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TREATMENT OF WOOL

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This invention is a continuation in part of our application No. 362,695, filed October 24, 1940.

The invention relates to the treatment, in whole or in part, of materials consisting wholly or partly of wool to reduce or substantially eliminate their usual tendency to felt when washed in aqueous liquors. The wool may be in the form of loose fibres or in a manufactured form such as yarn and fabric which may have received some treatment such as scouring, bleaching, dyeing and hot pressing.

The term "wool" includes all animal fibres which are capable of being felted.

In describing this invention, the term "felt" is to be understood to refer to that property of ordinary wool which causes the individual fibres to close upon each other in washing, or other treatment in aqueous liquor in which the wool material is repeatedly squeezed and rubbed, so that the wool material becomes denser and more compact. This property of felting is responsible for the shrinkage which wool yarns and fabric suffer when washed, so that such materials may be made less shrinkable by treating them according to this invention.

According to the present invention, the tendency of wool to felt is reduced by restricted treatment with an alkaline substance dispersed in a monohydric alcohol capable of dissolving at least part of said substance, so as to reduce the tendency of the wool to felt without seriously affecting its quality. It is not necessary that all the alkali be dissolved since an alcohol with a small solubility for caustic alkali can be employed and undissolved caustic alkali in fine suspension, dispersion or emulsion may be present in the treating mixture. The term "dispersed" is used to include mixtures in which the solid is dissolved, partly dissolved, suspended or emulsified. After such treatment it is desirable to remove or otherwise render innocuous unchanged alkali and medium and the decomposition products to an extent such that the wool will not deteriorate during subsequent storage.

Materials containing wool which has already been partially felted may be prevented from further felting by the process herein described.

It is believed, although this does not limit this invention in any way, that the action of the alkaline treatment is mainly confined to the surface of such wool fibre, and that any substantial extension of this action to the interior of the fibre adversely affects its quality. The term quality is here to be understood to refer to a number of properties of the wool, such as tensile

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strength, softness of handle, colour and durability, which have to be taken into account in assessing the value and usefulness of a wool material. In general, the quality is reduced if the wool suffers a loss of weight and is made weaker, harsher, yellower and less durable.

The term alkaline substance is defined as a substance which in presence of water would give OH ions and it includes alkalis and organic bases or substances producing or capable of producing alkali or organic base, of stronger basic nature than ammonia.

Suitable alkaline substances are those which, in the presence of a suitable amount of moisture, may exert a gelatinizing action on wool and may to some degree produce chemical changes or decomposition of the wool, and are preferably those which are soluble or easily suspended, dispersed or emulsified in the organic media used. With solid alkaline substances it may be convenient mechanically to grind them to a fine state of subdivision either in the dry state or with any suitable organic liquid medium which may or may not be subsequently removed.

Among those alkaline substances found effective are organic ammonium, sulphonium and iodonium hydroxides such as benzyltrimethyl ammonium hydroxide; tertiary amine oxides; hydrazine and its hydrate; alkylates of metals such as sodium butylate and potassium ethylate; sodamido; metal derivatives of organic substances which hydrolyze with water such as the sodio derivatives of acetoacetic ester, acetone, and methyl ethyl ketone; sodium and potassium oxides, peroxides or hydroxides; alkaline substances which are gaseous at normal temperature and pressure may be used. This list is illustrative and is not intended to be exhaustive.

Suitable organic media include aliphatic alcohols such as methyl, ethyl, oleyl, ricinoleyl alcohol, and particularly alcohols containing 3 to 8 carbon atoms such as isopropyl and butyl alcohols; aromatic alcohols such as benzyl alcohol or phenylethyl alcohol $C_6H_5—CH_2—CH_2OH$, and hydro aromatic alcohols such as cyclo-hexanol, the methyl and ethyl and butyl ethers of ethylene glycol, and esters such as diethyl citrate, ethyl acetate, amyl acetate, benzyl chloride and ketones such as acetone or methyl ethyl ketone; pyridine, carbon tetrachloride, trichlorethylene, dichlorethylene, butadiene, isobutylene and petroleum distillates such as paraffin, hexane, white spirit (boiling range 150–260° C.) and petrol. This list is also illustrative and not exhaustive.

