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(54) Method of treating wool.

(57) A continuous process for the production of shrink-resistant wool comprises:

i) subjecting the wool to an oxidative chlorinating pretreatment;

ii) subsequently treating the wool with an aqueous solution of a polymer containing quaternized amino groups, preferably one derived from an amino-acrylic monomer; and

iii) drying the wool to a chosen moisture level in the range 12% to 18% by weight on the weight of the dry wool, using at least 20% less energy than is required to dry to the same moisture level wool which has been rendered shrink-resistant by conventional continuous processes.

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METHOD OF TREATING WOOL

This invention relates to improvements in the treatment of wool to impart shrink resistance thereto and is more especially concerned with the treatment of materials consisting of or containing wool which are available in continuous lengths, or which can readily be joined together into a continuous form to enable the same to be subjected to substantially uniform treatment.

Woollen materials which are available in continuous lengths include yarns, tops and woven and knitted fabrics consisting of or containing wool. Such lengths may also be formed by sewing together knitted garments such as socks: these are then separated after completion of the treatment.

The invention can also be applied to loose wool. For this purpose, means are required for transporting loose wool in a continuous fashion. Such means are known in the field and include conveyer belts and rotating rake systems.

Two-step shrink-proofing processes in which wool is treated first with a chlorinating agent and subsequently with a pre-formed synthetic polymer are well known. A wide variety of polymers can be used in aqueous solution or dispersion, including polyamide-epichlorohydrin resins and polyacrylates. A review of work in this field by J. Lewis appears in Wool Science Review, May 1978, pages 23-42.

But despite achieving commercial success, known processes could be improved in various ways:-

i) Drying. Wool treated by conventional processes needs to be rather thoroughly dried at elevated temperatures. This is believed necessary in order to cure the polymer, but is inconvenient in those plants where the drying capacity is the limiting factor. In such plants, which are common, capacity could be increased by raising the drying temperature but only at the cost of increased yellowing and degradation of the wool. A method with reduced drying requirements would be welcomed.

ii) Application of polymer. If the process is being carried out in a backwasher, it is practically necessary to apply the polymer by means of a suction drum, in order to ensure even distribution of the polymer on the fibres. But a suction drum is an expensive item of equipment and a method which could achieve even deposition of polymer without the need for suction would be welcomed.

iii) Quantities. Chlorination tends to yellow and degrade the fibres and polymer deposition alters the handle of the wool. Both chemicals are sufficiently expensive to have a significant effect on the cost of the product. A method which used lower amounts of chemicals to achieve an equivalent degree of shrinkproofing would be welcomed.

Amino-acrylic polymers are well known and are described, for example, in British Patent Nos.1318781 and 1393273. The use of such polymers for the shrinkproofing of wool has been suggested, for example in U.S. Patent Nos. 3678098 and 3842054, although not it is believed in a two-stage continuous process.

The present invention results from our discovery that certain polymers containing quaternized amino groups confer surprisingly good shrink-resistance and other advantages when used in a two-stage treatment of the conventional kind. Preferred forms of the invention overcome each of the three disadvantages noted above.

According to the present invention there is provided a continuous process for the production of shrink-resistant wool comprising the steps of:-

i) subjecting the wool to an oxidative chlorinating pretreatment;

ii) subsequently treating the wool with an aqueous solution of polymer so as to cause the polymer to exhaust on to the wool fibres; and

iii) drying the wool to a chosen moisture level; characterized in that a polymer containing quaternized amino groups is used in step ii), and in that the wool is dried in step iii) to from 12% to 18% moisture, by weight on the weight of the dry wool, using at least 20% less energy than is required to dry to the same moisture level wool which has been rendered shrink-resistant by conventional continuous processes.

A major advantage of the present process is that it requires significantly less energy than other conventional shrink-proofing processes, in order to achieve a comparable degree of drying of the wool. Indeed, it is estimated that there is at least a 20% saving in energy over such processes.

There is also evidence to suggest that wool treated by the present process requires less heat treatment than wool treated by known processes. The wool is preferably dried to a moisture level of 12-15% (by weight), whereas conventional processes involve drying to a lower moisture content. A low moisture level may lead to handling difficulties during subsequent processing of the wool.

