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64 Textile treatment.

67 A method of treating keratinous fibres which comprises applying to pre-chlorinated loose wool, slubbings or tops a condensate of a water soluble or dispersible aldehyde with a nitrogenous base.

**EP 0 209 323 A2**

TEXTILE TREATMENT

This invention relates to a process of treating textiles, and in particular relates to a method of treating keratinous textile fibres to increase the resistance of fabrics or garments made therefrom to felting shrinkage.

Fabrics of keratinous fibres, in particular sheep's wool, are subject to felting shrinkage when washed. There are many processes designed to reduce this inconvenient tendency, from simple oxidation treatments to applications of synthetic polymers to the fibres.

To give adequate resistance to shrinkage, enabling fabrics and garments to exhibit full machine washability, polymer treatments are preferred. Most such treatments are only really successful when applied to the fibres when the latter are already processed as far as fabric or garment form - application at an earlier stage, loose fibre, sliver or yarn, not being successful. The main exception to this is the so-called 'chlorine/Hercosett' process in which a polyamine-epichlorhydrin condensate resin is applied to pre-chlorinated wool fibres in the form of sliver or 'top'. The tops are then gilled and spun into yarn in the normal way, and woven or knitted into fabrics and garments which exhibit good resistance to felting shrinkage. Over the years, no other resin has been as

successful in top treatments.

While the chlorine/Hercosett process has enjoyed a high degree of success it is not without disadvantages. Firstly, it is necessary for consistently acceptable results to employ an addition of 2% o.w.f. This results in a cost, at current prices, of about 10p. per kilo of wool treated. At lower treatment levels, washability is sensitive to various factors such as neutralisation conditions, use with certain dyestuffs, and reduction bleaching.

In addition, Hercosett as sold has a low solids content leading to high transport costs, and it also has a limited shelf life.

Furthermore relatively high chlorine levels are required, and a cationic softener is normally required to assist in drying/curing of the resin and to facilitate gilling following drying. With certain dyes (chromes) cationic softeners can reduce rubbing fastness. The wool must be dried to 5-10% regain to completely cure the polymer. In press packing, localised hardening of tops can require recombining prior to spinning. With low resin treatment levels, hardness and compaction of wool can occur in top dyeing with drastic reduction in backwashing efficiency. Extension of open knit structures can occur during

washing, as can extension in washing of worsted or woollen spun knitwear when overtreated.

The invention seeks to provide a process of improving the resistance to felting shrinkage of keratinous fibres improved in the above respects.

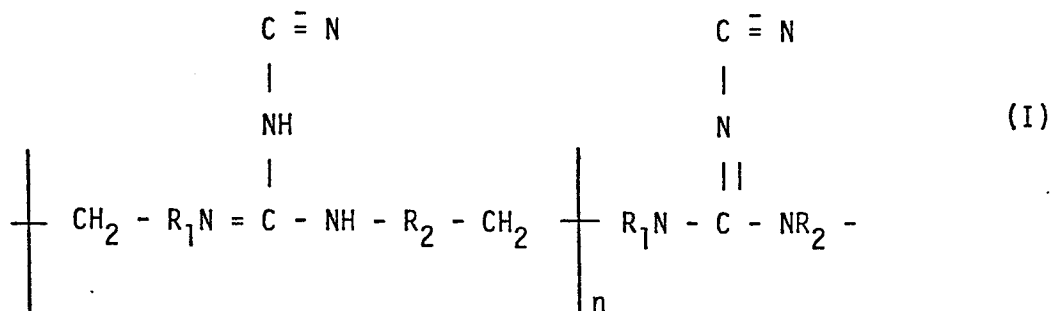
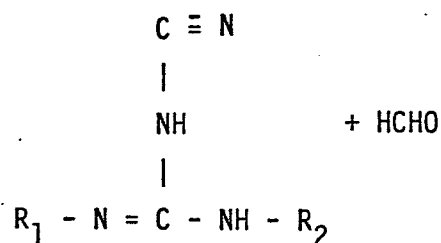
According to the present invention there is provided a method of treating keratinous fibres which comprises applying to pre-chlorinated loose fibre, slubbings or tops a condensate of a water soluble or dispersible aldehyde with a nitrogenous base .

The condensate is applied before spinning or subsequent processing, for instance as a sliver or top treatment, to pre-chlorinated wool and, as such, may be looked upon as a cheaper replacement for 'Hercosett' in the above mentioned 'chlorine/Hercosett' process, which is improved in the above respects. Application of the condensate should follow chlorination: simultaneous application may give some improvement in levelness of chlorination but gives no improvement in shrink-resistance over that provided by the chlorination itself. It is therefore surprising that the sequential treatment of the invention should give such a marked improvement of properties.

