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54 **Manufacturing process for internal siliconization of synthetic fibres and internally siliconized synthetic fibres manufactured according to this proces.**

57 Method for internal siliconization of synthetic fibres, with the proviso that polymeric siloxanes are added to the high molecular weight polymer: injected into the polymere melt and mixed into the polymer, wich polymer is cut into pellets that further are spin into internal siliconized fibres by which spinning process cristalline faults are introduced which migration assures a permanent siliconization.

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MANUFACTURING PROCESS FOR INTERNAL SILICONIZATION OF SYNTHETIC FIBRES AND INTERNALLY SILICONIZED SYNTHETIC FIBRES MANUFACTURED ACCORDING TO THIS PROCES.

The use of synthetic fibres as a replacement for natural raw materials has many applications, and is not just for woven products. Depending on the nature of the end product and its intended use, these synthetic fibres need to meet specific requirements to guarantee good quality and ease of maintenance. Often the synthetic fibres need to undergo a particular process. A standard process is the siliconization of synthetic fibres, which gives them a number of beneficial properties which are necessary if, for instance, they are to be used as fillers : their lubricity and elasticity is increased and they appear to be more resistant to washing with warm water and cleaning with chemicals. If, for instance, the synthetic fibres are to be used for carpet fibres then the anti-soiling properties (soil resistance) are greatly increased through siliconization.

A number of methods are used to siliconize synthetic fibres and these can be separated into two major groups. On the one hand we have a number of methods which consist of an external application of silicone products (emulsions and/or silicone oils) at the end of the process. One disadvantage on these methods is that the various subsequent processes (strengthening, texturing, etc.) are influenced negatively. It is also difficult to apply the silicone films evenly, especially when using low doses. Another disadvantage is the fact that the contact bonding between the silicone and the surface of the fibre is inadequate, especially when polyolefin resins are used, guaranteeing only limited resistance to washing and chemical cleansing.

The second group of methods for applying silicone to synthetic fibres is the incorporation of silicone products within the synthetic fibres, especially as a separation product (for instance for use with injection molding) or as a lubricant. The disadvantage here lies in the processing problems which are encountered in the manufacture of "non-woven" and fibres.

The purpose of the invention is to create a manufacturing process for continuous siliconization of synthetic fibre surfaces, thereby allowing the manufacture of synthetic fibres which do not display the aforementioned drawbacks.

An object of the invention is manufacturing process for internal siliconization of synthetic fibres, characterized by the addition of polymer siloxane products to the high polymer raw materials which are to be used for the manufacture of synthetic fibres. The polymer siloxane is injected evenly into powder raw materials. This is then heated to a high temperature whilst being rotated vigorously, followed by cooling to a temperature which is lower than the melt viscosity of the polymer raw material.

By using this method a material is obtained which guarantees an even distribution of the incorporated polymer siloxane, this material being another object of the invention.

Another characteristic of this method is that further processing of the raw material can be carried out, this may take the form of either a compound and/or masterbatch, depending on the concentration of the polymer siloxanes which have been introduced.

The method, according to the invention, is characterized in a subsequent step when, by means of mechanical action on the spinning thread, standard crystalline faults are created which lead to larger surfaces and exit points for the silicone distribution on the separate fibre surfaces.

"Standard crystal defaults" actually means mechanical deformation of the fibre via the so called texturation process, causing a pressure increase on the surface of the fibre resulting in a squeezing on-set of the silicone out of the polymer matrix.

The method, according to the invention, is also characterized by the fact that the polymer siloxanes are loaded in such a way that the silicone level on the surface of the fibres is kept as low as possible during the spinning process (as close to zero as possible) and that the siliconized surface is only achieved after all steps, necessary for the manufacture of the synthetic fibres, have been completed, though continuous migration and distribution of the polymer siloxane products, from the transverse section to the surface of the siliconized synthetic fibres.

The already existing installations and methods for the manufacture of synthetic fibres are also used to implement the method.

The method, according to the invention, is characterized by the fact that the raw materials (high molecular polymers) can be used without exclusion of other materials :

- polyolefin resins, more especially polypropylene and polyethylene (HDPE, LLDPE, LDPE)
- and/or the above mentioned polymers with addition of various additives - butadiene styrol derivatives are highly suitable.