Advantage can be taken of these surprising findings in one or both of two ways. The temperature of the drier can be reduced, thus saving power. The drying time can be reduced,thus increasing throughput. It is believed that plants operating the process of the present invention will have a greater capacity and be generally more efficient because of these less stringent drying requirements. The reduced energy requirement should also result in a significant saving in overall running costs.

While Applicants do not wish to be bound by theory, they presently believe that the reason for this ease of drying may be this. Conventional driers consist of perforated heated drums round which the wool is fed and into which air is sucked. The wet wool forms a soggy blanket through which air initially passes only with some difficulty. Wool fibres treated with hydrophilic resin by conventional shrink-resist processes tend to stick together and retain this blanket form as they dry. But wool fibres treated with possibly more hydrophobic resins by the process of this invention readily spring apart, due to their natural resilience, thus bulking the wool, facilitating the passage of air through the mass and speeding the drying process.

As previously stated, the chlorinating pretreatment is a conventional procedure and a number of suitable treatments are already well known. Chlorinating agents such as hypochlorite or sodium dichloroisocyanurate may be employed, (optionally together with potassium permanganate or permonosulphate) at levels of 0.25-2.0% active chlorine,by weight on the weight of the dry wool (O.W.W.), most preferably 0.5-1.2%. It should be noted that the optimum level of chlorine employed is dependent upon the level of polymer used in the next stage of the process. The pretreatment will,by itself, produce a small degree of shrink-resistance in the wool.

The wool is subjected to antichlorination treatment with sulphite and rinsed and is then ready for the polymer application stage.

The polymer used contains quaternized amino groups in sufficient proportion to cause the polymer to exhaust from aqueous solution on to the (anionic) wool fibres. When the polymer is derived from a monomer or polymer containing tertiary amino groups, some or preferably all of these are quaternized, sufficient to confer the desired cationic character on the polymer. The polymersmay be homopolymers or copolymers. Two classes of such polymers are described, of which B) is preferred:-

A) Polymers derived from one or more monomers at least one of which is a heterocyclic compound carrying an ethylenically unsaturated group. Heterocylic compounds with tertiary nitrogen atoms which can be quaternized include pyridine and imidazole. Monomers from which the polymers can be derived include 2- and 4-vinyl pyridines and 2- and 4-vinyl imidazoles.

B) Polymers derived from one or more monomers at least one of which is an amino-acrylic monomer, which term is used to include acrylic and methacrylic acids, acrylamide, methacrylamide and derivatives thereof having a tertiary nitrogen atom, such as :-

dimethyl amino ethyl acrylate;
dimethyl amino ethyl methacrylate;
diethyl amino ethyl acrylate;
diethyl amino ethyl methacrylate;
dimethyl amino neo pentyl acrylate;
dimethyl amino neo pentyl methacrylate;
dimethyl amino propyl acrylamide;
dimethyl amino propyl methacrylamide.

Tertiary nitrogen atoms may be quaternized before or after polymerisation of the monomers. Quaternisation techniques are well known in the art and will not be described here. Quaternizing agents may be monofunctional or polyfunctional, and it may be useful to use a polyfunctional agent either alone or in admixture with a monofunctional one, in order to achieve some degree of cross-linking of the polymer after application to the wool fibres.

Suitable quaternizing agents include:

Monofunctional -	methyl chloride; dimethyl sulphate; lauryl bromide; myristyl bromide; benzyl chloride;
Difunctional -	epichlorhydrin; 1,5-dibromopentane.

Monomers containing tertiary nitrogen atoms may be homopolymerized, or alternatively may be copolymerized with one or more other monomers such as:-

acrylamides;
methacrylamides;
acrylates;
methacrylates;
siloxanes;
vinyl esters;
vinyl alcohols.

The nature of this other monomer, if used, may be chosen to impart desired properties to the copolymer. For example, acrylamide may give rise to a copolymer having greater water-solubility than does methyl methacrylate. The molar proportion of the amino-group-containing monomer in the polymer (100% in homopolymers, less than 100% in copolymers) is chosen with various factors in mind. The proportion must be sufficiently large for the polymer to be soluble in water at the required concentration and to exhaust on to wool fibres at a satisfactory rate. If the proportion is too high, then the shrink-resist properties of the polymer on the wool may not be realised immediately or may be affected by subsequent dyeing or washing treatments, or dye uptake and fastness may be adversely affected. On the other hand, lower proportions may give rise to the flocculation problems discussed below. Copolymers in which the mole ratio of amino-group-containing monomer(s) to other monomer(s) is from 1:1 to 1:10, particularly from 1:2 to 1:5, are preferred.

