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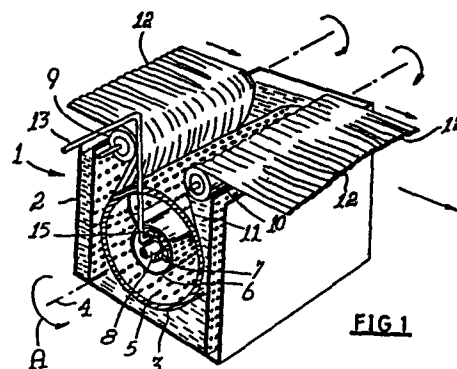
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⑥④ Chlorination of fibrous material.

⑤⑦ Method and an apparatus for the chlorination of a fibrous material (12), particularly wool and the like, in which a solution (11) of free chlorine in water, which is contained in the bowl (2) of a suction drum (1) of conventional construction, is sucked through the material (12) while the latter is passed around the lower half of perforated hollow cylinder (3). The suction in the bore of cylinder (3) is effected by an impeller (7) which is operative in a concentrically disposed tube (6). Solution (11) is obtained by dispersing the chlorine gas in it in the form of small bubbles by means of a mesh (15) which has apertures (16) and which is provided over the free end of a chlorine gas delivery tube (13). Mesh (15) is located one to two cm above shaft (8) of impeller (7) at a point located on that side of the apparatus where impeller (7) causes extensive turbulence in the solution to ensure that the chlorine is substantially completely dissolved in the solution.



CHLORINATION OF FIBROUS MATERIAL

THIS invention relates to the chlorination of fibrous material, particularly wool and the like, for the purpose of rendering such material shrinkresistant.

In this specification the term "wool" is intended to
5 include wool in the form of slivers as well as loose stock. The term wool sliver, in turn, is intended to include combed sliver, card sliver, top sliver, roving, yarn and thread while the term loose stock is intended to include scoured wool, carbonised wool,
10 broken top etc.

It is well known that the so-called felting shrinkage of wool and the like is related to the unidirectional movement of the fibres during washing and that such shrinkage can be counteracted by rendering the fibres
15 less liable to migrate in one direction only by means of a chemical degradative treatment and/or by treating them with a suitable resin.

In the case of wool sliver and loose wool, which have to undergo further processing, such as gilling,
20 drawing, spinning, weaving, knitting, etc. it is

imperative for optimum results that substantially each fibre of such material has to be treated in the aforesaid manner.

This has been found to be very difficult to achieve
5 successfully commercially and at present there are relatively few commercial processes available for the continuous shrinkresist treatment of such materials.

The most common of the aforesaid chemical degradative treatments used in the shrinkresist
10 treatment of wool and the like involves oxidation and, of the various processes which have been suggested in this regard, the most common one is chlorination.

The term "chlorination", as used in this context, is
15 very broad and includes the use of chlorine in various chemical forms according to a wide variety of treatments. These include aqueous chlorination treatments, chlorination in organic solvents, and dry gaseous chlorination. Of these, the aqueous
20 chlorination processes are by far the most popular for the oxidative treatment of wool.

Although it has been known for many years that an aqueous chlorine solution, formed by dissolving free chlorine in water, could be used in the shrinkresist treatment of wool and the like, there was a general belief that processes utilising such solutions were not readily controllable and/or satisfactory and that they tended to damage the wool fibre. Also, the fact that chlorine gas is very poisonous possibly detracted from its potential use as a chlorinating agent for such purposes. In this regard it may be pointed out that chlorine gas is not readily soluble in water and that any chlorine gas which would escape from the treatment liquor during such a treatment would be extremely hazardous to or, at least, uncomfortable for the operators of such a process.

There is extensive reference in the prior art to the development of chlorinating agents other than solutions of free chlorine in water which can be used in such shrinkresist treatments of wool and the like. In this regard it is significant to note that such development away

from free chlorine solutions took place in spite of the cost advantage which such solutions have over other chlorinating agents.

In this regard it is significant to note that
5 the costs per unit of reactable chlorine in the case of such aqueous chlorine solutions is only approximately 1/5 that of an acidified hypochlorite solution and that this ratio is
10 even less in the case of some of the other more sophisticated chlorinating agents which have been developed and commercialised over the years.

Accordingly, in spite of statements such as that contained on page 122 of The Textile
15 Manufacturer of March 1967 to the effect that for the continuous shrinkresist pre-treatment of wool the cheapest and simplest technique is to add chlorine from a gas cylinder at a controlled rate to a sulphuric acid solution, it was stated
20 by one of the co-authors of the same article in 1971, that the only economically viable shrinkresist treatment for wool tops then available was the CSIRO process which was developed in 1969 and which utilises as

chlorinating agent an acidified hypochlorite solution in a pad mangle- (British Knitting Industry 1971, p77). This statement was repeated, for example, in 1981 (Textilveredelung
5 Volume 16, p45 (1981)).

