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NOTICE OF ENTITLEMENT
(To be filed before acceptance)

We, **Rhône-Poulenc Viscosuisse S.A.**, of Gerliswilstrasse, 6020 Emmenbrücke, Switzerland, being the applicant in respect of Application No. 69358/94 state the following:-

The Person nominated for the grant of the patent has entitlement from the actual inventors by virtue of their employment by the applicant at the date of the invention.

The person nominated for the grant of the patent is the applicant of the application listed in the declaration under Article 8 of the PCT.

The basic application listed in the declaration made under Article 8 of the PCT is the first application made in a Convention country in respect of the invention.

Rhône-Poulenc Viscosuisse S.A.

By our Patent Attorneys,
WATERMARK PATENT & TRADEMARK ATTORNEYS


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Louis C. Gebhardt
Registered Patent Attorney

12 May 1997



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- (54) Title
PROCESS FOR PRODUCING SOIL-REPELLENT AND ABRASION-RESISTANT MONOFILAMENTS AND THEIR USE
- International Patent Classification(s)
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- (71) Applicant(s)
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- (56) Prior Art Documents
EP 269023
WO 93/18086
- (57) Claim

1. A process for producing soil-repellent and abrasion-resistant monofilaments for screen fabrics, having a diameter of between 0.1 to 1.0 mm, from linear silicon-modified polyethylene terephthalate, characterized in that 0.15 to 5 weight-% of polydialkyl siloxane are inserted into the chain of the polymer by condensation and further characterized in that the product monofilament has an Si content of 0.05 to 2.0 weight-% referred to the weight of the monofilament.

3. A soil-repellent and abrasion-resistant monofilament produced by the process of claim 1 or 2, wherein the monofilament has a diameter of between 0.1 to 1.0 mm, consisting of a silicon-modified polyethylene terephthalate, characterized in that the monofilament has a tenacity of at least 36 cN/tex, an elongation of <44% referred to the unstretched filament, and simultaneously a soiling index of <10 after five soiling cycles.



<p>(51) Internationale Patentklassifikation ⁶ : D01F 6/84</p>	<p>A1</p>	<p>(11) Internationale Veröffentlichungsnummer: WO 95/01471 (43) Internationales Veröffentlichungsdatum: 12. Januar 1995 (12.01.95)</p>
<p>(21) Internationales Aktenzeichen: PCT/IB94/00183 (22) Internationales Anmeldedatum: 28. Juni 1994 (28.06.94) (30) Prioritätsdaten: 1990/93-8 2. Juli 1993 (02.07.93) CH (71) Anmelder (für alle Bestimmungsstaaten ausser US): RHONE-POULENC VISCOSUISSE S.A. [CH/CH]; Patentabteilung RP, D-6020 Emmenbrücke (CH). (72) Erfinder; und (75) Erfinder/Anmelder (nur für US): BARIS, Halim [TR/CH]; Gesegnetmattstrasse 2, D-6006 Luzern (CH). FLEURY, Etienne [FR/FR]; 31, rue du Lac, F-69540 Irigny (FR). (74) Anwalt: HERRMANN, Peter; Rhone-Poulenc Viscosuisse S.A., Patentabteilung RP, CH-6020 Emmenbrücke (CH).</p>	<p>(81) Bestimmungsstaaten: AU, BR, CA, FI, JP, US, europäisches Patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Veröffentlicht Mit internationalem Recherchenbericht. Vor Ablauf der für Änderungen der Ansprüche zugelassenen Frist. Veröffentlichung wird wiederholt falls Änderungen eintreffen.</p>	

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(54) Title: PROCESS FOR PRODUCING SOIL-REPELLENT AND ABRASION-RESISTANT MONOFILAMENTS AND THEIR USE