The polymers may be prepared by solution polymerisation in a water-miscible organic solvent. The proportion of amino-group-containing monomer(s) needs to be chosen bearing in mind the need to perform the polymerisation and subsequent quaternization reactions in solution. In order to achieve a concentrated polymer solution at useable viscosity, a chain transfer agent may be included. Isopropanol is a suitable solvent and has the advantage of acting also as a chain transfer agent.

Polymerization conditions are well known in the art and not critical. Preferred conditions result in a concentrated solution of polymer in 50:50 isopropanol:water. This is diluted with water to 5% solids for eventual use as make-up for the treatment bath. In continuous operation, the polymer concentration in the bath reaches an equilibrium value generally in the range 0.1% to 1% by weight.

These polymers are water-soluble under acid conditions, and are preferably stored and transported at a pH of from 3 to 6. Under alkaline conditions, cross-linking may occur, particularly when quaternization has been effected using a difunctional agent such as epichlorhydrin.

A feature of the present process is that very low levels of polymer may be used and still produce acceptable shrink-resist properties. As stated previously, the precise level of polymer employed will depend on the concentration of chlorine used in the pretreatment, i.e. use of a high level of chlorine will generally mean that a lower level of polymer is required and vice-versa. Typically, the polymer should be applied to the wool at a level of 0.4-2.0% (o.w.w), preferably 0.5-0.8%.

In use, the aqueous polymer solution should be acid stabilised (preferably pH 3.5-4.0) and diluted to approximately 5% solids before being fed to the application bowl. The application bowl itself should be previously set at an alkaline pH (in the range pH 7-10 preferably pH 8.5-9.5) and maintained at this value by the addition of an agent such as sodium carbonate.

It has been found that during operation the polymer bowl sometimes becomes increasingly cloudy and eventually some flocculation may occur. Tests have established that this problem is caused by carry-over of liquor from the sulphite bath employed for the antichlorination treatment. It is believed that the flocculated material may well be protein degradation products, these being anionic and therefore incompatible with the polymer. The quantity of flocculated material which develops in the bowl is quite small in relation to the amount of polymer which has been added and this would be consistent with it being a protein-polymer complex because the amount of solubilised protein brought forward into the polymer bowl will also be very small.

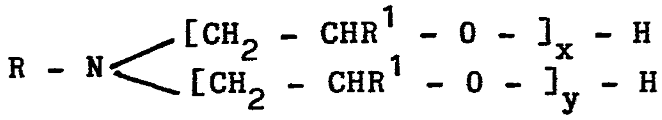
As will be apparent, the presence of even small amounts of such material can have a catastrophic effect in blocking holes in suction drum equipment and generally forming a sticky coating on all surfaces. It is true that flocculation is only produced by liquor from a sulphite bath which has been operating for some time and does not occur with clean sulphite solution.

However, this is obviously a considerable problem in a continuous process.

One way of avoiding the problem may be to use a homopolymer or a copolymer containing a high proportion of quaternary nitrogen atoms. However, although such polymers seem less prone to cause flocculation, they may give rise to other problems on dyeing. A preferred solution to the problem comprises the use of a water-soluble cationic material which may be either monomeric or polymeric. This material should react preferentially with the proteinaceous material and keep it in solution or suspension. It should be used in an amount small enough as not to substantially affect the exhaustion of the polymer on to the wool fibres; an amount of from 2% to 20% by weight on the weight of the polymer is suitable. The material may be added to the polymer concentrate followed by dilution of the mixture to the desired concentration for addition to the treatment bath. Suitable materials are available commercially; their chemical constitutions are often not published in detail, but it is believed that they generally contain tertiary or quaternary nitrogen atoms:-

Unisol UA 400	I.C.I. p.l.c.
Ethomeen T/25 and S/20	Akzo Chemie UK Ltd.
Ethoquad 0/12 and HT/25	Akzo Chemie UK Ltd.
Empigen BCT 40	Marchon Division of Albright and Wilson
Nalfloc 7607; 8102; 7107	Nalfloc Ltd.
Magnafloc HR 120	Allied Colloids Ltd.
Zetag 51	" " "

It will be noted that many of these materials are flocculating agents. They are here being used for the opposite purpose of keeping proteinaceous matter in solution or suspension. The Unisol and Ethomeen materials are believed to have the general formula:-



$$R - N \begin{cases} [CH_2 - CHR^1 - O -]_x - H \\ [CH_2 - CHR^1 - O -]_y - H \end{cases}$$

wherein R is $C_1 - C_{22}$ alkyl or alkenyl (preferably $C_{12} - C_{18}$ alkyl or alkenyl),
R^1 is H or CH_3, and
$x + y = 2 - 40$ (preferably 10 - 40).