Of all the aqueous chlorination processes which have been made available to the industry over the years, the acidified hypochlorite process referred to above is still today the one most
10 commonly used for the chlorination of wool.

This process is based on the use of an alkali metal hypochlorite, particularly sodium hypochlorite, from which compound a suitable chlorinating agent can be prepared by acidifying
15 a solution of it with an acid, such as sulphuric acid, according to the following reaction:



It is generally accepted that the acidified hypochlorite solution does not contain
20 hypochlorous acid only, but that it comprises a mixture of hypochlorous acid and free chlorine, as is illustrated by the following equilibrium

reaction:



This equilibrium, and, accordingly, the relative amounts of chlorine and hypochlorous acid (which
5 are both capable of reacting with wool), at any given instance, seems to depend on the pH of the solution.

It has been found that the acidified hypochlorite process can best be carried out in
10 a suction drum type apparatus.

In such a suction drum, the material to be treated is made to pass around a hollow rotatable cylinder onto which the material is sucked and which is at least partly
15 submerged in the treatment liquor contained in the chlorination bowl of the apparatus. The cylinder is perforated over its entire circumference so that suction applied to its inside causes the treatment liquor to pass from
20 outside the cylinder through the material and via the perforations to the inside of the

cylinder. The suction is usually effected by means of a suitable impeller operative within the cylinder or by means of a suitable pump located elsewhere in the system.

- 5 Although the chlorination of wool with an acidified hypochlorite solution in a suction drum generally produces acceptable results, the process also suffers from a number of significant drawbacks. For example, the
- 10 temperature of the liquor in the bowl generally increases during the treatment, mainly due to the neutralisation reaction between NaOCl and H_2SO_4 , as well as due to the fact that any excess free alkali present in the sodium
- 15 hypochlorite solution has to be neutralised by the sulphuric acid. In this regard it may be pointed out that the pH of commercially available sodium hypochlorite solutions is usually above 11 and that these solutions
- 20 usually contain between 0,3 and 1,2% NaOH (mass/mass). The aforesaid increase in temperature not only results in an increase in the degree of yellowness of the wool, but also gives rise to a loss of chlorine to the

atmosphere.

In order to avoid this, it may be necessary to cool the reaction medium which means that provision has to be made in the system for
5 chilling apparatus.

Furthermore, there usually is an increase or build-up in the concentration of inorganic salts in the bowl during the treatment which stems from the fact that the sodium hypochlorite
10 solution contains some NaCl produced during its manufacture, while some Na_2SO_4 is formed during the reaction between NaOCl and H_2SO_4 . As a result of this, it may be necessary to change the reaction medium more
15 frequently: a time consuming operation which may give rise to production losses.

Other drawbacks relate to the fact that the liquid metering pumps required for the sodium hypochlorite/sulphuric acid system must be made
20 of special materials which are resistant to acids and strong oxidising agents and in practice it is found that these pumps frequently

break down. Furthermore, the chlorination with acidified hypochlorite is best carried out in a fairly narrow pH range, namely 1,5 to 2,0, and
5 deviations from this can cause problems. It is therefore also necessary to monitor and control the pH of the liquor in the chlorination bowl and this requires specialised equipment.

Furthermore, the concentration of the sodium
10 hypochlorite stock solutions tends to vary from batch to batch and also tends to decrease on storage and should accordingly be determined daily in order to calculate the amount of hypochlorite which has to be fed to the
15 chlorination bowl.

In a recent development away from the acidified hypochlorite process, a special type of apparatus, the so called "Kroy machine" was suggested for the shrinkresist treatment of wool
20 in which an aqueous solution of free chlorine is used as chlorinating agent. The principle of operation and the use of this apparatus have been described extensively in the literature and details thereof are, for example, disclosed in

S A Patent No. 76/2929, U S Patent No. 4,261,188
and British Patent No. 1,524,392.

In this apparatus, which has been designed to overcome the presumed difficulties associated
5 with a solution of free chlorine in water, the wool to be treated is made to enter the aqueous chlorine solution at an angle of not more than 10° from the vertical and is then immersed to a depth of at least one meter in the solution. It
10 is claimed that with this apparatus a very uniform degree of chlorination of the wool is possible and the explanation which is given for this is that as the wool is progressively drawn into the solution the air in the material is
15 gradually driven out and, due to the progressive increase in hydrostatic pressure and the resulting capillary action, the solution is progressively drawn into the material.

Applicant believes that in the case of the Kroy
20 apparatus and process, the potential problem of chlorine gas escaping from the treatment liquor is overcome by the fact that the chlorine gas is released into the treatment liquor at a

substantial depth and also because the material being treated is contacted with the solution for a period of time sufficient to ensure that most of the chlorine in the solution is removed
5 through reaction with the material. Apart from this, sophisticated seals are employed on the apparatus to try and minimise the escape of such chlorine gas. It is, however, significant to note that, in spite of all this, the new
10 apparatus still makes extensive provision for the collection of unspent chlorine gas which may escape from the treatment liquor.

