided a mild color tone, high water absorption, excellent handle (in the meaning of grip and/or feeling) and was free from the problems originated from yarn slippage and a reduction of yarn tenacity. An investigation of the cross-section of the obtained fibers yields sharp edges such as shown in figure 10.

Comparative example 1:

The fabric as mentioned in example 1 has been treated directly with the boiling 1.5 % aqueous sodium hydroxide solution therefore omitting any degrading pre-treatment with sulfuric acid. The complete removal of the component A took 110 min and yielded a weight reduction of the treated and dried fabric in the amount of 48%. This far higher weight reduction indicates that the alkali treatment does not only remove the component A but also remove in an significant amount the component B.

Comparative example 2:

The fabric according to example 1 has been treated in the boiling 1.5 % aqueous sodium hydroxide solution in

the additional presence of 0.8 % DYK-1125 (a quaternary ammonium salt available from Ippo Co., Ltd.). The component A was completely removed in a short period of 15 min. However the treated and dried fabric shows a weight reduction in the large amount of 65 %, indicating that the component B has been removed in a rather high extent.

The fabrics obtained to comparative examples 1 and 2 have been dyed by an conventional method. The dyed fabrics show a considerable yarn slippage and poor tenacity. The tenacity has been determined with the Elmendorf tearing tester apparatus. The results are the following:

Tenacity of the fabric treated according to		
Process of the present invention	1500 (g)	1100 (g)
Comparative Example 1	800 (g)	500 (g)
Comparative Example 2	400 (g)	200 (g)

The process according to the comparative examples 1 and 2 realize the valuable properties of the inventive treated product in a far less amount. The fibers obtained by the comparative examples 1 and 2 show a fiber cross-section in some extent as shown in figures 11 and 12, indicating that the original shape of component B has been deformed.

Example 2:

From the starting components

20 % staple fiber of polyethylene terephthalate copolymerized with 4 molar % of 5-(sodium sulfo) isophthalic acid, and

80 % wool fiber

blended yarn has been prepared. Said yarn was woven into

a twill structure and from the obtained twill structure a fabric has been manufactured by conventional twilling process.

In order to effect the degrading pre-treatment according to the present invention said fabric was immersed in a boiling 0.5 % aqueous hydrochloric acid solution for 60 min. The subsequent alkali treatment for completely removal of the polyester component was effected by immersing the pre-treated fabric in a boiling 0.1 % aqueous sodium hydroxide solution for 35 min. Thereafter the fabric was dyed by a conventional method. The obtained wool fabric showed an outstanding capability connected with an excellent handle.

Comparative example 3:

The same fabric as mentioned in example 2 was treated directly in the boiling 0.1 % aqueous sodium hydroxide solution for 35 min omitting any degrading pre-treatment with aqueous hydrochloric acid solution. Said alkali treatment was insufficient to remove the polyester component completely. After a conventional dyeing process the obtained dyed fabric do not show the characteristic features of the fabric obtained by the inventive process.

Comparative example 4:

The same fabric as mentioned in example 2 was treated in a boiling 1.5 % aqueous sodium hydroxide solution omitting any degrading pre-treatment with hydrochloric acid solution. The alkali treatment was effected for a sufficient period in order to remove the polyester component completely. The investigation of the weight reduction proved, that said longer alkali treatment removed in addition a significant part of the wool

component. The finally obtained fabric was completely different to the product of the process according to the present invention.

Example 3:

A sample hosiery was prepared by knitting island-in-the-sea-type fibers (225 denier, 24 filaments) having a cross-section as shown in figure 1. The specifications of the used fibers are as follows:

Component A: Polyethylene terephthalate copolymerized with 4 molar % of 5-(sodium sulfo) isophthalic acid;
Component B: Polyethylene terephthalate;
Ratio A:B: 22:78;
Denier of component B in monofilament: 0.2 den;
Component B: 36 filaments.