When included in the polymer solution, compounds of this type have been found to reduce or completely inhibit the problem of turbidity and precipitation. Typical compounds which have this effect include:

Oleyl amine + 10 moles ethylene oxide,
Tallow amine + 15 moles ethylene oxide,
Tallow amine + 15 moles ethylene oxide quaternised with dimethyl sulphate and
Tallow amine + 40 moles ethylene oxide (used in Example 5 below).

Following application of the polymer the wool is dried. As stated earlier, the drying stage requires significantly less energy than that required by other comparable shrink-proofing processes, in order to achieve the same degree of drying of the wool. Indeed, care must be taken to avoid over drying the wool since this may cause handling problems due to the development of static electricity.

In order to obtain successful results with the present process, it is essential that the polymer is applied evenly to the wool. The use of a suction drum bowl, a standard item of equipment found in many wool treating plants, is considered to be a perfectly acceptable means for achieving this. However, we have surprisingly found that even application of polymer from a conventional backwasher bowl can be achieved with only minor and inexpensive modification of the equipment.

The following examples are included to illustrate the invention. Example 1 demonstrates the effectiveness of various polymers according to the present invention in imparting shrink-resistance to wool. Examples 2 and 3 are comparative examples serving to demonstrate the advantages of the present process over various other commercially available shrink-proofing processes. Example 4 relates to a number of industrial trials which have been carried out to illustrate the improved drying properties exhibited by the process of the invention. Example 5 is a comparative example illustrating the effectiveness of the additive in reducing turbidity and precipitation in the polymer bowl.

Example 1

64's quality wool tops were treated with one of several chlorine-containing continuous oxidative shrink resist pretreatments by well known standard procedures described in British Patent Specifications Nos. 1,073,441, 1,475,367 and 2,044,310.

After passing through the usual antichlor treatment bowl the wool was passed through a bowl containing the required polymer so that the wool picked-up 1% polymer solids and finally the tops were passed through a dryer operating at 75-80°C. The wool was spun to 2/24's worsted count and knitted to a cover factor of 1.29 Direct Tex. A swatch was then tested to the I.W.S. TM 185 3 hours standard in a Cubex machine.

The following polymers were tested (all ratios quoted are mole ratios):-

Type (a) polymer - 2:1 MMA: DMAEMA quaternised with myristyl bromide.

Type (b) polymer - 3.14:1 MMA: DMAEMA quaternised with epichlorohydrin.

Type (c) polymer - DMAEMA homopolymer prequaternised with epichlorohydrin.

Type (d) polymer - DMAPMA homopolymer quaternised with epichlorohydrin.

Type (e) polymer - 3:1 MMA: DMAPMA quaternised with epichlorohydrin.

Type (f) polymer - 3:1 MMA: 4 vinyl pyridine quaternised with epichlorohydrin.

Type (g) polymer - 3.125:1 BMA: DMAPMA quaternised with epichlorohydrin.

Type (h) polymer - 5.25: 1:1 MMA: acrylamide:DMAPMA quaternised with epichlorohydrin.

Type (i) polymer - 3.13:1 MMA:DMAEMA quaternised with 50/50 myristyl bromide/epichlorohydrin.

Type (j) polymer - 2:1 methacrylamide: DMAPMA quaternized with epichlorohydrin.

wherein MMA is methyl methacrylate,

DMAEMA is dimethyl amino ethyl methacrylate,

DMAPMA is dimethyl amino propyl methacrylamide

and BMA is butyl methacrylate.

