Patented Sept. 12, 1950 2,522,338

UNITED STATES PATENT OFFICE

2,522,338

PROCESS FOR SHRINK-PROOFING WOOL AND WOOLEN FABRICS

Everett N. Angus and Joseph W. Creely, Oaklyn, and Ralph M. Fischer, Paterson, N. J.; said Angus and said Creely assignors to Eavenson & Levering Company, Camden, N. J., and said Fischer assignor to American Cyanamid Company, New York, N. Y., a corporation of Maine

No Drawing. Application February 19, 1945, Serial No. 578,792

5 Claims. (Cl. 28—75)

1

This invention relates to an improved process for the control of shrinkage, felting and fulling in woolen textile materials by the application of curable resinous materials thereto. A principal object of the invention is the treatment of wool and wool-containing fibers with resins or resin-forming materials by a method, which avoids loss of shrinkage control that may be caused by mechanical working of the fibers.

It is known that varying degrees of shrinkage control can be obtained by impregnating wool and woolen textile materials with curable thermosetting resins, followed by polymerizing or curing the resins in the wool to a water-insoluble condition. Both wool and wool-containing fabrics such as natural loose wool, yarn, threads and knitted, felted and woven cloth have been treated by this method with various types of thermosetting resins, such as urea-formaldehyde, thiourea formaldehyde and melamine formaldehyde resins. In most cases the wool is impregnated with an aquous solution, containing these or other similar resin-forming condensation products in their uncured or monomeric condition together with a curing catalyst, after which the wool is dried and heated for a short time at elevated temperatures, to cause polymerization or curing of the resin to a water-insoluble condition.

There are several practical advantages in applying this type of shrinkage control to raw wool, or to woolen textile materials in an early mechanical stage of manufacture. For example, where the treatment takes place in the finished product, much of the desired handle, flexibility, softness and resiliency may frequently be lost because of the binding of fibers together with resin. We have found, however, that a totally unexpected problem arises, when the shrinkage control methods outlined above are applied to raw or partly finished woolen textile materials, that must be subjected to mechanical working in a later manufacturing stage. It is to this problem and its solution that our present invention is particularly directed.

The nature of the problem dealt with by our invention is most clearly explained by the following example. If clean raw stock is impregnated with an aqueous solution of methylated methylol melamine, containing a curing accelerator such as diammonium phosphate, and then dried and cured by heating for a few minutes at 300° F., a satisfactory shrinkage control is obtained, when judged by the rate of increase of felt density in a miniature felting machine. A good measure of felt density was found to be the saturated wet weight of the felted pads produced in such a

2

machine. However, we found that most, if not all, of the shrinkage control is lost when the stock so treated is carried through a mechanical stage of manufacture, such as carding and spinning into yarn. A large number of tests conducted in this manner has shown us that the effect of mechanical processing, in which the permanent relative position of the fibers is changed, is to partially or completely destroy the shrinkage control that would normally be obtained, when this processing follows the curing of a thermosetting resin within the wool.

Experiments carried out with many different types of thermosetting resin-forming materials have shown that the results described above are not limited to methylated methylol melamine. On the contrary, the action of any thermosetting resin capable of reducing the shrinking, felting or fulling of wool is seriously reduced by extensive mechanical working, which results in fiber displacement. This is particularly true of the carding and spinning operations, although it is also noticeable to a somewhat lesser extent when woolen yarn, containing a cured thermosetting resin capable of imparting shrinkage control, is knitted or woven into cloth. The extent of damage or loss in shrinkage control apparently bears a direct relationship to the severity and extent of fiber displacement, which the wool receives after the thermosetting resin has been cured therein.

Although extensive mechanical working of wool containing a fully cured thermosetting resin will impair or destroy shrinkage control, we find that it has no harmful action on resin-impregnated wool in which the resin has not been fully cured. Therefore, in accordance with our present invention, we may impregnate the wool with an unpolymerized or substantially monomeric thermosetting resin solution, then subject the wool to the necessary mechanical working incident to the manufacture of yarn, cloth, etc., and then cure the monomeric resin to a polymerized or water-insoluble condition. By this method of treatment, the practical advantages resulting from impregnation of the raw or partially prepared woolen textiles with the resin solution are retained, while the loss of shrinkage control resulting from mechanical working is avoided. However, certain precautions should be observed, which will now be explained.