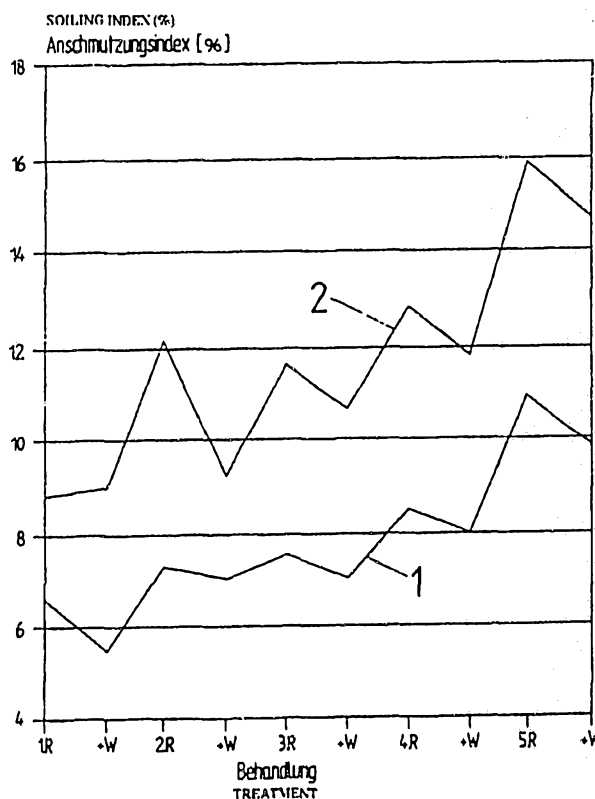
(54) Bezeichnung: VERFAHREN ZUR HERSTELLUNG VON SCHMUTZABWEISENDEN UND ABRASIONSBESTÄNDIGEN MONOFILAMENTEN UND DEREN VERWENDUNG

(57) Abstract

A process is disclosed for producing soil-repellent and abrasion-resistant monofilaments having 0.1 to 1.0 mm diameter from a linear PET/polydialkylsiloxane, in which the Si component with 0.15 to 2.0 % by weight Si content is incorporated into the polymer chain. The soil-repellent and abrasion-resistant monofilament has at least 36 cN/tex solidity, less than 44 % elongation in relation to the non-elongated filament, and at the same time a soiling index lower than 10 after 5 soiling cycles. This monofilament is suitable for producing screening fabrics for paper machines, fishing lines and fishing nets.

(57) Zusammenfassung

In einem Verfahren zur Herstellung von schmutzabweisenden und abrasionsbeständigen Monofilamenten mit einem Durchmesser von 0,1 bis 1,0 mm, aus einem linearen PET/Polydialkylsiloxan, wird die Si-Komponente mit einem Si-Gehalt von 0,15 bis 2,0 Gew.-% in die Polymerkette eingebaut. Das schmutzabweisende und abrasionsbeständige Monofilament weist eine Festigkeit von wenigstens 36 cN/tex auf, bei einer Dehnung von < 44 %, bezogen auf das ungedehnte Filament, und gleichzeitig einen Anschmutzungsindex von < 10 nach 5 Anschmutzungszyklen und ist zur Herstellung von Siebgeweben für Papiermaschinen, von Angelschnüren und Fischereinetzen vorgesehen.



Process for Producing Soil Repellent and Abrasion-Resistant
Monofilaments and Their Use

The invention relates to a process for producing soil-repellent and abrasion-resistant monofilaments of a diameter of between 0.1 to 1.0 mm from linear polyethylene terephthalate/polydialkyl siloxane for producing screen fabrics, as well as monofilaments for their use.

"Soil-repellent" properties for screens of paper-making machines are understood to be in connection with wet soiling, which also presupposes hydrolysis resistance. The soil repellence is intended to be used for extending the cleaning cycles of a screen, wherein cleaning is performed under a high-pressure water stream and, if required, by rubbing with a brush. Soil-repellent monofilaments are worked into industrial fabrics which are employed in the wet portion, in the pressing as well as the drying portion of a paper-making machine. Such screens are subjected to moisture and temperatures far above 100°C, as well as increased mechanical stress during cleaning. Pure polyester monofilaments have disadvantages based on their tendency to soil, their susceptibility to hydrolysis and their relative brittleness. An improvement in their hydrolysis resistance was achieved by adding additives in the form of stabilizers. An increase in soil repellency was attempted, besides the addition of additives, also by suitable finishing treatments of the filament and aftertreatment of the fabric.