The method for internal siliconization of synthetic fibres is also characterized by the following method : polymer dimethyl - and methyl phenyl siloxane with a viscosity reach of 1000 to 20.000 square mm.s-1 (25 degr. C), and mixtures of these products. The polymer siloxanes are added to the high polymer raw

materials in concentrations of between 0.5 and 20 percentage weights based on the total weight of those high polymer synthetic fibre materials.

It is essential for the operation method, according to the invention, that the polymer siloxanes used are measured in such a way that the migration towards the surface of the synthetic fibre and the distribution of the siloxanes is slowed down, thereby preventing disturbances during the spinning process of the synthetic fibre, and that it is followed by stabilization. This will guarantee a permanent silicone film even after washing and/or chemical cleaning thanks to a continuous migration of the polymer siloxanes to the surface which provides a good grip for the synthetic fibres.

The method, according to the invention, is further characterized by the ability to spin the material into synthetic fibres with varying cross sections - after the high polymer raw materials suited to the manufacture of synthetic fibres and the correct doses of polymer siloxanes are added and mixed. The cross section can be oval, round, triangular, trilobal and/or cellular with various titres, for instance 1.7 to 200 dtex - for instance 4 to 150 mm, with various pigmentations, for instance black, white or coloured, and with various textures, for instance, two-dimensional and/or three-dimensional folds, as well as various curves, for instance 2 to 12 curves per cm.

Another object of the invention is the internally siliconized synthetic fibres produced by this method which are characterized by an even and permanent migration of the polymer siloxane products from the transverse cut to the surface even after the silicone layer has been removed by washing and/or chemical cleaning.

During a preferential embodiment, the synthetic fibres manufactures using the above mentioned high polymer raw materials, according to the method, according to the invention, have a trilobal transverse cut of 6.7 dtex, a staple length of 60 mm, a white colour and three-dimensional folds with 6 to 7 curves per cm.

Another possible method, according to the invention, deviates from the aforementioned and is characterized by the fact that, using siloxane products with a high degree of viscosity, and using suitable measuring equipment (for instance gear pump), they can be added directly, in the correct doses, to the high molecular materials during the spinning process in the extrusion equipment.

The following description gives a few examples of the method, according to the invention. Each example uses a different high polymer raw material and/or added polymer siloxane product. The invention is not limited to these methods nor to the use of the suggested raw materials and siloxane products, their dosage, their specific characteristics or the operating temperatures and implementation means.

Example one

The raw material used is polypropylene with an MFI of 14 [230° C, 21, 8N] and a density of 0.91 g.cm.cube. A 2 percentage weight of polymer dimethyl siloxane with a viscosity of 1000 mm square.s-1 (measured at 25 degr. C) and a density of 0.97g.cm.cube (measured at 25 degr. C) is added evenly, and in the correct quantities, to polypropylene powder at a temperature of 120 degr. C, using an injection mechanism and whilst in constant spinning. After dosing, the product is cooled to room temperature, whilst still in constant spinning. After a rest period the mixture is granulated using the known method.

The granular substance which contains the polymer siloxane and which has been prepared according to the invention, is diluted on the synthetic fibre apparatus and extruded in 1:1 proportions with polypropylene granules which contain 5% butadine styrol derivative and 0.5% titanium dioxide.

The extrusion and the orientation are done following known methods.

In order to achieve the required standard crystalline faults which will lead to a migration and distribution of the silicone product, the spinning-thread is conducted at high speeds of 1400-1500 m/s against lamellae in a hot air jet chamber. This will give them a three-dimensional fold.

The spinnerettes are equipped with trilobal openings making a titre of 6.7 dtex possible. The staple length is set at 60 mm.

Extrusion conditions.

Mixing of components.

The polymer powder and additives are mixed in a fluidised bed mixer 15" on low speed and 30" on high speed. The polymeric siloxanes are sprayed in the mixer (while spinning) and mixed on high speed into the polymer powder. The mixing takes ± 10 min until a temp. of 120° C max is reached (for PP). The polymer is cooled to room temperature. Low speed = 500 m/min
High speed = 1500 m/min

Pelletising of mixture

The mixture is pelletised on a W&P ZSK 53 Extrusion temp. (° C) :

5		Z1	Z2	Z3	Z4	Z5	Melt
		190	190	190	190	200	210

10 Rpm screw : 144
Output : 100 Kg/h

Extrusion settings for spinning of silicon modified PP

15 Extruder : single screw extruder 40 mm dia. 28 l/d
Temperatures : (° C)

		Z1	Z2	Z3	Z4	Z5
		200	210	220	230	240
20		SPINBEAM		PUMP		SPINNERETTE
		250		250		250

25 Extruder rpm : 35
Regulating pressure : 80 Bar
Pump : 2X52 holes
Take up speed : 480 m/min
Strain ratio : 3

30 Drawing speed : 1432 m/min
Drawing temperature : 120° C
Texturation temperature 135° C
Cut speed : 1230 m/min

Clarification.