The Kroy apparatus is completely different from the suction drums which are in common use in the
15 industry and in order to utilise the claimed benefits of the new process it is necessary to replace these drums at great cost with the new type of apparatus.

From the prior art, and from the fact that the
20 International Wool Secretariate is currently promoting the Kroy apparatus and process as an economically viable apparatus and process for chlorinating wool with a solution of free

chlorine in water in spite of the heavy investment in new equipment which this requires the industry to make, and to which the industry apparently is objecting, it is clear that until
5 the advent of the applicant's present invention all the development in this regard was focussed, for real or presumed reasons, away from suction drum technology.

It is an object of the present invention to
10 provide a method and means for chlorinating a fibrous material, particularly wool and the like, with an aqueous solution of free chlorine which the applicant believes have useful advantages over the known arrangements.

15 According to the invention a method of chlorinating a fibrous material, particularly wool and the like, includes the step of forcing a solution comprising free chlorine which is dissolved in a liquid medium to pass through
20 the material in a manner so that the solution can react uniformly with substantially every fibre in the material.

Applicant believes that by forcing the solution in this manner to react with substantially every fibre in the material, the chlorine in the solution is given the maximum opportunity to
5 react with the material so that there is substantially no unspent chlorine left in the solution emerging from the other side of the material. This, accordingly, reduces the amount of chlorine available for escaping from the
10 solution during the treatment process. Furthermore, because the solution reacts uniformly with substantially every fibre in the material, a very uniform degree of chlorination is possible.

15 Further according to the invention the method includes the step of locating and holding the material in the path of the solution in a manner to substantially prevent channeling.

In this manner the possibility of some of the
20 fibres escaping from reacting uniformly with the solution is minimised.

Further according to the invention the solution

is forced to pass through the material by means of suction applied to one side of the material.

Still further according to the invention the suction is that provided in a suction drum of
5 conventional construction in which the material is passed through a chlorination bowl charged with the solution.

The method according to the invention can accordingly be carried out with substantially
10 the same apparatus which is conventionally used for the shrinkresist treatment of wool and the like using acidified hypochlorite as the chlorinating agent.

Further according to the invention the chlorine
15 solution is prepared by passing chlorine gas into water under conditions which ensure that substantially all of the gas is dissolved in the water.

By ensuring that the chlorine gas is dissolved
20 as completely as possible in the water, the likelihood of any such gas escaping from the

solution prior to, or even after, passing the solution through the material, is substantially minimised. Furthermore, as a result of such complete dissolution a very homogeneous solution
5 is formed which ensures that the material is very evenly chlorinated.

In this regard it may be pointed out that one of the reasons why free chlorine solutions gave unsatisfactory results in the past was that due
10 to large bubbles of undissolved chlorine gas in the treatment liquor coming into contact with the material being treated, the material was unevenly chlorinated.

Further according to the invention the
15 dissolution of the gas is enhanced by passing the chlorine gas into the water under conditions of vigorous agitation.

Still further according to the invention the chlorine gas is dispersed into the water in the
20 form of small bubbles.

By dispersing the gas in this manner the

dissolution of the gas into the water is substantially enhanced.

Still further according to the invention the chlorine gas is passed into the water at such a
5 level below the surface of the water where maximum use can be made of the hydrostatic pressure and any turbulence in the water for enhancing the dissolution of the gas in the water.

Further according to the invention the solution is prepared by dispersing chlorine gas into water contained in a suction drum in the form of small bubbles at a point in close proximity to
10 the impeller of the apparatus.

Preferably the gas is released approximately one to two cm from the impeller shaft at a point where the impeller of the apparatus causes severe turbulence in the water.

15 Applicant has found that such turbulence conveniently serves to enhance the dissolution of the gas in the water.

Further according to the invention apparatus for carrying out the aforesaid method includes a suction drum of conventional construction in which the material to be treated can be drawn
5 over a hollow rotatable cylinder which is perforated over at least part of its circumference and which includes an impeller which is operative in the cylinder to cause a solution of chlorine gas in water contained in
10 the chlorination bowl of the apparatus to be sucked via the perforations in the cylinder through the material while the latter is located on the drum

In one form of the invention the apparatus may
15 include a chlorinating means located outside the suction drum for providing the free chlorine solution.

In another form of the invention the apparatus may include means for dispersing chlorine gas in
20 the form of small bubbles in the cylinder.

In this form of the invention the suction in the

cylinder of the apparatus is preferably caused by an impeller operative in the cylinder, the dispersing means being located in the cylinder in a position in close proximity to the impeller.

- 5 In one form of the invention the dispersing means may comprise a fine mesh through which the gas is introduced to the water.

Such mesh may, for example, be of a suitable metal, such as stainless steel, or a suitable
10 synthetic resinous material, or sinter glass etc.