"Abrasion-resistant" monofilaments are understood to be those monofilaments with which no actual weight loss could be

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noted during fibrillation testing as well as only little or no fibrillations.

It was found that co-polyesters modified with siloxanes were excellently suited for the above field of employment, in spite of a concern because of their low hydrolysis resistance, because the characteristic of their surface can be easily modified thanks to improved sliding properties and smaller coefficients of friction, by means of which the brittleness is clearly reduced and soil repellency considerably improved. The flat shaped articles produced can be easily treated again to improve their chemical properties, such as hydrolysis resistance.

Polyester/polydimethyl siloxanes, which are available either in granulate form or are modified by additives directly prior to extrusion, have proven to be especially suitable. Polyoxysilanes of the type which constitute modified polyesters and are provided for use in accordance with the invention are known from US-A-5,123,392. The known chemically modified polymers are used for producing hydrophilic staple fibers, but are also said to be usable for fabrics made of filament yarns. The hydrophilic properties make possible the rapid absorption of moisture and are therefore mainly used in a mixture with cotton or wool. In their present form yarns having the hydrophilic properties mentioned, however, are unsuited for paper-making machines or fishing lines.

Silicon-modified polyesters which are suitable for producing fibers are also known from EP-A-0 269 023. In the course of producing the polymer, the silicon units are formed in a polyester matrix. During extrusion in air and drawing, these silicon units migrate into the outer envelope of the filament.

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The surface obtained acts like a hydrophobic film. The chemical composition is not homogeneous in cross section.

It is the object of the invention to make available a monofilament which is simultaneously soil-repellent and abrasion-resistant and homogeneous in cross section, meets the great demands made on an industrial fabric in respect to its chemical, soil-repelling, mechanical and abrasion-resistant properties and finds its use in the paper industry in particular.

The object of the invention is attained by means of the invention in that 0.15 to 5.0 weight-% of polydialkyl siloxane are inserted into the chain of the polymer by co-condensation, i.e. in the course of the poly-condensation.

The tenacity of the monofilaments is ^{preferably} greater than 36 cN/tex with an ^{preferably} elongation of < 44% referred to the unstretched filament, wherein a particularly good soil repellency and little fibrillation occur as essential characteristic in a surprising manner. The soil repellency has been determined to be a soiling index of < 10 following five soiling/cleansing cycles, compared with > 14 for a polyester filament without polydimethyl siloxane.

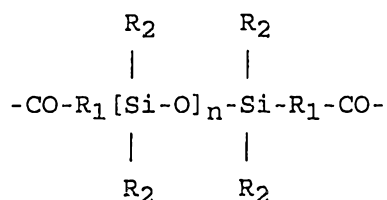
It is practical to use a polyester consisting to at least 50 weight-% of polyethylene terephthalate and a polydialkyl siloxane, wherein the polyethylene terephthalate/polydialkyl siloxane in the finished monofilament contains 0.05 to 2 weight-%, preferably 0.1 to 1.0 weight-% of silicon referred to the polymer, and the homogeneous mixture is extruded into a monofilament. It should be stressed as a particular advantage that the produced monofilament has a homogeneous composition over the entire cross section.



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A polyethylene terephthalate to be considered is, preferably, one which has an intrinsic viscosity of > 0.60 dl/g.

Difunctional polydiorgano siloxanes of the general formula:



are preferably considered as copolymers, wherein R1, R2 represent equal or different radicals with 2 to 20 carbon atoms. Polydimethyl siloxanes (PDMS) with R2 = CH₃ have proven themselves to be particularly suitable.