The temperature meant is maximum 120° C to avoid softening and sticking of the PP, and the polysiloxane is actually spread on the surface of the powder base-material.

40 However the melt-mixing is present when the polysiloxane is injected directly into the extruder using a volumetric dosing device.

This method differs from the pre-mixing practice because the mixing and homogenisation of the two components (PO & Polysiloxane) takes place during the extrusion process of the polymer into fibres.

Example two

45 In contrast with the first example, a high polymer (HDPE) is used with an MFI of 11/190 degr. C and a density of 0.95 g.cm cube.

The extrusion equipment, and especially the temperature controls, are set for HDPE. This also applies to the temperature in the mixer during the addition of the silicone product to the powder raw material. The
50 other steps used are the same as in the first example.

Spinning of fibres in this example is done on the same principle as in example 1 but spinning temperatures are adjustd to the polymer type.

Example three

55 In contrast to example two, a polyethylene (LLDPE) is used with an MFI of 18/190 degr. C and a density of 0.93 g.cm cube. Once again the mixing and extrusion temperatures are set correctly for the raw material used. The other steps in the process, according to the invention, are the same as in example one, and

example two.

Spinning of fibres is done as previously described but temperatures are adjusted to the polymertype.

Example four

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As in example one, the raw material used is PP with an MFI of 14 (230 °C, 21, 8N) and a density of 0.91 g/cm³ at 23 °C. A polymeric dimethylsiloxane with a viscosity of 20 000 mm²/s (25 °C) is added evenly (4% siloxane weight conc. in the matrix), and in the correct quantities, to the melt of a high polymeric PP at temperatures of 200 °C-230 °C. The polymeric siloxanes are injected in a twin crew extruder with a volumetric pump. The PP and silicon is mixed in the extruder and the material is granulated.

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The other steps are as in example 1.

Spin temperatures are adjusted to the polymer type.

Example five

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As in example four, the raw material is PP and a polymeric methyl phenyl siloxane is used (viscosity 1000 mm²/s). In contrast with example four, the polymeric siloxane is dosed with a volumetric pump directly into the spinning extruder and in such way that the silicon level is 2% in respect to the total weight of the fibre raw material.

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An advantage of the operating method, according to the invention, lies in the fact that the various sequential procedures which the synthetic fibres are submitted to during their manufacture and/or processing, are not influenced negatively by siliconization, as this only becomes operational later on. (the positive synthetic fibre characteristics remain stable during processing and manufacture).

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Another advantage of the operating method, according to the invention, and the siliconized synthetic fibres, according to the invention, is that, after manufacture and processing of the synthetic fibres, an even distribution of the silicone film is achieved, and that this remains permanent, through continuous migration of the siloxane products from the centre to the surface of the synthetic fibres, replacing any silicone layer lost through washing of chemical cleaning. This gives a high resistance to washing and chemical cleaning.

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The above mentioned and aspired positive characteristics of the siliconized synthetic fibres are obviously achieved in this example (high lubricity, high degree of elasticity, resistance to washing and chemical cleaning, anti-soiling properties) with the added advantage that these characteristics are maintained for a longer period due to the continuous migration and distribution of the siloxanes.

This migration and distribution then occurs evenly and continuously maintaining the known properties of siliconized synthetic fibres, even after washing and cleaning with chemicals.

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Claims

1. Method for internal siliconization of synthetic fibres during manufacture, using existing methods and installations, with the proviso that the polymer siloxanes added to the high polymer synthetic fibre raw materials, are suitable for filament formation. These materials are mixed and, after a rest period, standard crystalline faults are created during spinning using mechanical action.
2. Method for internal siliconization of synthetic fibres, according to claim 1, with the proviso that the polymer siloxanes are injected evenly into the basic powder raw material, which whilst being rotated vigorously, are heated evenly to a high temperature and which are subsequently, still under constant spinning, allowed to cool to a temperature level below the melt viscosity of the polymer raw material.
3. Method for internal siliconization of synthetic fibres according to claims 1 and 2, with the proviso that for the production of the polymeric basic raw material used for internal siliconization, an extrusion process will be applied producing pellets, in which extruder polymeric siloxanes being accurately dosed have been directly injected, even in the polymere melt distributed said polymere melt being mixed with the siloxanes through a perforated plate of the extruder are pressed before the delivered threads are cut in pellets, which pellets are used in the spinning of internal siliconized fibres.
4. Method for internal siliconization of synthetic fibres, according to one of the previous claims, with the proviso that after mixing the polymer siloxanes and the high polymer synthetic fibre raw materials one allows the materials to rest.

