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PROCESS FOR SHRINKPROOFING WOOL

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1 Claim. (Cl. 8—127.6)

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This invention relates to the improvement of wool and other keratinous textile fibers, and more particularly to processes for reducing the shrinking tendency of such fibers by treatment with strongly acid aqueous solutions of chlorohydantoins.

A number of keratinous textile fibers, of which wool is the most important, are characterized by their tendency to felt when subjected to mechanical working. This tendency is essential in the finishing of some woolen goods to give a dense surface, as in the manufacture of felts for hats, paper machine blankets, and the like. However, since it causes shrinkage during laundering, it is a serious disadvantage in many other wool applications, such as knit goods, baby clothing, blankets, underwear, and socks. Considerable attention has therefore been given to methods of reducing or eliminating shrinkage due to felting.

In particular, it is well known that halogenation procedures render wool resistant to shrinkage on laundering. Included among the materials which have been employed for this purpose are the free halogens, inorganic and organic hypohalites, and N-halogenated organic compounds, such as N-chloroamides and imides. All of these have usually been applied from aqueous solutions. However, the serious disadvantages encountered in the commercial practice of present chlorination processes have greatly inhibited the usefulness of these methods. Thus, chlorinated wool generally is unevenly treated, harsh, and badly tendered, as indicated by decreased tensile strength and wearing qualities. In the continuous treatment of piece goods, the difficulty of "tailing" is encountered, that is, overtreatment of the first part of the fabric entering the bath and undertreatment of the latter part.

This invention is directed toward improving the physical characteristics of keratinous textile fibers by reducing their tendency to shrink on laundering. A further object is the production of wool of decreased tendency to shrink which is free from harshness, discoloration, and loss of strength. A still further object is to provide a new and improved halogenating process for reducing the shrinking tendency of wool.

These objects are accomplished by the following invention wherein a keratinous animal fiber such as wool is exposed to the action of an N-chlorohydantoin dispersed, and preferably dissolved, in an aqueous medium of pH 1 to 3.

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In carrying out the invention, wool or wool-containing textile material is immersed in a dilute solution of an N-chlorohydantoin and a strongly acidic buffer. The length of the treatment may be varied in accordance with the temperature and concentrations of the reagents used. However, as described in the following examples, a period of five minutes to twenty-four hours may be employed. The concentration of active chlorine and temperature of the treating bath are so adjusted that a satisfactory treatment is obtained during these treating periods. The treatment may be conveniently carried out at room temperature or at higher temperatures up to the range including 166° F. After treatment the excess solution is removed by wringing, centrifuging, or rinsing. It is often desirable to apply a dehalogenating agent, such as sodium thiosulfate or sodium bisulfite, to the treated wool in order to remove any excess treating agent or active chlorine.

As described above, the halogenating agent is generally used entirely in solution, and the best results are obtained in this way. However, agents of limited solubility can be used in suspension with satisfactory results.

In determining the effect of the treatment on the shrinking tendency of wool, it has been found convenient to use a uniform testing procedure, referred to hereinafter as the laundering test. In this test equal areas are marked on a small sample of wool fabric treated according to this invention and on a sample of otherwise similar untreated fabric, respectively. Then each is passed a definite number of times (usually 150) through a rubber-roll wringer, dipping after each pass into an aqueous solution at 77° F. containing 0.1% sodium carbonate and 0.25% of a sulfonated higher alcohol preparation known to the trade as "Gardinol" Special WA Paste. Other dilute alkaline soap solutions can be used in this test. After laundering, the samples are again measured. The area losses sustained by the samples are determined and the per cent reduction in shrinking tendency is calculated from the following formula:

$$\frac{(\textit{Area loss of untreated fabric} - \textit{area loss of treated fabric}) \times 100}{(\textit{Area loss of untreated fabric})} = \textit{Percent reduction of tendency to shrink}$$

The figures given in the following examples were determined by this method. The laundering test is also a convenient method of following the treatment, since a small sample may be taken from the textile material which is immersed in

the chlorinating solution at the end of a period judged to be suitable for obtaining the desired effect and tested in the above manner to determine the per cent reduction in shrinking tendency. If the material has not yet been treated to the desired degree, the process may be continued until another test indicates that the desired shrink-proofness has been achieved.