The silicon content in the monofilament is 0.05 to 2.0 weight-%, in particular 0.1 to 2 weight-%, preferably 0.1 to 1.0 weight-%.

The use of the soil-repellent monofilaments in accordance with the invention, having a diameter of 0.1 to 1.0 mm, in particular 0.2 to 0.6 mm, is particularly suited to the production of screen fabrics for paper-making machines.

The invention will be described in detail by means of an example.

Exemplary Embodiment

The production of the polymer takes place in a known manner by transesterification of dimethyl terephthalate and ethylene glycol and the addition of 3.9 weight-% of polydimethyl siloxane



(PDMS) and manganese acetate in stirred autoclaves at 160 to 230°C, wherein the generated methanol is quantitatively distilled off. The subsequent poly-condensation is performed over 200 min in a temperature range between 245 to 285°C and with the use of an antimony catalyst, wherein condensation is performed in a first step in a progressive vacuum (1000 -> bar) over 40 minutes and in a second step in a high vacuum (< 1 mbar).

The polyethylene terephthalate/polydimethyl siloxane (PET/PDMS) is melted at a temperature of 290°C. Extrusion takes place by means of a spinneret with 13 capillaries and a capillary diameter of 1.8 mm at a polymer flow rate of 486 g/min. The extruded monofilament are cooled in a water bath at 70°C. After cooling the monofilaments are spin-prepared and stretched to a residual elongation of approximately < 44%, relaxed and wound at a speed of 100 m/min. The resulting fiber has a soiling coefficient of less than 10 (< 10) after five treatments, with a 500% stretching has a tenacity (Ft) of 36.1 cN/tex at an elongation (Dt) of 38.8% referred to the unstretched monofilament.

However, in place of the PET/PDMS as the polymer it is also possible to start with pure, commercially available polyester (polyethylene-terephthalate) and to add polydimethyl siloxane (PDMS) upstream of the extruder by means of a metering device.

The following method was developed for the determination of the soil-repellent effect of monofilaments for screen fabrics for paper-making machines:

Principle of the Testing Method
(Soiling Test)

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Rust-resistant small steel plates are exactly wound with three layers of the monofil. After rinsing out the spinning preparation and colorimetric measurements, the top of the sample is soiled eight times in accordance with a fixed method by means of "transfer printing" with wet newsprint in an ironing press at 130°C. Following colorimetric measuring, cleansing, drying and another colorimetric measurement are performed in a specific manner. This soiling/cleansing process is performed five times in a row for the simultaneous determination of the "soil release" properties. At each stage the DL* (D65)-values are indicated as quantitative results, with the cartels before they were first soiled respectively used as references. DL* (D65) corresponds to the brightness difference between the sample and the reference in daylight. To exclude the effects of color differences in the samples, the brightness decrease in percent is indicated, which is defined as

$$AI = - [DL^*] / L^*_0 \times 100$$

and is called the soiling index.

Principle of the Test Method (Fibrillation Test)

To determine the fibrillation of monofilaments for screen fabrics for paper-making machines, a bundle of monofilaments of the same kind is beaten against a standardized solid body for 10 minutes at a frequency of 2000 to 3200 rpm.

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The results of the fibrillation test are combined below, wherein the grading has the following meaning:

Grading of the Fibrillation

- 0.0 - 0.5 : no or very weak fibrillation
- 0.5 - 1.5 : weak fibrillation
- 1.5 - 3.5 : moderate to strong fibrillation
- 3.5 - 4.5 : very strong fibrillation
- > 4.5 : very great damage

The fibrillation test results are collected in the following table.

Table

Example	Grade at Beat Frequency [rpm]			
	2000	2500	3000	3200
1	0.0	0.1	0.5	2.5
2	0.5	1.0	0.9	4.5
3	0.4	1.1	1.5	5.0
4	0.2	0.8	1.0	5.0

Example 1 shows the result with the monofilament in accordance with the invention, wherein no fibrillation at all occurs at 2000 beats per minute, while at 3200 beats a fibrillation of less than



half that of the prior art occurs. Example 2 shows the result with a polyester made "soil-repellent" by means of a fluoropolymer, and the Examples 3 and 4 standard polyesters. The invention will be described in more detail by means of the drawings.

