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(54) **Treatment of wool.**

(57) A process at present widely used for the treatment of wool to improve its resistance to shrinkage entails subjecting the wool first to an oxidation treatment (eg with chlorine) and then to treatment with an epichlorhydrin resin to supplement the shrink resistance imparted by the oxidation treatment. Resin treatment is accompanied or followed by treatment with a softening agent to soften the harsh handle associated with use of the epichlorhydrin resin. This process generates an aqueous effluent which, through its content of chlorine covalently bound to carbon, is harmful.

By the present invention generation of such effluent is avoided, without loss of desirable properties in the finished wool, by using as the "supplementary treatment resin" a colloidal melamine resin, with a silicone polymer as softening agent.

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TREATMENT OF WOOL

This invention relates to the treatment of wool, and is particularly concerned with a process for improving its resistance to shrinkage.

A process very widely used for this purpose entails subjecting the wool to an oxidation treatment, usually a chlorination, as with free chlorine or a hypochlorite or dichloroisocyanurate salt; removing unreacted (and potentially very damaging) oxidising agent from the wool fibres, as by passage through a reducing medium such as sodium sulphite solution, followed by a water rinse; and then treating the wool with a synthetic resin to impart a resistance to shrinkage which supplements that given by the oxidation treatment. Usually thereafter, but sometimes simultaneously with the resin treatment, the wool is treated with a softening agent to soften the harsh handle which it would otherwise have as a result of the resin treatment; and in due course the treated wool is dried evenly at a moderately raised temperature to ensure that adequate curing of the resin has taken place.

The process just outlined can be applied to the treatment of wool tops (ie wool fibre before it is spun) as well as to the treatment of yarn and of woollen piece goods and garments.

Probably the resins most used in the process are those obtained by reaction of epichlorhydrin (a compound having a reactive epoxy group attached to the radical $-CH_2Cl$) with a polyamide whose repeating unit contains basic -NH-or $-NH_2$ groups, as distinct from the non-basic -NH- groups of the amide linkage -CO.NH-. Such a resin is for example that obtained by the reaction of epichlorhydrin with the condensation product of a polyalkylene amine eg triethylene diamine ($H_2NCH_2CH_2NHCH_2CH_2NH_2$) and adipic acid ($HO_2C.CH_2CH_2CH_2CH_2CO_2H$). Not all such resin is exhausted onto the wool from whatever medium the epichlorhydrin resin is applied, and there is now strong evidence that the resin-containing aqueous effluent from the process is harmful because of the presence in it of chlorine covalently bound to carbon, as in the $-CH_2Cl$ group.

We have now found that effluent problems can be greatly reduced, and a treated wool product obtained with properties just as good as those that result when an epichlorhydrin resin is employed, by using a colloidal melamine resin as the resin to impart shrinkage. Colloidal melamine resins are used in the treatment of paper to improve its wet strength, and are obtained by dissolving a partially condensed melamine formaldehyde resin in an acid such as hydrochloric, formic, acetic or lactic acid under controlled conditions, and further diluting with water. The solution becomes infinitely miscible with water, and on aging develops a bluish haze. The solution contains positively charged colloidal particles of the melamine resin.

According to the invention, there is provided a process for the treatment of wool to improve its resistance to shrinkage, by subjecting it first to an oxidation treatment and then to a resin treatment to supplement the shrink resistance imparted by the oxidation treatment, the resin treatment being accompanied or followed by treatment with a softening agent to soften the harsh handle associated with use of the resin, in which process the resin employed is a colloidal melamine resin and the softening agent employed is a silicone polymer.

Preferably, the silicone polymer is dispersed in the colloidal melamine resin,ie in the aqueous phase which constitutes the dispersion medium of that system.

There is described in EP-A-80272 a process for the shrink-resist treatment of wool, by

a chlorinating the wool with an acidified hypochlorite

b removing excess chlorinating agent from the wool

c applying to the acidified wool an acid colloid solution of a methylol melamine resin, which solution may contain a suitable softening agent, such as for example those of the polyethylene type.