The following examples are illustrative of the practice of this invention. All proportions, unless otherwise stated, are by weight, one part of wool being used in each of the examples.

Example I

Wool flannel is immersed at 158° F. in 30 parts of a 0.15% solution (saturated at 77° F.) of 1,3-dichloro-5,5-dimethyl-hydantoin in 0.2 molar sodium acid sulfate solution at a pH of 1.5. The sample is removed after twenty minutes and the excess treating solution is wrung out. The wool is then immersed for five minutes in 3% aqueous sodium bisulfite solution and rinsed in water. The wool flannel is soft, free from discoloration, and substantially shrinkproof, the tendency to shrink having been reduced 90%. The tensile strength of the product is substantially unchanged from that of the untreated flannel. Repetition of the experiment in which the treating period is reduced to five minutes also yields an undamaged product, the shrinking tendency of which is reduced 70%.

Example II

Wool flannel is immersed at 77° F. in 30 parts of a 0.15% solution of 1,3-dichloro-5,5-dimethylhydantoin in 0.2 molar phosphoric acid solution at a pH of 2.1. After twenty-four hours, the material is removed, freed from the excess solution, and rinsed thoroughly in water. The laundering test shows that the shrinking tendency of the flannel has been reduced 51%. The tensile strength of the treated flannel is substantially unchanged from that of the untreated material. A similar sample treated under identical conditions except for the use of 60 parts of solution yields a flannel which is 100% reduced in shrinking tendency.

In contrast to these desirable effects obtained by using the chlorohydantoin at a low pH, a less shrinkproofed and highly damaged product is obtained when higher pH's are used. For example, wool flannel is immersed at 77° F. in 30 parts of a 0.15% solution of 1,3-dichloro-5,5-dimethylhydantoin in 0.2 molar monosodium phosphate solution at a pH of 4.3. After twenty-four hours, the product is removed, freed from excess solution, and rinsed thoroughly in water. The fabric is both unevenly and overtreated, badly harshened, and discolored. The laundering test shows that the shrinking tendency of the sample has been reduced only 34%.

Example III

Wool flannel is immersed at 158° F. in 30 parts of a 1.1% solution of N-monochloro-5,5-dimethylhydantoin in 0.2 molar phosphoric acid solution at a pH of 1.2. After five minutes, the fabric is removed, rinsed in water and then in 2.0% aqueous sodium thiosulfate solution. The treated flannel is soft, untendered, and 73% reduced in shrinking tendency. The tensile strength of the product is substantially unchanged from that of the untreated flannel. Repetition of the experiment in which the treating time is extended

to 20 minutes also yields an undamaged flannel which is 72% reduced in shrinking tendency.

Example IV

Wool flannel is immersed at 77° F. in 30 parts of 0.8% solution of N-monochloro-5-methyl-5-isobutylhydantoin in 0.2 molar phosphoric acid at a pH of 1.3. After twenty-four hours the fabric is removed, freed from excess solution, and rinsed thoroughly in water. The product, which is reduced 70% in shrinking tendency, is not discolored and shows a tensile strength substantially equivalent to that of the untreated product. A repetition of this experiment in which trisodium phosphate (pH >9) is substituted for the monosodium phosphate buffer yields a very discolored, harshened, and tendered product which disintegrates during the standard laundering test. This treated product exhibits greatly reduced tensile strength in comparison with the untreated flannel.

Example V

Wool flannel is immersed at 158° F. in 30 parts of a 0.2 molar phosphoric acid solution containing 0.15% of calcium hypochlorite (70% "available chlorine") and 0.13% of 5,5-dimethylhydantoin. After twenty minutes, the fabric is removed from this reagent combination which functions similarly to 1,3-dichloro-5,5-dimethylhydantoin. The flannel is freed from the excess solution by centrifuging and is then rinsed thoroughly in water. The fabric is evenly treated, soft, and has a tensile strength substantially the same as the untreated wool. The laundering test shows that the shrinking tendency of the sample has been reduced by 85%.