Furthermore, the monofilaments produced in accordance with the invention from PET/PDMS are less rigid than known monofilaments.

Shown are in:

Fig. 1, a force/elongation diagram

Fig. 2, soiling test results

Fig. 3, results of the fibrillation test.

From the force/elongation diagrams in Fig. 1 it can be seen that in a surprising manner the tenacity of the monofilament in accordance with the invention has not been reduced by copolymerization.

The values of the soiling indices represented in Fig. 2 show the effects of successive "soiling/cleansing" cycles. Mean values of two samples after soiling and cleansing are respectively represented. The curve 1 represents the monofilament fiber in accordance with the invention. Curve 2 represents a comparison fiber of polyester in accordance with the prior art, i.e. without the polydimethyl siloxane component. Following (five) 5 soiling/cleansing cycles, indices below 10 are attained with the monofilament in accordance with the invention.

Fig. 3 shows in the form of photographs the results of a fibrillation test. Fig. 3a shows a polyester with a fluoropolymer; Fig. 3b the monofilament in accordance with the invention. It can be seen from this that the monofilament in



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accordance with the invention shows considerably less fibrillation. Under the same conditions, a sample made of polyethylene terephthalate in accordance with the prior art even shows a large weight reduction.

In the soiling test the monofilament fiber in accordance with the invention results in an improvement by at least a factor of 1.5 in respect to the prior art. The soil repellency of the fibers modified with siloxanes shows a clear improvement in comparison with the standard and approximately corresponds to that of the fluorine-containing special fibers which are questionable for reasons of environmental load. The abrasion resistance of the fibers is clearly increased in respect to the prior art. The monofilaments in accordance with the invention are preferably suited for use in paper-making machine fabrics.

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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. A process for producing soil-repellent and abrasion-resistant monofilaments for screen fabrics, having a diameter of between 0.1 to 1.0 mm, from linear silicon-modified polyethylene terephthalate, characterized in that 0.15 to 5 weight-% of polydialkyl siloxane are inserted into the chain of the polymer by condensation and further characterized in that the product monofilament has an Si content of 0.05 to 2.0 weight-% referred to the weight of the monofilament.

2. A process in accordance with claim 1, characterized in that the silicon-modified polymer is extruded into an aqueous solution.

3. A soil-repellent and abrasion-resistant monofilament produced by the process of claim 1 or 2, wherein the monofilament has a diameter of between 0.1 to 1.0 mm, consisting of a silicon-modified polyethylene terephthalate, characterized in that the monofilament has a tenacity of at least 36 cN/tex, an elongation of <44% referred to the unstretched filament, and simultaneously a soiling index of <10 after five soiling cycles.

4. A monofilament in accordance with claim 3, characterized by a fibrillation grade <0.1 at 2000 rpm.

5. The use of the monofilament of claim 3 or 4 or the product monofilament of claim 1 or 2 in the production of screen fabrics for paper-making machines, fishing lines or fishing nets.

DATED this 3rd day of April 1997

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LCG:JGC:KR DOC 11 AU6935894.WPC



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6. Use of the soil-repellent and abrasion-resistant monofilament with a diameter of between 0.1 to 1.0 mm, consisting of a silicon-modified polyethylene terephthalate, with a tenacity of at least 36 cN/tex, an elongation of < 44% referred to the unstretched filament, and simultaneously a soiling index of < 10 after five soiling cycles, for producing screen fabrics for paper-making machines, as well as fishing lines and fishing nets.