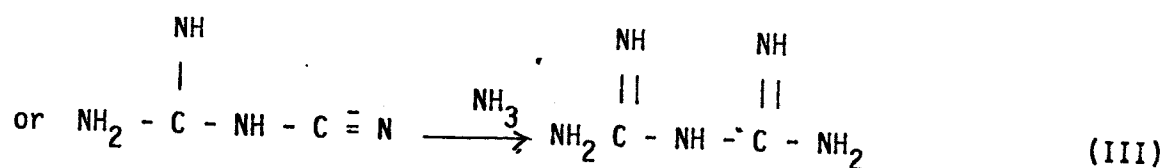
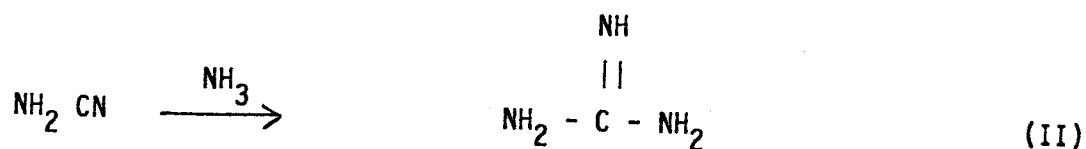
The condensates of the invention may be aminoplasts including reaction products of nitrogenous bases with

aldehydes which are either water soluble or water dispersible. Typical nitrogenous bases are melamine, guanamines and other triazines, urea, ethylene urea, propylene urea, rhiourea, guanidine, biuret, cyanamide, dicyandiamide, urons, and various derivatives thereof, as well as cyclic imines such as ethylene imine and propylene imine. Preferred are cyanamides and dicyandimidee. The aldehyde may be selected so as to form water soluble or dispersible condensation products with nitrogenous bases, but formaldehyde is the preferred aldehyde.

In particular the condensates employed in the process include:



together with condensates of cyanamides and cyanamide derivatives, such as guanidine and diguanide:



with formaldehyde; where  $\text{R}_1$  and  $\text{R}_2$  can be hydrogen or alkylene radicals and may contain other nitrogen atoms, e.g. be diamines, tetramines or polyalkylene amines.

Compounds of formula (I) in which both  $\text{R}_1$  and  $\text{R}_2$  are hydrogen are commercially available as cationic fixing agents for cellulosic fibres dyed with direct dyes. One such compound is Matexil FC-PN (ICI).

It has been surprisingly found that the above compounds, although not requiring crosslinking or 'curing' nevertheless impart shrink resistance to keratinous fibres when applied to sliver or top and overcome or reduce the problems listed above.

Pre-chlorination of the fibres is necessary for the

process of the invention to be effective. The pre-chlorination can be carried out immediately before treatment with the condensate or at some earlier time, either by batch or continuous methods. Any of the known methods of wool chlorination may be used, but acid/hypochlorite or chlorine gas treatments are preferred to DCCA treatments.

The invention will be illustrated further by the following example.

#### EXAMPLE

Following a chlorination pre-treatment level of 1.5% o.w.w. applied as either chlorine gas dissolved in water or a mixture of sulphuric acid and sodium hypochlorite (i.e. acid chlorination) the wool top is then antichlored and neutralised using a combination of sodium sulphite/metabisulphite with sodium carbonate and bicarbonate at pH 8.5 - 9.0 and 30<sup>o</sup> - 40<sup>o</sup> C. After rinsing the product (Matexil FC-PN) is applied continuously to a suction drum bowl at a feed rate of between 1.0 and 1.5% o.w.w. with an initial bowl make up of 5 g/l sodium bicarbonate and 4 g/l product. The bowl pH is automatically maintained at pH 7.5 using a feed of sodium carbonate. Subsequent processing involves a rinsing stage (optional) prior to drying.

Thereafter the top is processed into yarn and subsequently into fabric or garments. Wash and testing garments produced from fibres treated as above shows resistance to felting shrinkage to IWS 'superwash' standard is achieved.

Advantages of the process of the invention include:

1. Unlimited shelf life.
2. High solids content.
3. Lower chlorine levels can be used.
4. Insensitive to dyeing/bleaching/neutralisation in terms of washability.
5. Softener not required - good bulk without it.
6. Curing of the polymer not necessary.
7. Hardness of wool in ecru packing and dyeing eliminated.
8. Improved spinning performance.
9. Extension in open knit structures markedly reduced.
10. Improved handle.
11. Improved cockling performance/loop distortion.