In another form of the invention the dispersing means may comprise a tube for passing the chlorine gas into the water, the end of the tube being closed off and the tube being provided
15 with a plurality of fine holes extending through the wall of the tube in positions spaced a small distance from the said closed off end.

In yet another form of the invention the dispersing means may comprise a plurality of
20 small bored tubes, with or without dispensing devices such as those disclosed above, for

passing the chlorine gas into the water.

Applicant has found that with the method and apparatus of the invention it is possible to chlorinate wool very evenly to the extent where
5 it is possible to obtain the advantages of the aforesaid new process without the need of having to convert to the new apparatus.

If required, the wetting of the material can be facilitated by the use of a suitable wetting
10 agent.

It will be appreciated that the processing speed, i.e. the speed of the material through the chlorinating liquor, and thus the contact time of the material in the chlorinating liquor,
15 will be determined by a number of parameters, such as, the diameter of the suction drum; the rate at which the chlorinating liquor is passed through the wool; the concentration of chlorine in the bowl and the rate of addition of chlorine
20 to the bowl; the fibre diameter and/or material thickness; maximum speed of the plant; capacity of the dryer; etc.

Applicant has found that a processing speed of between 0,1 m/min and 10m/min, or even higher, is possible with the process according to the invention.

- 5 As illustrated in the following table, applicant has found that for the same level of treatment, chlorine gas dissolved in water caused less yellowing of wool than an acidified hypochlorite solution.

Treatment	Whiteness Index*	
	Wool Lot	Wool Lot
	222	0B
1% Active chlorine ($\text{NaOCl} + \text{H}^+$)	-4,5	-6,1
2% " " "	-2,3	-5,3
3% " " "	-0,2	1,8
2% Chlorine gas solution	-11,9	-15,4

- 10 *The more negative the value, the whiter the wool.

In practice applicant has found that the

temperature of the chlorinating solution in the suction drum can vary between 5 °C and 40 °C without having any significant adverse effects on the shrinkresistance being obtained with
5 material being treated.

Applicant has also found that, in general, the temperature of the solution in the bowl increases only slightly during the treatment and it is suspected that this is mainly due to the
10 fact that there is no neutralisation reaction as is the case when alkaline hypochlorite and sulphuric acid are used. Thus, for example, applicant has measured the temperature in the chlorination bowl after 2
15 hours of continuous treatment and found that acidified hypochlorite gives a more pronounced rise in temperature than chlorine gas dissolved in water. For example, in the case where no chiller or cooling device was used in the
20 chlorination bowl, and with an ambient air temperature of 26 °C, the temperature of the solution in the bowl increased from 23 °C, in the case of acidified hypochlorite to 38 °C, while in the case of chlorine gas in water it

only increased to 28°C.

Although the pH of the chlorinating solution in the present invention depends mainly on the concentration of chlorine in the bowl, and
5 remains fairly constant when no material is passed through the bowl and while there is no change made to the concentration of the solution, applicant has found that when material is passed through the bowl the pH of the liquor tends to
10 decrease, probably as a result of the reaction between the chlorine and hypochlorous acid in the solution and the material. The pH of the chlorinating solution therefore generally tends to decrease slowly as the time of treatment
15 increases and it seems to reach equilibrium conditions at a pH value of between 1,0 and 2,8. In practice acceptable results have been obtained at pH values varying from 0,5 to 5,0.

20 The level of chlorination, i.e. the amount of chlorine applied to the material, depends on the properties of the material used, the presence of grease and foreign matter, such as lubricants

and oils on the fibre, as well as on the level of shrinkresistance required. In practice the level of the chlorination has been found generally to vary between 0,4% and 5,0% Cl_2 (on mass of material). At higher Cl_2 levels applicant has found that a very lustrous product is formed, probably because most of the scales of the fibres are removed. If it is required to enhance the formation of such a product, the upper level of the chlorine could be increased to 10%. It is also possible to operate at a lower level than the stated 0,4%. It will be appreciated that the chlorination process according to the invention can be used as oxidative shrinkproofing treatment only, or it can be followed by a subsequent resin treatment. Any resin or polymer having the properties required for continuous material treatment can be used, e.g. the acid colloid of melamine type resins, polyamide-epichlorohydrins, polyurethanes, polyacrylates, certain silicones such as polysiloxanes, certain cationic polyamides, etc.

In view of the fact that the material leaving the suction drum bowl carries some water with it

from the bowl, some water should preferably be added to the bowl during continuous treatments. Such added water could be utilised for introducing chemicals such as, for example, a
5 wetting agent, to the reaction medium.

In practice it is also advisable to discharge the contents of the bowl after an appropriate period of treatment (e.g. every 2 hours) so as to dispose of the build-up of impurities in the
10 bowl.

Although the actual "life" of the bowl depends on various factors, such as, the nature of the material, the amount of chlorine gas used, the capacity of the bowl, the amount of water
15 removed from the bowl by the material leaving it, etc, applicant has found that it is possible to run the process according to the invention for longer periods (in some cases even longer than four hours) than what the case generally is
20 with other processes before having to discharge the contents of the bowl.