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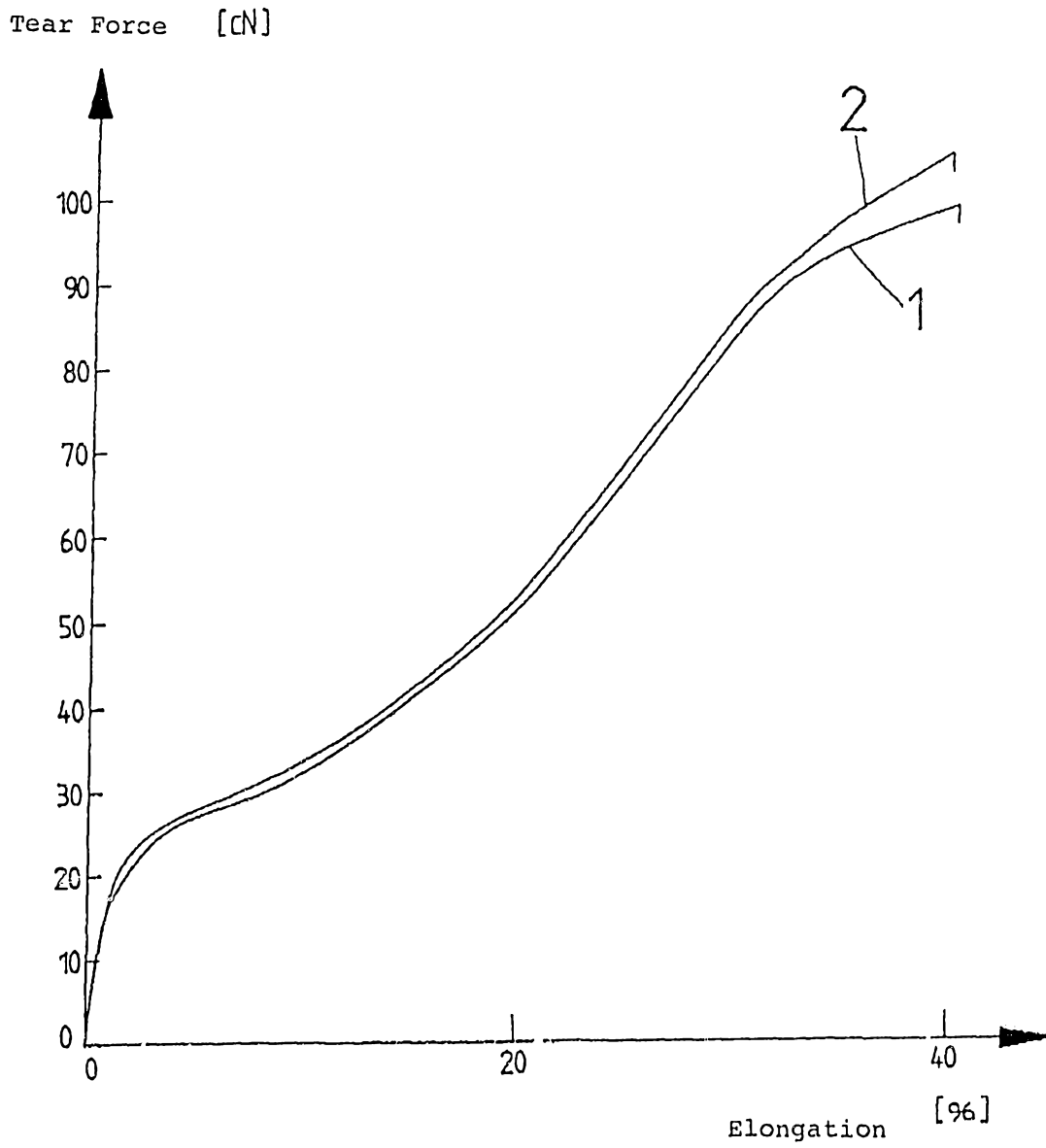