The results obtained are given below:

	Treatment	% Area Felting Shrinkage (TM 185)		
		1 hr	2 hrs	3 hrs
i)	1.65% Available chlorine from hypochlorite at pH 7.5			
	No polymer addition	-2.9	-14.9	-25.4
	+ Type (a) polymer at pH8.0	+0.5	-3.5	-7.7
ii)	0.6% Available chlorine from sodium dichloroisocyanurate in the presence of potassium permonosulphate at pH 2.5			
	No polymer addition	-49.6		
	+ Type (b) polymer at pH8.0	-3.9	-5.7	-12.5
	+ Type (c) polymer at pH8.0	-2.7	-4.2	-7.4
	+ Type (d) polymer at pH9.0	-6.2	-7.8	-12.1
	+ Type (e) polymer at pH9.0	-1.6	-1.2	-3.4
	+ Type (f) polymer at pH9.0	-1.5	-4.2	-5.6
	+ Type (j) polymer at pH9.0	-2.0	-8.3	-9.1
iii)	0.6% Available chlorine from hypochlorite at pH 1.8			
	No polymer addition	-45.7		
	+ Type (f) polymer at pH 9.0	-1.8	-5.5	-6.7
	+ Type (g) polymer at pH 9.0	-3.2	-4.3	-6.5
	+ Type (h) polymer at pH 9.0	-3.1	-4.3	-6.4
iv)	1.2% Available chlorine from hypochlorite and 1.2% $KMnO_4$ at pH 7.5			
	+ Type (i) polymer at pH 9.0	-2.6	-0.2	0.0

Example 2

Wool tops were oxidatively treated by the chlorination process ii) of Example 1 and spun and knitted into fabric.

Polymer was then applied to the fabric by exhaustion from a bath at pH 9.0.

Effect of drying levels on efficiency of polymer

		Moisture Content %	Area Felting Shrinkage (%) TM185 (3 hours)
Polymer Type (b) applied to give 1% solids o.w.w.	Dried at 80°C	9.0	- 5.9
"	Air dried and conditioned	14.5	- 1.7
"	Undried	Wet	- 1.4
Conventional Polymer applied to give 2% solids o.w.w.	Dried at 80°C	8.5	- 21.0
"	Air dried and conditioned	14.5	- 36.4
"	Undried	Wet	- 55.3
"	Dried at 80°C plus 30 mins at 100°C		- 27.0
Pre-treated only			- 51 (1 hour)

This experiment was performed on a batch process, but the results are believed applicable to continuous processes.

Example 3

Wool tops were treated according to the process of the invention on a commercial backwasher range and dried by passing through a 3 drum dryer operating deliberately at a high temperature of 90°C. A quantity of tops were removed before entering the dryer and allowed to dry at room temperature. Samples were then immediately spun to 2/24's worsted count, knitted and tested according to the TM 185 test.

	% Area Felting Shrinkage (TM 185)	
	Processed through dryer at 90°C	Air Dried
Sample (A) Wool 21.6 micron	-6.6	-3.8
Sample (B) Wool 18.6 micron	-4.0	-4.1

Example 4

The treatment substantially as described in Example 1 ii) using polymer b), has been applied in a series of trials on three industrial plants which normally operate the same oxidative process as that being used for the pretreatment in these experiments. In each case the trials were run for approximately 1 hour with the machine speed and the initial dryer temperature being the same as for normal production.

Plant A	Using normal speed and dryer temperature for this plant, the wool emerged excessively over-dried. Steam input was reduced steadily throughout the run, the wool still

	being adequately dry at the end.
Plant B - Trial 1	This plant has a 3 drum dryer, normal temperature being 100, 90 and 85°C. Using these conditions the emerging wool was excessively over-dried.
Plant B - Trial 2	Dryer temperatures were reduced to 75-80°C on all drums, the wool being well dried throughout.
Plant C - Trial 1	Normal dryer temperature is 60-70°C. The wool was over-dry and gave problems on leaving the dryer due to electrostatic charge. Rapid cooling of the dryer by opening the side doors improved the running, the wool still being dry.
Plant C - Trial 2	Dryer temperature was set at 50-60°C. Problems were still encountered at the beginning of the run. Steam was turned off entirely and the run was completed with the wool satisfactorily dry.
Plant C - Trial 3	Initially the internal temperature of the dryer was 35°C. Steam remained turned off throughout the run, the final temperature being approximately 30°C. The wool was adequately dried throughout.