One embodiment of apparatus according to the

invention will now be described by way of example with reference to the enclosed drawings wherein:

5 Figure 1 is a diagrammatic perspective view of a suction drum and related equipment according to the invention; and

Figure 2 is a similar view showing part of the apparatus of figure 1 in more detail.

10 In this embodiment of the invention a suction drum 1 is provided which includes a chlorination bowl 2 in which a hollow elongated cylinder 3 is located for rotation at a predetermined variable speed in the direction of arrow A about its longitudinal axis 4 by means of a motor or the
15 like (not shown).

Cylinder 3 includes over its entire circumference a plurality of perforations 5 by means of which bowl 2 communicates with the bore of cylinder 3.

20 An electrically operated impeller 7, which is

mounted on a rotatable shaft 8, is located in a tube 6 which extends concentrically in the bore of cylinder 3. Tube 6 is perforated over the lower half of its circumference.

5 On rotation of shaft 8, impeller 7 forces chlorinating liquor 11 to move rapidly away from it and out of tube 6 thus causing a suction action in the bore of cylinder 3 which causes the liquor to pass from bowl 2 via perforations
10 5 into the bore of cylinder 3. While impeller 7 is in operation, there is accordingly a constant flow of liquor 11 from bowl 2 to the inside of cylinder 3. At the same time impeller 7 causes extensive turbulence in the liquor contained
15 inside cylinder 3 especially in the immediate proximity of impeller 7.

The other end of tube 6 leads into the cavity defined by the double wall at the end of bowl 2, from where the chlorinating liquor 11 is returned, in
20 conventional manner, to the inside of bowl 2 via the apertures on the inside member of the double walls. If required, water may be added to the liquor so returned to bowl 2 and such fresh water may, for example, be used to convey

such other chemicals as may be required to be introduced to the bowl.

A pair of rollers 9 and 10 serves as the conveying means for a continuous ^{web 12} of adjacently disposed
5 longitudinally extending fibres of wool which may comprise wool slivers (or loose wool supported on a suitable netting or the like) which passes around the lower outside half of cylinder 3.

10 On rotation of cylinder 3, web 12 is accordingly continuously pulled over rollers 9 and 10 to pass through chlorination bowl 2 and around cylinder 3.

Drum 1, cylinder 3, tube 6, impeller 7 and rollers 9 and 10 are preferably of a suitable
15 corrosion resistant material such as stainless steel, for example.

Bowl 2 is filled with chlorinating liquor 11 to a level at which cylinder 3 is substantially completely submerged.

20 A tube 13, of which the one end is connected via a suitable flow meter and regulator (both ^{not} shown)

to a cylinder of chlorine gas (not shown), has its other end 14 located in the bore of cylinder 3 at a point approximately 1 to 2 cm from impeller shaft 8.

5 End 14 of tube 13 is closed off with a fine wire mesh 15 of stainless steel. The apertures 16 of mesh 15 are of such a size that gas issuing through them will be dispersed as fine bubbles into chlorinating liquor 11.

10 In operation, chlorine gas is introduced into the water in the bore of cylinder 3 as a continuous stream at a predetermined flow rate via apertures 16 in wire mesh 15.

Because mesh 15 disperses the chlorine gas in
15 the form of tiny bubbles, and because of the vigorous turbulence caused by impeller 7 in this area, as well as the fact that the gas is introduced at a substantial depth in liquor 11, the chlorine gas is substantially completely
20 dissolved in liquor 11 to form a substantially homogeneous solution. As a result of this, there is substantially no loss of gas to the atmosphere from the surface of liquor

11.

Furthermore, because the chlorinating liquor 11 is forced to pass through the wool web 12 while the latter passes around cylinder 3,

5 substantially each fibre of material in web 12 is contacted by chlorinating liquor 11 thus allowing the latter a substantial period of time for reacting with each of such fibres. The suction caused to web 12 also serves to locate
10 and to hold the material on the cylinder in the path of the chlorinating liquor in this manner preventing channelling of the material. As a result of this, each fibre of the material is uniformly treated with the solution and a very
15 even chlorination of the material is accordingly possible.

It will be appreciated that the material 12 may be treated on the upstream side of roller 9 in any of the conventional manners required for
20 rendering such material in a form suitable for chlorination and that the material 12 on the downstream side of roller 10 may be subjected to such further treatment as may be required such as rinsing, dechlorination, resin application
25 etc.

The method according to the invention will now be described further by way of the following examples and with reference to the drawings.

Example 1

5 Bowl 2 of suction drum 1 was filled with water at 15,5 °C after which 0,1% (mass/volume) Alcopol* 650 (Allied Colloids Ltd), a wetting agent which is resistant towards oxidising agents, was added. Chlorine gas was then fed as
10 a continuous stream via wire mesh¹⁵ meter (not shown) to give a chlorine content in the bowl of approximately 0,1% (mass/volume).