Fig.1

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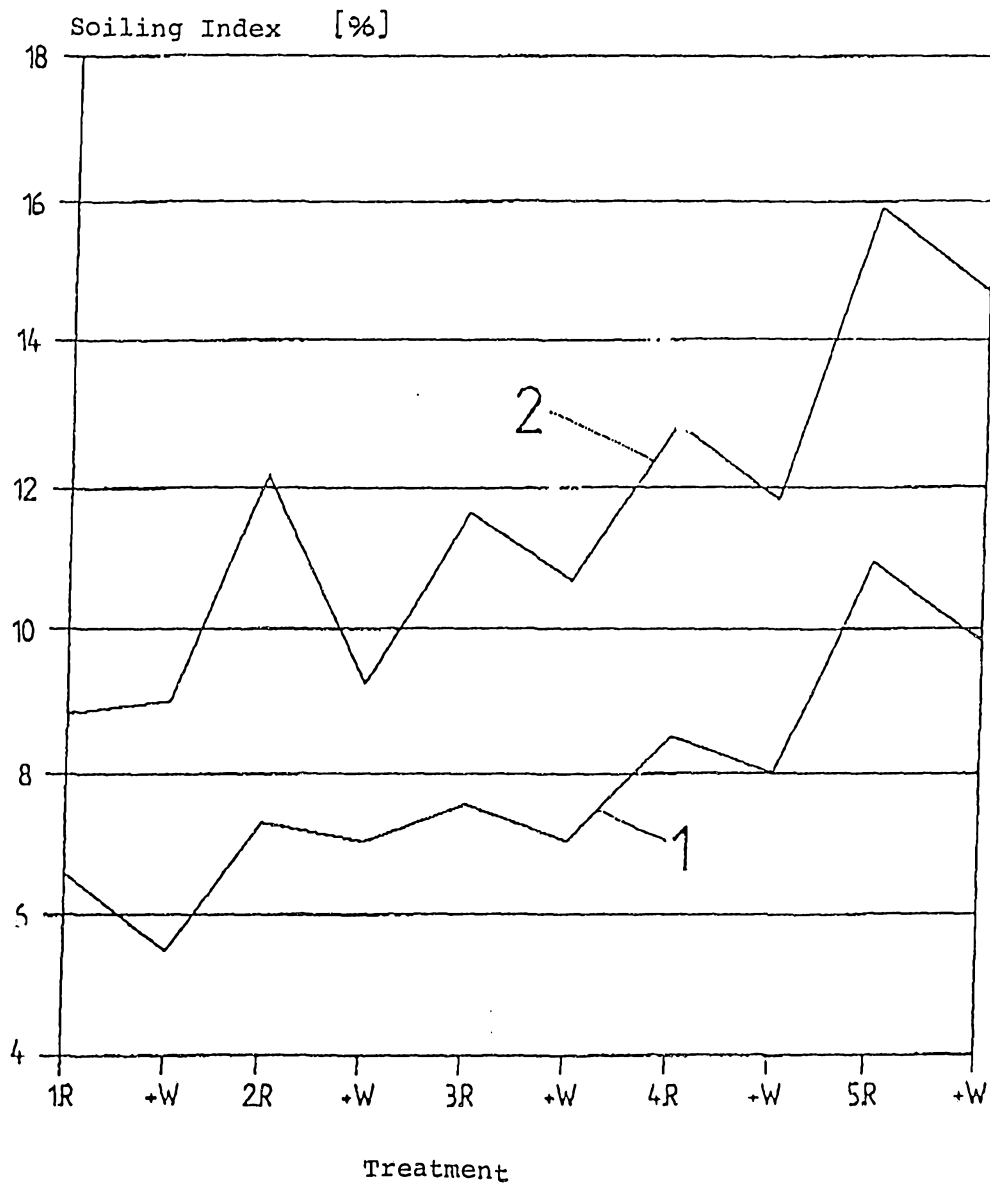