It has been observed that under certain condi-

tions of carrying out this invention, the wool may not only have its felting power decreased, but it may also suffer changes which reduce its original quality. It is therefore necessary to arrange conditions of restricted treatment which will produce the required decrease of felting power accompanied if necessary by any other change in the quality of the wool which is permissible or desired for the purpose in view.

In carrying out this invention, adequate control of the results obtained is possible because the conditions of treatment of the wool may be varied widely. It has been found that the quality and the decrease of felting power of the treated wool can be controlled by varying—

(1) The composition of the reagent as regards its content of one or more of the alkalis and one or more of the organic media mentioned above,

(2) Duration of treatment,

(3) Alkali-concentration,

(4) Temperature,

(5) Relative proportions of wool and reagent,

(6) The moisture content of the reagent,

(7) The moisture content of the wool immediately before its treatment with the alkali.

(8) The proportion of alkali in solution to total amount present in the organic medium.

Consideration must be given to all these conditions of treatment of the wool in order to obtain the result desired.

Thus if the wool is boiled with a solution of caustic soda in ethyl alcohol, the wool may become yellow in colour so that a temperature of not more than 60° C. is usually advisable, but if the wool is in contact with a restricted amount of the reagent then it may be heated to a high temperature.

It is further to be noted that this invention is carried out with alkali solutions, suspensions or emulsions which contain less than 15% of water by volume and may be substantially free from water. This water, if present, may arise from direct addition or its presence may be adventitious arising from difficulties of obtaining completely water-free ingredients of the alkali solution or preventing the entrance of water during the preparation of such alkali solution. The alkali solutions used in this invention are preferably those which contain not more than about 2% of water (by volume).

It has been observed that the action on wool of alkali-solutions containing appreciable amounts of water is generally harmful in that the quality of the wool is lowered. Wool harmed by such treatment usually has a pronounced yellow colour which may be accompanied by an impoverished handle and loss in weight.

The effect on the resulting decrease of felting power of the wool pronounced by adding water to the alkali solution varies considerably according to the composition of the alkali solution.

The moisture content of the wool when treated with the alkali solution influences both the reduction of felting power and the lowering of quality of the wool so that this moisture content may be varied widely according to the results which are desired. In general, wetting of the wool with water before treatment promotes a lowering of the quality of the treated wool. But to some extent, this harmful action may be counteracted by reducing the duration of the treatment or otherwise adjusting the conditions of the treatment. But even when these adjustments are made, the treated wool is generally less satisfactory than

when it is treated in an air-dry state. On the other hand, wool which is completely free from moisture is less reactive to the alkali treatment. Wool may also be conditioned in the vapour of hydroxylic substances other than water before treatment.

We have found that wool can be made nearly non-felting without appreciable loss of quality by treating it with an alkali-solution when the wool contains 12–18% of water, and these conditions are preferred.

Generally, the action of this alkali-treatment on wool with a given moisture content increases with increase of temperature and with increase of duration of treatment. At temperatures below 10° C. the action is slow, and at temperatures exceeding 60° C. it is rapid and more harmful to the wool; it is preferred to carry out the treatment at 15–20° C. As the duration of treatment is extended, a point is reached at which further treatment is harmful to the wool, without further reducing appreciably its felting power.

Again the treatment may be carried out under reduced pressure or increased pressure. In methods of carrying out the invention involving the use of reagents having constituents which are gaseous at ordinary temperature and pressure (e. g. butadiene) increased pressure is desirable. These volatile constituents may then be removed at the required time merely by releasing the pressure.

Conditions may also be arranged so that the effective concentration of base is increasing or decreasing during the treatment or is maintained constant in solution by having undissolved alkali present in the treating medium as either suspension, dispersion or emulsion.