Since the uncured thermosetting resins are applied preferably as dilute aqueous solutions, it is necessary to dry the impregnated wool, at least to the extent necessary for the carding, spinning and other similar manufacturing steps to which

it is usually subjected. We find, however, that both the control of shrinking, felting and fulling, and the scuff or abrasion resistance of the woolen textiles, are improved considerably, if the drying of the woolen textiles is completed at relatively low temperatures prior to heating at higher temperatures to cure the resin. Therefore, the preferred practice of our invention includes the steps of impregnating the wool with an aqueous solution of a monomeric thermosetting resin capable of imparting control of shrinkage, felting and fulling thereto, then partially or completely drying the wool, then subjecting the wool to the necessary mechanical working incident to its manufacture into yarn or knitted or woven cloth in the absence of water, then further drying the woolen textiles if necessary, and finally converting the monomeric thermosetting resin to its polymerized, water-insoluble condition.

Our invention in its broader aspects is not limited by the type or types of thermosetting resins used to impart shrinkage control to the wool. It is known that shrinkage control is obtainable by impregnating the wool with 3%–30%, or more, of its dry weight of a thiourea-formaldehyde condensation product, such as dimethylol thiourea. Urea-thiourea-formaldehyde condensation products may also be used. However, considerably better shrinkage control is obtained by the use of alkylated or alcohol-reacted methylol melamines and particularly with methylated methylol melamine, and this is the preferred thermosetting resin which we employ in practicing our invention. Quantities on the order of 2.5%–15% or more of methylated methylol melamine, based on the dry weight of the wool or wool-containing textiles, have given excellent results when applied by the process of our invention.

Any suitable curing method may be employed to convert the monomeric thermosetting resin to its cured, water-insoluble condition after the mechanical working is completed. A curing catalyst such as diammonium hydrogen phosphate, either alone or in admixture with a substantially equal quantity of hexamethylene tetramine may be added to the impregnating bath in quantities of about 4%, based on the weight of the resin. Alternatively, the textiles may be treated with an acidic curing agent, during or just prior to the curing of the resin, as by subjecting them to vapors of sulphur dioxide or other acid gases followed by heating at 200°–400° F. This two-stage process, in which the curing catalyst is applied to the textiles separately from the monomeric resin solution, presents the further important advantage of improved bath stability; i. e., the resin solution used to impregnate the wool is not subject to rapid deterioration during use. The normal life of an impregnating bath, containing an added curing catalyst, is usually not longer than 24 hours, after which time a fresh bath must be prepared, whereas aqueous solutions of methylated methylol melamine and other similar thermosetting resins are stable for much longer periods of time, if no curing catalyst is added. The improved bath stability resulting from the two-stage method of operation constitutes another important advantage of our invention.

From the foregoing description it is apparent that the improved process of our invention removes several present objectionable limitations in that it allows any desired further mechanical operations to be given the woolen textiles without loss of ultimate shrinkage control. Under

present methods of treating fibers for shrinkage control, such as various chlorination procedures, treatment with alcoholic potash solutions, etc., the further mechanical action on such treated stock produces a percentage of waste which in the case of noils might be used in the manufacture of felt. Being shrink-proof, this waste would be worthless for felt manufacture. Under the improved process of our invention, such waste could be washed to remove resins or resin-forming materials and become usable without loss.

We explain this phenomenon in the following way: When wool is treated with a resin forming mixture and then dried and cured by heating, the fibers are stiffened somewhat and set so that they have the least amount of potential energy due to strains as long as they are in the same mechanical stage of manufacture. If agitated and washed they will not move and no felting or shrinkage will occur. If, however, the individual fibers acquire potential energy due to strains set up in them by mechanically processing the fibers into a fabric and the fabric is washed the fibers will tend to move to relieve the stresses set up in them. In so moving the fibers will felt and the fabric will shrink.

The following examples illustrate the invention, percentages all being by weight:

Example 1

Clean woolen yarn of less than 0.50% grease content is treated by dipping skeins into a bath of the following composition at 80° F. and soaking for 15 minutes:

25.6% methylated methylol melamine
0.96% diammonium phosphate (catalyst)
0.10% dioctyl sodium sulfosuccinate (wetting agent)
73.34% water
pH=7.0

The skeins are then centrifuged to a wet pick up of 50%. By percentage of wet pick up is meant

$$100\frac{(B-A)}{A}$$

where

A=initial dry weight of skeins
B=weight of skeins after soaking in the bath and centrifuging.

The skeins are dried best at a temperature of 100°–160° F. to a moisture content of about 10% water. The yarn may then be stored up to 2 weeks and then knitted into a finished fabric. After knitting, the finished piece may be dried at 150° F. for 45 minutes with an air velocity of 300 ft./min. They can then be cured at the same air velocity for 15 minutes at 200°–450° F. Such a fabric is much more resistant to shrinkage in washing than an untreated piece. It also has a very open appearance and soft feel not unlike an untreated piece.