The degrading pre-treatment of said sample hosiery was effected by immersing said hosiery in a 10 % aqueous phosphoric acid solution for 30 min at 130°C. The subsequent alkali treatment was effected by introducing the pre-treated sample in a boiling 1.5 % aqueous sodium hydroxide solution for 4 min. The combined results of both treatments is a complete removal of the component A and a final product comprising a beatiful hosiery knit consisting of micro fine fibers. The weight reduction amounts 22.2 %. An investigation of the cross-section of the remaining micro fine fibers proved that the island portions have not been reduced substantially; the tenacity of these micro fine fibers amount 730 g/filament.

Comparative example 5:

A further sample of the same hosiery as used in example 3 was treated directly in the boiling 1.5 % aqueous sodium hydroxide solution, therefore omitting any degrading pre-treatment with aqueous phosphoric acid solution. In order to succeed in a complete removal of the component A an alkali treatment period as long as 150 min was necessary. Said long alkali treatment yielded in a weight reduction of 44.0 %, proving a significant removal of polyethylene terephthalate microfine fibers. In fact a weight lost of the micro fine fibers in an average value of approximately 30 % has been established. The tenacity of the remaining micro fine fibers has been decreased as low as 330 g/filament.

In addition, the micro fine fibers obtained by the process of the present invention according to the example 3 showed a uniform fiber thickness of 0.2 den, while the respective micro fine fibers obtained according to the comparative example 5 showed a large fluctuation of fiber thickness in the range of 0.1-0.2 den.

Although the present invention has been described with reference to the specific details of particular embodiments it will be obvious to those skilled in the art that various changes and modifications may be made therein without departing from the invention, and it is, therefore, aimed in the appended claims to cover all such changes and modifications as fall within the true spirit and scope of the present invention.

Claims:

1. A process for the treatment of a fibrous structure which structure comprises two components or more at least one component thereof comprises a polyester, in order to remove the polyester component, characterized in that the removable polyester component contains SO_3M groups wherein M represents hydrogen or a metal, and prior to that removal treatment there is provided a pre-treatment of the fibrous structure with a degrading agent for said SO_3M groups containing polyester.

2. A process according to claim 1, wherein said removal treatment is effected by the action of an alkaline reagent.

3. A process according to claim 1 or 2, wherein said degrading agent comprises an acid medium.

4. A process according to claim 3, wherein said acid medium is an inorganic acid.

5. A process according to claim 4, wherein said inorganic acid is used in the form of a diluted boiling aqueous acidic solution.

6. A process according to anyone of the claims 1 to 5, wherein the fibrous structure comprises a mixture of two or more types of polyester fibers consisting of different polyester compositions.

7. A process according to anyone of the claims 1 to 5, wherein the fibrous structure comprises multi-component fibers made from two or more polyester components having different polyester compositions.

8. A process according to claim 7, wherein the multi-component fiber provides a plurality of cores within the fiber cross-section, and the SO_3M groups containing polyester forms the material between said cores.

9. A process according to claim 7 or 8, wherein the multi-component fiber comprises at least 5 cores within the fiber cross-section.

10. A process according to claim 7 or 8, wherein the multi-component fiber comprises at least 10 cores within the fiber cross-section.