Twelve wool tops were then passed continuously as a web 12 through suction drum 1 at a speed of
15 3 metre/minute and the chlorine gas flow rate was adjusted to give 2% chlorine on the mass of the wool. Upon emerging from suction drum 1, the wool 12 was squeezed through squeeze rollers to remove excess liquor, followed by rinsing and
20 drying. The wool slivers showed an even or uniform treatment upon staining with a dye. The slivers were converted into yarn, from which a fabric was

* Registered Trade mark

produced. This fabric showed a percentage area shrinkage of about 8 to 10% when washed for 3 hours in a Cubex* machine, indicating that the chlorinating treatment produced an excellent

5 . degree of shrinkresistance.

Example 2

Twelve wool tops 11 were treated with 2% chlorine (on mass of wool) in suction drum 1 in the manner described in Example 1. Upon

10 emerging from suction drum 1, the slivers 12 were squeezed through squeeze rollers and then passed into a dechlorination bowl containing a solution of 1% (mass/volume) sodium bisulphite and 1% (mass/volume) sodium bicarbonate. Thereafter

15 the wool 12 was passed through squeeze rollers into a rinse bowl containing water, and then through squeeze rollers into a bowl containing 1% (volume/volume) acetic acid. The wool was then passed through squeeze rollers into a resin
20 bowl containing 2,0% (mass/volume) acid colloid prepared from Cassurit HML (Hoechst Ltd) and 1,0% (mass/volume) Polythem N-40 (Textile Chemicals

*Registered

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Ltd) as softening agent. The wool 12 was then passed through squeeze rollers (at a wet pick-up of 60%) into a dryer where it was dried and the resin cured. The slivers 12 were then
5 converted into yarn, and a fabric was knitted from the yarn. This fabric showed a percentage area shrinkage of about 1,0% when washed for 3 hours in a Cubex* machine, indicating an excellent degree of shrinkresistance.

10 Example 3

Twelve wool tops 11 were treated with 2% chlorine (on mass of wool) in suction drum 1 in the manner described in Example 1. Upon emerging from suction drum 1, the slivers 12
15 were squeezed through squeeze rollers and then passed into a dechlorination bowl containing a solution of 1% (mass/volume) sodium carbonate and 0,5% (mass/volume) sodium sulphite. Thereafter the wool 12 was passed through
20 squeeze rollers into a rinse bowl containing water, and then through squeeze rollers into a bowl containing 0,5% (mass/volume) polyamide epichlorohydrin polymer

*Registered Trade Mark

(e.g. Hercosett^{*}) 125 at pH 7,5 and the addition of the polymer was continued, at a rate of 2,0% polymer (on mass of wool). The wool 12 was then passed through squeeze rollers into a bowl
5 containing 0,2-0,5% softening agent (while a softening agent solution was simultaneously pumped into the bowl at a rate of 0,5% softener (on mass of wool). The wool 12 was then passed through squeeze rollers into a drier where the
10 wool was dried and the polymer cured. The slivers 12 were converted into yarn, and a fabric was knitted from the yarn. This fabric showed a percentage area shrinkage of less than 3,0% when washed for 3 hours in a Cubex*
15 machine, indicating an excellent degree of shrinkresistance.

Some typical results which have been obtained on two different wool lots 12 using chlorine gas in suction drum 1 under various conditions, are
20 given in Table I. For purposes of comparison some results obtained using the conventional sodium hypochlorite/sulphuric acid process (1,0 to 3,0%

*Registered Trade Mark 1.

active chlorine) in a similar apparatus, are also given in table I.

In table II some of the results obtained with a method according to example 3 above are given,
5 while in table III some results are given which were obtained carrying out a similar method but on loose wool using a modified suction drum apparatus. Polyester netting was used to feed the wool to the suction drum 1 and to support it
10 on the lower half of cylinder 3 in the bowl.

It will be appreciated that the process according to the invention can also be utilised for woven and knitted fabrics and that it also extends to blends of wool with other fibres.

15 It will further be appreciated that also intended for inclusion in the scope of this invention is a fibrous material which has been shrinkresist treated with the process and/or
20 apparatus according to the invention.

It will still further be appreciated that there are many variations in detail possible with a

process, apparatus, and product according to the invention without departing from the scope of the appended claims. Thus, for example, if required, a catalyst or the like may be employed
5 for enhancing the wool chlorination reaction.

CLAIMS

1

A method of chlorinating a fibrous material,
particularly wool and the like, including the step
of forcing a solution comprising free chlorine
which is dissolved in a liquid medium to pass
5 through the material in a manner so that the
solution can react uniformly with substantially
every fibre in the material.

2

The method of claim 1 including the step of
locating and holding the material in the path of
10 the solution in a manner to substantially prevent
channeling.