At any stage in the treatment, the reaction may be interrupted and the wool suitably exposed to the action of reactive agents. Alternatively, the reactive agent may be applied to the wool before it receives the alkali treatment.

From the above general statements on the manner in which conditions such as composition of the alkali solution, temperature and duration of treatment, and the moisture content of the wool at the time of its treatment, affect the results obtained, it will be understood that these conditions can be much varied and that similar results can be obtained by different sets of conditions.

For the production of any particular desired result, it may be necessary to make a few simple trials to ascertain the most suitable conditions of treatment to be used.

The treatment of wool with these alkali dispersions, i. e. solutions, suspensions or emulsions, may be carried out in any convenient manner, all the conditions being adjusted so that wool having the desired properties and decreased felting power is produced. Thus the wool may be led through the alkali-solution, then squeezed to remove loosely adhering solution, and allowed to lie at room temperature for a sufficient period to obtain the result desired. Alternatively, and this is one of the preferred methods, the wool may be steeped in the alkali solution until the desired effect is obtained, and then excess liquor removed by any suitable method such as centrifuging to remove as much as possible of the alkali solution. It is found that a weaker alkali-solution may be used when the wool is treated so that it remains in contact for a considerable period with a relatively larger volume of it than when the wool is rapidly impregnated with a relatively

small volume of the liquor and then allowed to lie at room temperature for completion of the action, but the increase of alkali concentration required in the last named impregnation method may be reduced by allowing the impregnated wool to lie at a relatively high temperature, say 50 to 100° C. or even higher.

After the wool has remained in contact with the alkali solution under suitable conditions to obtain the desired reduction of its felting power, the alkali in the wool should be removed by thorough washing with water followed by treatment with an acid or by direct acid treatment followed by washing. In the former case care must be taken to avoid damage to the wool which may result from allowing it to be in contact with a high concentration of aqueous alkali. Alternatively the alkali in the wool is combined with substances so as to form products in or on the wool which have useful softening, lubricating or other effects; high molecular weight acids such as oleic acid, oleyl hydrogen sulphate may be used for this purpose. Alternatively, when high molecular weight organic bases are used as alkaline reagents they may be rendered innocuous by neutralisation with low molecular weight acids as well as high molecular weight acids to give softening or other effects.

Neutralisation in the absence of water may be carried out by treating the alkaline wool with acid gases or vapour or with a solution of an acid in an organic solvent.

Although it is believed that this invention will be largely used for all over treatment of materials consisting entirely or in part of wool for the purpose of rendering them non-felting, it is to be understood that it may also be used for treating such materials in selected parts or areas for the production of special effects. For example an alkali-solution may be applied to a woven wool fabric by textile printing methods or otherwise to produce a striped or other pattern so that when the fabric is afterwards washed it will felt to a less degree in the treated parts.

The invention is especially useful for the treatment of wool materials also containing fibres such as those of cotton and viscose rayon, since the alkali has no adverse action on these fibres. Other fibres which may be present with the wool are, for example, nylon, linen, rubber threads covered with cellulose as for instance those used in elastic belts and garters of woollen goods.

Example 1

A solution of caustic soda was made in n-butyl alcohol (0.64 gm. NaOH in 100 cc. of solution), 12.5 gm. of wool fabric, conditioned for moisture content by exposure to the vapour of saturated NaCl solution was immersed in the solution for one hour at 20° C. This sample was then removed, centrifuged, plunged into acidulated water, well rinsed in water and dried.

On drastic washing the shrinkage in area was 7% compared with 34% for the untreated fabric.

Wool fabric similarly treated with a liquor containing 1.4 gm. of NaOH in monoethyl ether of ethylene glycol also acquired increased resistance to felting.

For determination of shrinkage in washing the following method may be used.