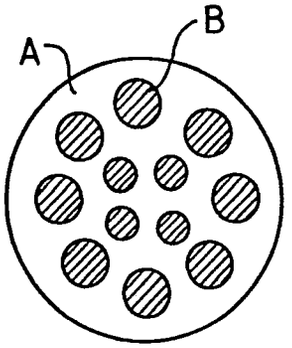

Fig. 1

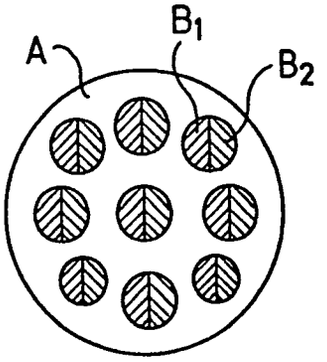

Fig. 2

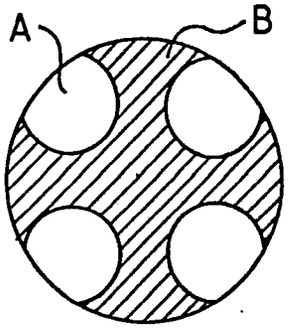

Fig. 3

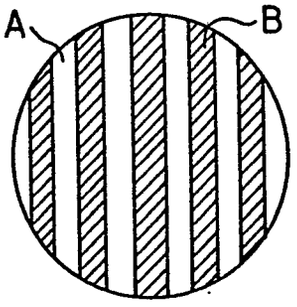

Fig. 4

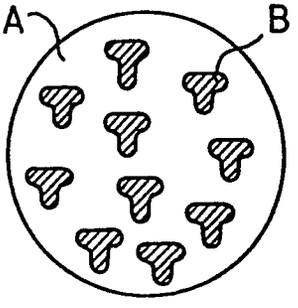

Fig. 5

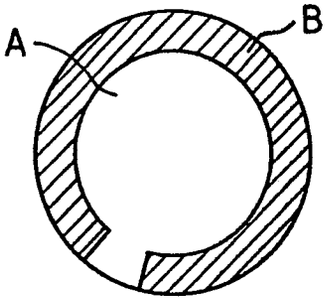

Fig. 6

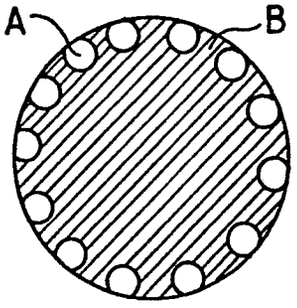

Fig. 7

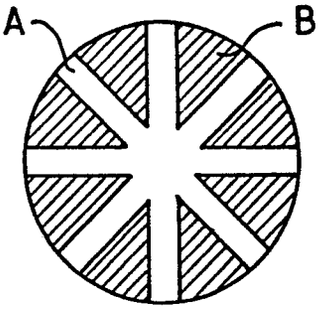

Fig. 8

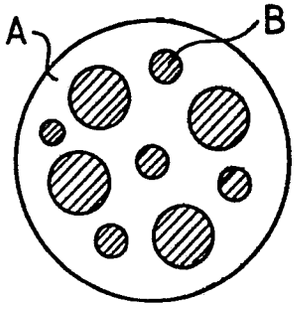

Fig. 9

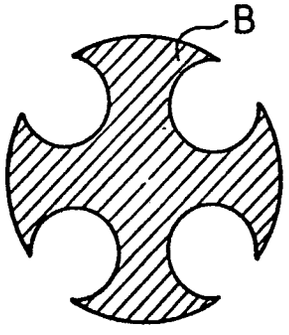

Fig. 10

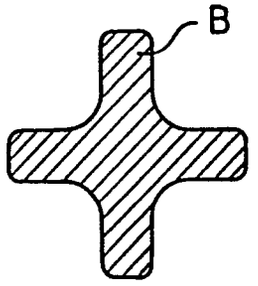

Fig. 11

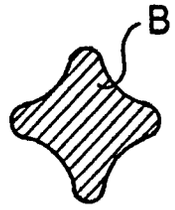

Fig. 12

European Patent Office

0064568
Application number
EP 81 10 3658

EUROPEAN SEARCH REPORT

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. 3)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
X	PATENTS ABSTRACTS OF JAPAN, vol. 4, no. 89, June 25, 1980, (C-16)(571) & JP - A - 55 51 820 (TORAY), 15. 04.1980 * the whole abstract *	1,2,6-10	D 01 F 11/08 D 01 F 8/14
X	PATENTS ABSTRACTS OF JAPAN, vol. 4, no. 124, September 2, 1980, (C-23) (606) & JP - A - 55 76 110 (TORAY), 09.06.1980 * the whole abstract *		TECHNICAL FIELDS SEARCHED (Int.Cl. 3) D 01 F 11/08 D 01 F 8/14 D 01 F 8/04 D 01 D 5/36
A	GB - A - 1 306 974 (TORAY)		
A	US - A - 4 118 529 (KURARAY)		
A	GB - A - 1 458 472 (TORAY)		

CATEGORY OF CITED DOCUMENTS

X: particularly relevant if taken alone
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P: intermediate document
T: theory or principle underlying the invention
E: earlier patent document, but published on, or after the filing date
D: document cited in the application
L: document cited for other reasons

&: member of the same patent family, corresponding document

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
The Hague	15.01.1982	VAN GOETHEM