5. Method for internal siliconization of synthetic fibres, according to one or more claims 1 to 4, with the proviso that the pre-mixture is further processed, after a rest period, as a compound and/or master-batch into synthetic fibres with a fibre transverse cut which can be round, oval, triangular or trilobal, with or without cells, with two or three dimensional folds and 2 to 10 curves per cm and a titre reach of 1.7 to 200 dtex with staple lengths of between 4 and 150 mm or continues filaments.
6. Method for internal siliconization of synthetic fibres, according to one or more of claims 1 to 5, with the proviso that standard crystalline faults are created, during the spinning process. This is done by conducting the spinning thread on to lamelle at high speeds, for instance 1400 to 3000 m/s in a hot air jet chamber, thereby producing a three dimensional fold.
7. Method for internal siliconization of synthetic fibres, according to claim 6, with the proviso that the jet chamber used to create the standard crystalline faults operates using pressurized moisture vapour.
8. Method for internal siliconization of synthetic fibres, according to one or more claims 1 to 7, with the proviso that the high polymer synthetic fibre raw materials which are best suited for fibre formation are used, namely the following :
 - polypropylene
 - high density polyethylene
 - low polyethylene
 - linear low density polyethylene
 - the above mentioned materials with various additives, preferably butadiene styrol derivatives and titanium dioxide
9. Method for internal siliconization of synthetic fibres, according to one or more of the aforementioned claims, with the proviso that preference is given to the following siliconization products :
 - polymer dimethyl siloxane
 - polymer methyl phenyl siloxane with a viscosity level between 1000 and 20,000 mm square/s
 - mixtures of these products
10. Method for the internal siliconization of synthetic fibres according to one or more of the aforementioned claims, with the proviso that the polymer siloxanes are added in concentrations of between 0.5 and 20 percentage weights, these being calculated on the basis of the total weight of the high polymer synthetic fibres raw materials.
11. Method for internal siliconization of synthetic fibres, according to one or more of the afore mentioned claims, with the proviso that siliconization products with a higher viscosity level are fed into the extrusion equipment during the spinning process and added to the high molecular raw materials in the correct dose, using suitable measuring equipment, for instance gear pumps.
12. Raw materials obtained by following the method, according to one or more claims 1, 2 and 3, with the proviso that an even distribution is set up for the incorporation of the polymer siloxane into the synthetic fibre raw materials.
13. Internal siliconized synthetic fibres, manufactured according to the method, according to one or more of the aforementioned claims, with the proviso that a migration to the surface and distribution of the polymer siloxanes occurs, and that this is slowed down to such an extent that the siloconized surface is only achieved after all the steps necessary for the manufacture of the synthetic fibres have been completed.



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EUROPEAN SEARCH REPORT

Application Number

EP 91 20 0262

DOCUMENTS CONSIDERED TO BE RELEVANT					
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)		
X	US-A-4 640 962 (R.L. OSTROZYNSKI et al.) * Claims; column 2, lines 47-54; column 7, lines 60-66; column 9, line 4 * - - -	1-4,8-13	D 01 F 1/10		
X	US-A-4 857 251 (R.S. NOHR et al.) * Claims; column 25, lines 30-51 * - - -	1-4,8-13			
A	FR-A-1 462 790 (E.I. DU PONT DE NEMOURS) - - -				
A	WPI/DERWENT, AN = 78-24633A [13], Derwent Publications Ltd, London, GB; & SU-A-493 118 (GRIGOREVA) 13-09-1977 - - - - -				
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)		
			D 01 F		
The present search report has been drawn up for all claims					
Place of search The Hague		Date of completion of search 21 May 91	Examiner VAN GOETHEM G.A.J.M.		
<table><tr><td>CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention</td><td>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</td></tr></table>				CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure 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
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