3

The method of any one of the preceding claims
wherein the solution is forced to pass through the
material by means of suction applied on the
15 one side of the material.

4

The method of claim 3 wherein the suction is that

provided in a suction drum of conventional construction in which the material is passed through the chlorination bowl charged with the solution.

5

- 5 The method of any one of the preceding claims wherein the chlorine solution is prepared by passing chlorine gas into water under conditions which ensure that substantially all of the gas is dissolved in the water to form a substantial
10 homogeneous solution.

6

The method of claim 5 wherein the dissolution of the gas is enhanced by passing the chlorine gas into the water under conditions of vigorous agitation.

7

- 15 The method of any one of claims 5 or 6 wherein the chlorine gas is dispersed into the water in the form of small bubbles.

38

-8

The method of any one of claims 5, 6 or 7 wherein the chlorine gas is passed into the water at such a level below the surface of the water where maximum use can be made of the
5 hydrostatic pressure and any turbulence in the water for enhancing the dissolution of the gas in the water.

9

The method of any one of the preceding claims wherein the solution is prepared by dispersing
10 chlorine gas into water contained in a suction drum in the form of small bubbles at a point in close proximity to the impeller of the apparatus.

10

The method of claim 9 wherein the gas is released approximately one to two cm from the
15 impeller shaft at a point where the impeller causes severe turbulence in the water.

11

Apparatus for chlorinating a fibrous material, particularly wool and the like,
including a suction drum of conventional

39

construction in which the material to be treated can pass around a hollow rotatable perforated cylinder in which suction is applied to force a solution of free chlorine in water contained in the bowl of the apparatus to pass through the material by sucking the solution via the perforations in the cylinder through the material while the latter is located on the drum.

12

The apparatus of claim 11 including a chlorinating means located outside the suction drum for providing the free chlorine solution.

13

The apparatus of claim 11 including means for dispersing chlorine gas in the form of small bubbles in the cylinder.

14

The apparatus of claim 13 wherein the suction in the cylinder is caused by an impeller operative in the cylinder, the dispersing means being located in the cylinder in a position in close proximity to the impeller.

40

15

The apparatus of claims 13 or 14 wherein the dispersing means comprises a fine mesh through which the gas is introduced to the water.

16

5 The apparatus of claim 15 wherein the mesh is of a suitable metal, such as stainless steel, or of a suitable synthetic resinous material or of sinter glass.

17

10 The apparatus of any one of claims 13 or 14 wherein the dispersing means comprises a tube for passing the chlorine gas into the water, the end of the tube being closed off and the tube being provided with a plurality of fine holes extending through the wall of the tube in positions spaced a small distance from the said closed off end.

15

18

The apparatus of any one of claims 13 or 14 wherein the dispersing means comprises a plurality of small bored tubes by means of which the chlorine gas is passed into the water.

41

19

A method of chlorinating a fibrous material, particularly wool and the like, substantially as herein described and with reference to the drawings and examples.

20

5 Apparatus for chlorinating a fibrous material particularly wool and the like, substantially as herein described with reference to the examples and as illustrated in the drawings.

21

The method or apparatus of anyone of the
10 preceding claims wherein the material is wool slivers or wool tops.

22

The product of the method or apparatus of anyone of the preceding claims.

DATED AT THIS DAY OF 1983.

PATENT ATTORNEY FOR APPLICANT

TABLE I
THE FELTING SHRINKAGE OF FABRICS KNITTED FROM
WOOLS WHICH HAD BEEN SHRINKRESIST TREATED

CHLORINATION TREATMENT	DECHLO- RINATION	RESIN (%)	% AREA SHRINKAGE	
			Wool Lot 222	Wool Lot OB
% Cl ₂ (O.M.W.)	Temp °C	Wetting Agent	Speed (m/min)	
CHLORINE GAS SOLUTION				
1,0	16	Yes	3	Yes
2,9	16	Yes	3	Yes
1,0	16	Yes	3	Yes
1,0	33	Yes	3	Yes
1,5	20	Yes	3	Yes
2,0	20	Yes	3	Yes
2,0	16	No	3	Yes
2,0	16	Yes	3	No
1,4	20	Yes	6	Yes
1,0*	20	Yes	3	Yes
2,0	20	Yes	3	Yes
3,0	20	Yes	3	Yes

* % Active Chlorine

TABLE II

THE FELTING SHRINKAGE OF FABRICS KNITTED
FROM WOOL SLIVERS WHICH HAD BEEN TREATED
WITH 2% (ON MASS OF WOOL) Cl_2 GAS IN WATER
AND 2% (ON MASS OF WOOL) HERCOSETT 125*