A portion of the fabric is first steeped in water and then dried free from tension. A square of 10 cm. side is then marked on this relaxed fabric. The fabric is then washed (together with other fabrics for comparison when so desired) by hand with repeated squeezing in a ½% soap solution at 30–40° C. for a desired period, say 10 mins. The fabric is then rinsed in water, and dried free from tension. The marked square is then measured and the resulting contraction of area calculated and considered to be the area shrinkage.

Example 2

2 grams of solid caustic soda were dissolved in 50 ccs. of methylated spirit (commercial ethyl alcohol) and in this at room temperature was immersed a piece of air-dry scoured knitted fabric for three-quarters of an hour. The fabric was then mangled and rinsed with methylated spirit, washed with dilute aqueous acetic acid, rinsed with water and dried. It was found that in washing in a warm soap liquor this fabric had good resistance to felting.

Example 3

2 grams of air-dry scoured knitted wool fabric was immersed for 10 minutes at 30–40° C. in a mixture of 10 cc. of acetone and 30 cc. of ethylene diamine. It was then withdrawn, mangled, acidified with dilute hydrochloric acid and thoroughly washed. The resulting fabric was found to have considerable resistance to felting when washed with warm soap solution.

Example 4

Ten pounds of solid caustic soda was ground in a ball mill with 2 gallons of white spirit until the caustic soda had been ground to a very fine suspension. This gave a "sludge" of caustic soda in white spirit. 10 ccs. of this was diluted to 80 ccs. with white spirit.

A sample of knitted wool was wetted with ethyl glycol ether run through a mangle and then worked about in the suspension for 10 minutes at ordinary temperature. The wool was then again mangled, plunged into dilute acid and washed well in water. On vigorous washing and rubbing with a blank sample, resistance to felting and shrinking was shown.

The employment and use of three-part dispersions for the immersing of wool is described and claimed in our co-pending application, now U. S. Patent 2,367,273.

Example 5

Sodamide was reacted with excess of aniline with a stream of coal gas passing through the apparatus. The concentration of alkali in the resulting mixture was found to be equivalent to 0.07 g. NaOH per 100 ccs. Air-dry wool was immersed in this solution of sodioaniline in aniline for one hour at 80° C. After finishing in the usual manner this sample showed resistance to felting and shrinking when washed together with an untreated sample.

Example 6

12.5 gms. of air-dry wool fabric was treated for 24 hours at 20° C. with a liquor consisting of 0.1 gm. KOH dissolved in 100 cc. of ethyl alcohol. The fabric was then centrifuged and finished as in the preceding examples. The resulting fabric had a reduced tendency to felt.

The alcohol solutions of KOH as above may be replaced by a 6% solution of KOH in butyl alcohol at 45° C. the time of treatment being reduced to 2 minutes.

Example 7

0.3 gm. of lithium was added to 100 cc. propyl alcohol and allowed to stand until reaction was

complete. A sample of wool fabric was placed in the resulting mixture for 6 hours at room temperature and after removing it was washed and finished as in previous examples. The treated wool fabric had a reduced tendency to felt.

Example 8

10 ccs. of a commercial 40% aqueous solution of trialkyl benzyl ammonium hydroxide of approximate molecular weight 170 were treated with metallic calcium to remove water and the anhydrous base was dissolved in 90 ccs. of morpholine $NH(CH_2CH_2)_2O$. A sample of air conditioned wool fabric was immersed in this solution for half an hour, then centrifuged, immersed in dilute acid and well washed in water, soap and water and dried. It was found to possess good resistance to felting and shrinking when washed and rubbed with soap solution.

Example 9

Air-dry scoured knitted wool fabric was padded at room temperature with a solution of 10 parts of commercial benzyltrimethylammonium hydroxide and 90 parts of normal butyl alcohol. The commercial benzyltrimethylammonium hydroxide contained 40% organic hydroxide and 60% water. The fabric was then dried at 50° C. in air, further heated for five minutes at 120° C. then thoroughly washed in water, acidified with dilute aqueous acetic acid, thoroughly washed with water and dried. The fabric was then washed together with untreated fabric and thereby shown to have a much reduced tendency to felt. A 20% solution may also be used for a shorter time.