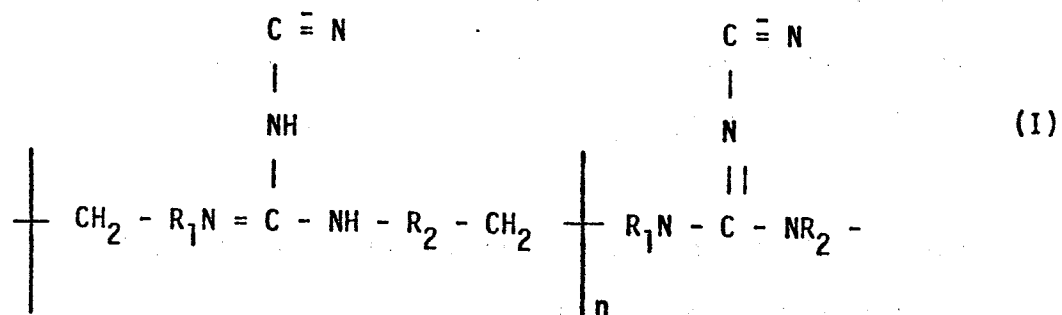
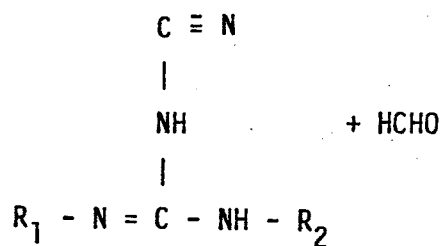


CLAIMS

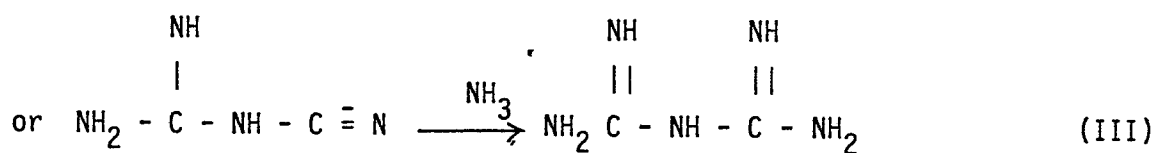
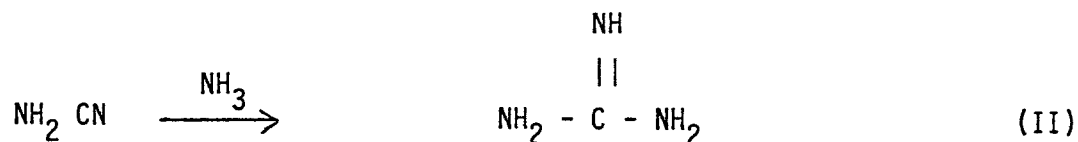
1. A method of treating keratinous fibres which comprises applying to pre-chlorinated loose wool, slubbings or tops a condensate of a water soluble or dispersible aldehyde with a nitrogenous base .
2. A method as claimed in claim 1 in which the condensate is an aminoplast.
3. A method as claimed in claim 2 in which the condensate is a reaction product of a water soluble or dispersible aldehyde with melamine, guanamines and other triazines, urea, ethylene urea, propylene urea, thiourea, guanidine, biuret, cyanamide, dicyandiamide, urons, and various derivatives thereof, as well as cyclic imines such as ethylene imine and propylene imine.
4. A method as claimed in any of claims 1 to 3 in which the nitrogenous base is cyanamide or dicyandimide.
5. A method as claimed in any of claims 1 to 4 in which the aldehyde is selected so as to form water soluble or dispersible condensation products with nitrogenous bases.
6. A method as claimed in claim 5 in which the aldehyde

is formaldehyde.

7. A method as claimed in any of claims 1 to 6 in which the condensate is:



together with condensates of cyanamides and cyanamide derivatives, such as guanidine and diguanide:



with formaldehyde; where  $\text{R}_1$  and  $\text{R}_2$  is hydrogen or an alkylene radical and may contain other nitrogen atoms, e.g. be a diamine, tetramine or polyalkylene amine.

8. A method as claimed in any of claims 1 to 7 in which top is treated with a chlorinating agent, then antichlored and neutralised; thereafter from 0.5 to 2% o.w.f. of the condensate is applied and the fibres dried.

9. A method as claimed in claim 8 in which the condensate is applied at an alkaline pH.

10. A method as claimed in claim 8 or 9 in which there is a rinsing step before drying the fibres.