Example 2

Same as Example 1 but use the following bath after standing over night:

200 g. thiourea
425 mls. formaldehyde (40% solution)
1 g. dioctyl sodium sulfosuccinate
6 g. diammonium phosphate
Water sufficient to make 1 liter of solution
pH=7.0 by addition of sodium hydroxide

Example 3

Same as Examples 1 or 2 but omit diammonium phosphate and treat with an equal weight of sulfur dioxide gas in a closed chamber in which a suitable fan is enclosed for circulating air containing the proper amount of sulfur dioxide. This sulfur dioxide treatment is postponed until after the piece is knitted and is applied just before drying and curing the resin.

This change in procedure is advantageous in several respects. The bath used in impregnating the wool with a resin-forming dispersion is stabilized by omitting the catalyst. It can be kept for a long time without spoiling. The treated wool can be kept for a longer time after impregnating and drying and before curing. Better shrinkage control is obtained using sulfur dioxide or other acid gaseous catalysts as compared with a catalyst such as diammonium phosphate which is used in the resin forming dispersion.

Example 4

Scoured raw stock of grease content preferably under 0.50% grease is treated as the yarn in any of the above examples. After drying to approximately 10% moisture content it is carded and spun into yarn and finally knitted or woven into a fabric. At this point the fabric is dried best at 150° F. for 45 minutes with an air velocity of 300 ft./min. It is cured well at 200°–450° F. for 15 minutes.

Having thus described our invention, what we claim and desire to protect by Letters Patent of the United States is:

1. A process for controlling the shrinking, felting and fulling of wool which comprises impregnating raw wool with from 2.5% to 30% of its weight of an uncured thermosetting resin selected from the group consisting of urea-formaldehyde, thiourea-formaldehyde and melamine-formaldehyde resins in the form of an aqueous solution, drying, carding and spinning the impregnated wool and finally heating the wool at a temperature within the range of 200–450° F. until the resin is converted to its cured and water-insoluble condition.

2. A process according to claim 1 wherein the wool after drying, carding and spinning is further dried to the substantial exclusion of moisture immediately prior to the final heating step.

3. A process of manufacturing a woolen fabric of reduced shrinkage upon washing which comprises impregnating raw wool with from 2.5% to 30% of its weight of an uncured thermosetting resin selected from the group consisting of urea-formaldehyde, thiourea-formaldehyde and melamine-formaldehyde resins in the form of an aqueous solution, drying, carding and spinning the wool into yarn, converting the yarn into fabric, and heating the fabric at a temperature within the range of 200–450° F. until the resin is converted to its cured and water-insoluble condition.

4. A process for controlling the shrinking, felting and fulling of wool which comprises impregnating raw wool with from 2.5% to 15% of its weight of an uncured water-soluble methylated methylol melamine in the form of an aqueous solution thereof, drying, carding and spinning the impregnated wool and finally heating the wool at a temperature within the range of 200–450° F. until the resin is converted to its cured and water-insoluble condition.

5. A process of manufacturing a woolen fabric of reduced shrinkage upon washing which comprises impregnating raw wool with from 2.5% to 15% of its weight of an uncured water-soluble methylated methylol melamine in the form of an aqueous solution thereof, drying, carding and spinning the wool into yarn, converting the yarn into fabric, and heating the fabric at a temperature within the range of 200–450° F. until the resin is converted to its cured and water-insoluble condition.

EVERETT N. ANGUS.
JOSEPH W. CREELY.
RALPH M. FISCHER.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
Re. 22,566	Johnstone	Nov. 21, 1944
1,673,797	Brown	June 19, 1928
2,050,156	Borghetty	Aug. 4, 1936
2,121,006	Bener	June 21, 1938
2,161,805	Dreyfus	June 13, 1939
2,329,622	Johnstone	Sept. 14, 1943

FOREIGN PATENTS

Number	Country	Date
437,642	Great Britain	Nov. 4, 1935

OTHER REFERENCES

"Journal of the Society of Dyers and Colourists," vol. 58, No. 12, Dec. 1942. Article by H. Phillips, pages 245–253. Copy in Scientific Library. T. P. 890. S6.

"American Dyestuff Reporter," vol. 33, Dec. 18, 1944. Article by J. Edward Lynn, pages P554–P559. Copy in Scientific Library.