In all plants, the wool was dried to a moisture content below 18% using at least 20% less energy than had been required to dry to the same moisture content the wool routinely shrink-resist treated in that plant.

Example 5

32 ends of wool top sliver (21.5 μ) were given an oxidative treatment under industrial conditions by passing through a precision pad mangle containing an aqueous mixture of sodium dichloro iso cyanurate and potassium permono-sulphate at a through put of 380 kilos wool per hour according to the procedure described in BP 1,073,441.

The pre-treated sliver then passed through a four bowl backwasher where they were successively given an antichlorination treatment, a water rinse, application of a polymer, and finally, application of a cationic softener.

The sulphite bowl was maintained at a concentration of 1% and pH 8.5-9.0 by appropriate addition of sulphite solution and alkali.

The polymer bowl was fed with a 5% solution of polymer type (b) at a rate to give 0.75% polymer solids on weight of wool passing through the bowl. The pH of the bath was maintained at pH 8.5-9.0 by addition of alkali.

After 1 hours continuous production the solution in the polymer bowl had become cloudy and some flocculated material was present. Continued operation became progressively more difficult and rapidly impossible.

In a second experiment, identical conditions were used throughout except that an addition of 0.4 gram/litre of an ethoxylated tallow amine (40 moles ethylene oxide) was made to the polymer bowl before starting the run. Further additions were made by dissolving sufficient of the same ethoxylated amine in the polymer feed solution to give 0.5 gram/kg wool being processed.

In this way it was possible to continue the treatment for 8 hours without any trace of flocculation or deposition in the polymer bowl.

Tops from both experiments were spun into 2/24's worsted count yarn, and knitted to a cover factor of 1.29 Direct Tex. A swatch was then tested to the IWS TM 185 3 hours standard.

			% Area Shrinkage
1.	Experiment 1	TM 185 - 180'	5.9
2.	Experiment 2	TM 185 - 180'	7.6

CLAIMS

1. A continuous process for the production of shrink-resistant wool comprising the steps of :-

i) subjecting the wool to an oxidative chlorinating pretreatment;

ii) subsequently treating the wool with an aqueous solution of a polymer so as to cause the polymer to exhaust on to the wool fibres; and

iii) drying the wool to a chosen moisture level; characterized in that a polymer containing quaternized amino groups is used in step ii), and in that the wool is dried in step iii) to from 12% to 18% moisture, by weight on the weight of the dry wool, using at least 20% less energy than is required to dry to the same moisture level wool which has been rendered shrink-resistant by conventional continuous processes.

2. A process as claimed in claim 1, wherein the chlorinating pretreatment is carried out at a level of 0.25-2.0% active chlorine (by weight on the weight of the dry wool).

3. A process as claimed in claim 1 or claim 2, wherein the polymer is a copolymer of at least one amino-group-containing monomer with at least one other monomer, in which the mole ratio of the amino-group-containing monomer(s) to the other monomer(s) is from 1:1 to 1:10.

4. A process as claimed in any of the preceding claims, wherein the polymer is derived from at least one amino-acrylic monomer.

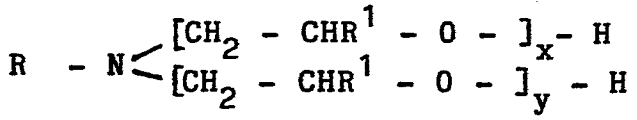
5. A process as claimed in any of the preceding claims, wherein the polymer is applied to the wool at a level of 0.4-2.0% (by weight on the weight of the dry wool).

6. A process as claimed in any one of the preceding claims, wherein the polymer contains amino

groups quaternized using epichlorohydrin.

7. A process as claimed in any of the preceding claims, wherein the polymer is applied to the wool in the presence of a water-soluble cationic material.

8. A process as claimed in claim 7, wherein the water-soluble cationic material has the formula



$$R - N \begin{cases} [CH_2 - CHR^1 - O -]_x - H \\ [CH_2 - CHR^1 - O -]_y - H \end{cases}$$

wherein R is $C_1 - C_{22}$ alkyl or alkenyl,
R^1 is H or CH_3, and
$x + y = 2 - 40$.

9. A process as claimed in claim 7 or claim 8, wherein the material is present at a level of 2-20% by weight on the weight of the polymer.

10. A process as claimed in any of the preceding claims, wherein the wool is dried to a moisture level of 12-15% (by weight).