Among the acidic buffers suitable for use in this invention are those highly ionized inorganic and organic acids, as well as their acid salts, which are sufficiently soluble to be capable of buffering N-chlorohydantoin solutions in the pH range of 1–3 and which do not react rapidly with and destroy the efficiency of the chlorinating agent. The acidic compounds which are useful in this invention are termed herein "strongly acidic buffers." Specific examples include phosphoric acid, sulfuric acid, succinic acid, oxalic acid, and sodium acid sulfate. The exact control of pH in the range of 1–3 is particularly critical in this invention. The shrinkproofing effect of chlorohydantoins at this pH is rapid and complete while not damaging the wool, whereas the use of the same reagents buffered to approximately pH 4 and above cause marked yellowing and tendering of the wool and effect only a partial reduction in the shrinking tendency of the treated product.

Satisfactory shrinkproofing effects may be obtained by employing treating temperatures ranging from 70° F. to at least 170° F.

1,3 - dichloro - 5,5 - dimethylhydantoin is preferred as the chlorinating agent since it is effective, easily handled, stable in storage, and sufficiently soluble in aqueous solutions. In fact, the relatively low solubility of 1,3-dichloro-5,5-dimethylhydantoin (0.15% at 77° F.) is an advantage in the preparation of shrinkproofing baths inasmuch as saturated solutions of this material contain the preferred concentration of chlorine for treating the wool as described in this invention. This limited solubility is of particular advantage in a continuous shrinkproofing process since the desired concentration is obtained and maintained by keeping an excess of solid reagent in contact with the treating bath.

Other dichlorohydantoins and monochlorohydantoins are also useful in the practice of this invention. Although many have a higher solubility and stability in water, in general they do not possess as favorable a balance of properties as dichlorodimethylhydantoin. The following are examples of chlorohydantoins that are particularly useful in halogenating wool: 1,3-dichloro-5,5-dimethylhydantoin, 1,3-dichloro-5-methyl-5-isobutylhydantoin, N-monochloro-5,5-dimethylhydantoin, and N-monochloro-5-methyl-5-isobutylhydantoin. The N,N-dichlorohydantoins constitute a particularly useful class. The N-bromohydantoins may also be employed but, because of their limited availability and increased cost are not preferred.

This invention is directed toward the shrinkproofing of keratinous textile material, e. g., wool. Thus, the treatment of woolen apparel including socks, sweaters, jackets, coats, bathing suits, scarfs, dresses, suits and baby clothing falls under the scope of the invention. Other woolen goods, such as blankets, may also be treated. Other keratinous fibers, such as rabbit fur, characterized by the tendency to felt may be improved in like manner by the process of this invention.

In contrast to products treated by earlier halogenation methods, those from this invention are

not subject to the disadvantages of uneven treatment, tendering discoloration and harshening.

The surprising advantage of the use of strongly acid buffered solutions of the N-chlorohydantoins is that they not only effect substantial shrinkproofing but also yield an undamaged product which is soft and free from discoloration. Thus, dichlorodimethylhydantoin is a particularly effective shrinkproofing agent when applied in aqueous solutions buffered with sodium acid sulfate, phosphoric acid or similar strongly acidic materials. This is in marked contrast to the action of similar solutions buffered to an approximate pH of 4 or above which caused marked discoloring, harshening, and tendering of the product, which is only slightly shrinkproof.

The above description and examples are intended to be illustrative only. Any modification thereof or variation therefrom which conforms to the spirit of the invention is intended to be included within the scope of the claim.

What is claimed is:

Process for reducing the tendency to shrink of a woolen fibrous material which comprises immersing the same in a solution, of pH 1 to 3, of 1,3-dichloro-5,5-dimethylhydantoin, the liquid of said solution consisting of water.

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