Fig.2

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Fig. 3a



Fig. 3b

INTERNATIONAL SEARCH REPORT

International Application No
PCT/IB 94/00183

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 D01F6/84

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)
IPC 6 D01F C08G

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

Electronic data base consulted during the international search (name of data base and, where practical, 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	EP,A,0 269 023 (UNION CARBIDE CORPORATION) 1 June 1988 cited in the application see the whole document ---	1-6
A	DATABASE WPI Section Ch, Week 7552, Derwent Publications Ltd., London, GB; Class A, AN 75-85481W & JP,A,50 090 715 (TORAY INDS KK) 21 July 1975 see abstract ---	1-6
P,A	WO,A,93 18086 (RHONE-POULENC FIBRES) 16 September 1993 see the whole document ---	1-6
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

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Date of the actual completion of the international search

21 November 1994

Date of mailing of the international search report

28. 11. 94

Name and mailing address of the ISA

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Authorized officer

Tarrida Torrell, J

INTERNATIONAL SEARCH REPORT

Inter national Application No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	PATENT ABSTRACTS OF JAPAN vol. 9, no. 218 (C-301) 5 September 1985 & JP,A,60 081 313 (TOURE MONOFUIRAMENTO KK) 9 May 1985 see abstract ---	1-6
A	PATENT ABSTRACTS OF JAPAN vol. 9, no. 218 (C-301) 5 September 1985 & JP,A,60 081 312 (TOURE MONOFUIRAMENTO KK) 9 May 1985 see abstract ---	1-6
A	PATENT ABSTRACTS OF JAPAN vol. 10, no. 49 (C-330) 26 February 1986 & JP,A,60 194 118 (UNITIKA KK) 2 October 1985 see abstract -----	1-6

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/IB 94/00183

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0269023	01-06-88	US-A- 4758637 CA-A- 1142196 JP-A- 63165432	19-07-88 26-03-91 08-07-88
WO-A-9318086	16-09-93	FR-A- 2688509	17-09-93

A. KLASSIFIZIERUNG DES ANMELDUNGSGEGENSTANDES
IPK 6 D01F6/84

Nach der Internationalen Patentklassifikation (IPK) oder nach der nationalen Klassifikation und der IPK

B. RECHERCHIERTE GEBIETE

Recherchiertes Mindestprüfstoff (Klassifikationssystem und Klassifikationssymbole)
IPK 6 D01F C08G

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C. ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
A	EP,A,0 269 023 (UNION CARBIDE CORPORATION) 1. Juni 1988 in der Anmeldung erwähnt siehe das ganze Dokument ---	1-6
A	DATABASE WPI Section Ch, Week 7552, Derwent Publications Ltd., London, GB; Class A, AN 75-85481W & JP,A,50 090 715 (TORAY INDS KK) 21. Juli 1975 siehe Zusammenfassung ---	1-6
P,A	WO,A,93 18086 (RHONE-POULENC FIBRES) 16. September 1993 siehe das ganze Dokument ---	1-6
-/--		

Weitere Veröffentlichungen sind der Fortsetzung von Feld C zu entnehmen

Siehe Anhang Patentfamilie

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Z Veröffentlichung, die Mitglied derselben Patentfamilie ist

Datum des Abschlusses der internationalen Recherche

21. November 1994

Abschließdatum des internationalen Recherchenberichts

28. 11. 94

Name und Postanschrift der Internationalen Recherchenbehörde
Europäisches Patentamt, P.B. 5818 Patentlaan 2
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Bevollmächtigter Bediensteter

Tarrida Torrell, J

C.(Fortsetzung) ALS WESENTLICH ANGESEHENE UNTERLAGEN

Kategorie*	Bezeichnung der Veröffentlichung, soweit erforderlich unter Angabe der in Betracht kommenden Teile	Betr. Anspruch Nr.
A	PATENT ABSTRACTS OF JAPAN vol. 9, no. 218 (C-301) 5. September 1985 & JP,A,60 081 313 (TOURE MONOFUIRAMENTO KK) 9. Mai 1985 siehe Zusammenfassung ---	1-6
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PCT/IB 94/00183

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