EP-A-80272 is not, however, concerned to avoid effluent problems arising from the presence of $-CH_2Cl$ or like groups in the resin conventionally used for wool treatment; nor does it mention the use of a silicone polymer as softening agent, a use which we have found has the advantage over polyethylene softeners of producing in the wool better stabilisation against shrinkage and better non-felting properties during washing.

The silicone polymer employed as softening agent may be cationic, but is preferably non-ionic. In general the silicone polymer used shows no chemical reactivity towards the colloidal melamine resin.

The melamine resin employed is preferably one which has at least two $-CH_2OR$ groups per melamine nucleus, where R = H or is an alkyl group containing from one to four carbon atoms.

The invention is further illustrated by the following Examples.

Example 1

An acidic melamine resin solution was produced by mixing the following ingredients in the following

proportions by weight.

Aqueous solution (80% reactive solids; pH at 1:1 H_2O dilution=8.5) of partially condensed melamine formaldehyde resin available as BEETLE^R 336	10kg
Concentrated hydrochloric acid (specific gravity=1.16)	1kg
Water at 40° C	36kg

The mixture was allowed to stand for 2 hours to develop colloidal properties, and was then diluted with an equal volume of water to give a colloidal melamine resin having a shelf life of more than 6 months.

The diluted composition (1 part by weight) was further diluted with water (4 parts) and to this mixture was added an aqueous emulsion of a non-ionic polydimethylsiloxane (0.5 parts by weight; silicone content 35% by weight). (The silicone release agent sold by Basildon Chemicals under the designation BC330 is very suitable.)

The mixed melamine resin/silicone polymer composition was then applied, from a bath adjusted with acetic acid to pH 5-6, to knitted wool garments which had been conventionally chlorinated with a dichloroisocyanurate salt, treated with antichlor, and rinsed.The bath temperature was slowly raised from ambient to 40° C during 10 minutes, and held at that temperature for a further 10 minutes to exhaust the melamine resin and silicone polymer onto the wool. Weight gain was about 1.5% by dry weight of the wool.The material was then removed from the bath, hydroextracted and tumble-dried at 60-80° C.

Example 2

For the treatment of wool tops the procedure of Example 1 was modified as follows.

The mixed melamine resin/silicone polymer was applied in the 4th bowl of a conventional 5-bowl unit to deposit about 1.5% of solid mixture onto the tops. The treated tops were then passed to the 5th bowl, in which a cationic substituted stearamide had been included as additional softener. The tops were finally dried at 70-80° C.

Claims

1. A process for the treatment of wool to improve its resistance to shrinkage, by subjecting it first to an oxidation treatment and then to a resin treatment to supplement the shrink resistance imparted by the oxidation treatment, the resin treatment being accompanied or followed by treatment with a softening agent to soften the harsh handle associated with use of the resin, characterised in that the resin used is a colloidal melamine resin and the softening agent is a silicone polymer.

2. A process according to claim 1, in which the silicone polymer is dispersed in the colloidal melamine resin.

3. A process according to claim 1 or 2, in which the silicone polymer is non-ionic.

4. A process according to claim 3, in which the silicone polymer is a polydimethylsiloxane.

5. A process according to any of claims 1 to 4, in which the melamine resin has at least two $-CH_2OR$ groups per melamine nucleus, where R = H or C_{1-4} alkyl.

6. A process according to any of claims 1 to 5, in which the oxidation treatment is a chlorination.

7. A process according to claim 6, in which the chlorination utilises a dichloroisocyanurate salt.

8. A process according to any of claims 1 to 7, applied to the treatment of wool tops.

9. A process according to any of claims 1 to 7, applied to the treatment of knitted wool garments.

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EUROPEAN SEARCH REPORT

Application Number

EP 91 20 0072

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)
Y	GB-A-2 217 741 (BIP CHEM) * the whole document *	1-9	D 06 M 15/423 D 06 M 15/643
D,Y	EP-A-0 080 272 (SOUTH AFRICAN INVENTIONS DEVELOPMENT CORPORATION) * claims *	1-9	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

D 06 M

The present search report has been drawn up for all claims

Place of search	Date of completion of search	Examiner
The Hague	24 May 91	BLAS V.M.

CATEGORY OF CITED DOCUMENTS

X: particularly relevant if taken alone
Y: particularly relevant if combined with another document of the same catagory
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