Example 10

A piece of air-dry scoured knitted wool fabric was steeped 20 minutes at room temperature in a mixture of 20 parts by volume of a commercial 40% aqueous solution of benzyltrimethylammonium hydroxide and 80 parts by volume of n-butyl alcohol. It was then withdrawn, mangled, washed, soured and rinsed. The fabric was then hand washed together with an untreated fabric and it was found that the treated fabric resisted felting almost completely.

It will be seen that in Examples 8, 9 and 10 the water content of the reagent is 0%, 6% and 12%. In general, the best results are obtained when the water content is not high and especially when using caustic soda, damage to the wool results if excessive amounts of water are present. It is preferred to use solutions containing not more than 15% by volume of water and desirably less than 10%.

Example 11

A solution of caustic soda was made in a decyl monohydroxylic alcohol fraction by shaking overnight with an excess of caustic soda at the ordinary room temperature. This mixture was centrifuged and the clear solution on analysis was found to contain 0.24 gram of caustic soda in 100 cc. solution. To 100 cc. of this solution 0.36 gram of very finely ground solid caustic soda was added. This latter remained undissolved and appeared as a fine suspension or dispersion throughout the mixture. A sample of air conditioned knitted wool fabric weighing 10 grams was immersed in this mixture at 18° C. with constant movement for one hour. It was then centrifuged and immersed in a dilute solution of sodium bicarbonate in order to neutralise the caustic alkali in the wool. Warm soap solution was then added and the material washed free from organic liquid.

It was then washed free from soap and dried. By conducting a hand milling test on a measured area of the treated and untreated sample the treated sample was found to have considerably resisted felting and shrinking.

The products according to the invention differ from non-felting wool prepared by treatment with chlorine or chlorine compounds in that they usually acquire a stiffer and fuller handle when wetted in slightly alkaline liquor but that on drying they lose this increased stiffness and fullness so as to regain their original handle. This characteristic of the wet product is in marked contrast to the thinner slippery handle of wool processed with chlorine or chlorine compounds.

We declare that what we claim is:

1. A process of reducing the tendency of wool to felt which comprises treating the wool at a temperature below 60° C. with a reagent consisting of an inorganic caustic alkali dissolved in a monohydric alcohol, the amount of water in the reagent being less than substantially 15 per cent. by volume and separating the wool from the alkaline liquid prior to any essential loss of weight and impoverishment of handle of the wool.

2. A process of reducing the tendency of wool to felt which comprises treating the wool at a temperature below 60 degrees C. with a reagent comprising an alkaline substance having a stronger basic nature than ammonia dispersed in a liquid capable of dissolving at least part of said substance, which liquid is preponderatingly a monohydric alcohol, said alkaline substance being capable in aqueous solution of producing a gelatinizing action on wool, the total quantity of water present in the reagent being less than 15 percent by volume.

3. A process of reducing the tendency of wool to felt which comprises treating the wool at a temperature below 60 degrees C. with a reagent comprising an alkaline substance having a stronger basic nature than ammonia dispersed in a liquid capable of dissolving at least part of said substance, which liquid is preponderatingly an aliphatic monohydric alcohol and containing not exceeding 15 percent of water by volume, said alkaline substance being capable in aqueous solution of producing a gelatinizing action on wool.

4. A process of reducing the tendency of wool to felt which comprises treating the wool at a temperature below 60 degrees C. with a reagent comprising an inorganic caustic alkali dispersed in a liquid aliphatic monohydric alcohol which has from 3 to 8 carbon atoms in the molecule, the amount of water in the reagent being less than 15 percent by volume.

5. A process of reducing the tendency of wool to felt which comprises treating the wool at a temperature below 60 degrees C. with an alkaline substance having a stronger basic nature than ammonia dispersed in a liquid organic solvent medium non-destructive of wool containing less than 2 percent by volume of water.

6. Process as in claim 5 in which a mineral caustic alkali is dispersed in a liquid hydrocarbon.

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