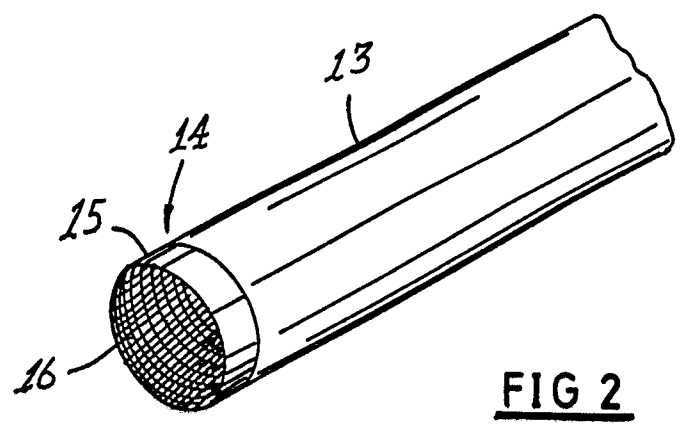
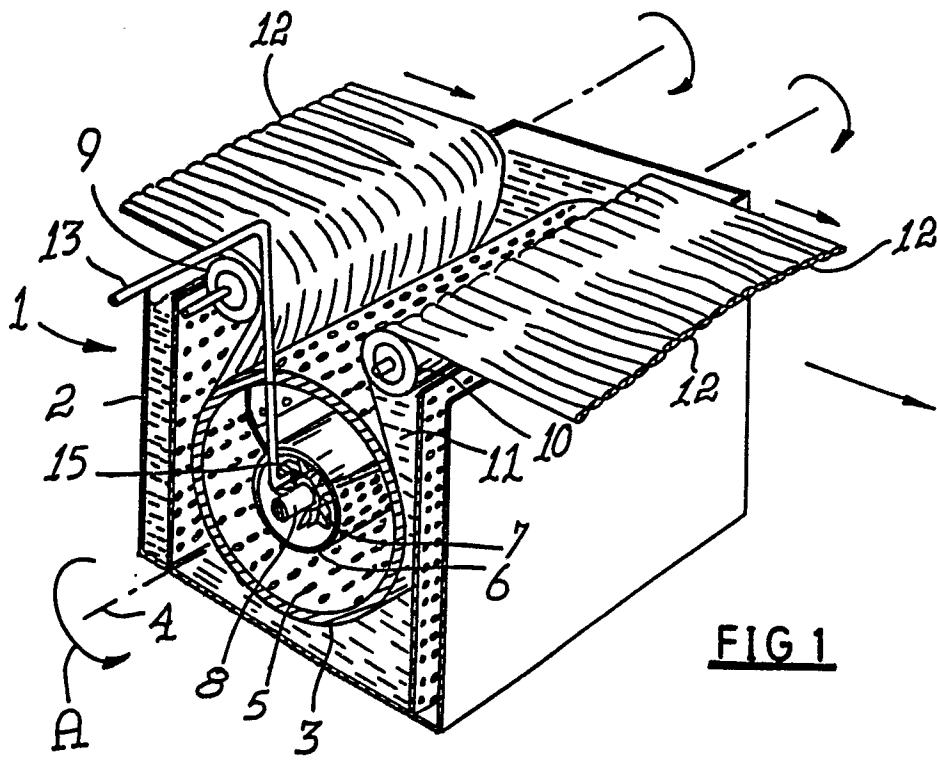
WOOL LOT	MEAN FIBRE DIAMETER (μm)	AREA SHRINKAGE (%)
A	20,6	5,8
B	21,7	9,0
C	22,9	5,7

TABLE III

THE FELTING SHRINKAGE OF LOOSE WOOL TREATED
WITH 2% (ON MASS OF WOOL) CHLORINE GAS AND
2% (ON MASS OF WOOL) HERCOSETT 125* IN A
MODIFIED SUCTION DRUM BACKWASH

WOOL LOT	MEAN FIBRE DIAMETER (μm)	FIBRE- LENGTH (mm)	AREA SHRINKAGE (%)
230	20,6	37,5	3,2

* REGISTERED TRADE MARK





European Patent
Office

EUROPEAN SEARCH REPORT

0103992

Application number

EP 83 30 4817

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. 3)
A	BE-A- 696 613 (VEPA) * Abstract; page 3, lines 16-19 *	1,3,4	D 06 B 3/22 D 06 M 3/02
A	--- GB-A-1 200 086 (VEPA) * Claims *	1,3,4 11	
A	--- GB-A- 417 719 (WOOL INDUSTRIES) * Claims; figure *	1	
A	--- FR-A- 982 839 (BRADFORD DYERS' ASSN) * Whole document *	1	
A	--- GB-A- 559 263 (BRANDWOOD) * Claims; figure *	1	TECHNICAL FIELDS SEARCHED (Int. Cl. 3)
A	--- TEXTILE RESEARCH JOURNAL, December 1957, pages 961-965 WM.H. HOUFF et al.: "Chemical damage in wool, Part III: Effects of aqueous chlorine" * Whole document * -----		D 06 M D 06 B
The present search report has been drawn up for all claims			

Place of search
THE HAGUE

Date of completion of the search
30-11-1983

Examiner
HELLEMANS W.J.R.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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