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ART OF TREATING VEGETABLE FIBERS

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The present invention relates to the art of treating vegetable fibers and more particularly to a process of treating a vegetable fiber to convert the same into a fiber resembling wool and to the product of such process.

It is an object of the present invention to provide an improved process for treating an inexpensive fiber such as jute, hemp, mallow, etc., in such a manner as to convert the same into a fiber resembling, and having largely the properties of ordinary wool.

It is another object of the invention to provide an improved process by the use of which an improved product having a quality and color better than heretofore obtainable is produced.

It is a further object of the invention to provide an improved process of treating fibers whereby the quantities of reagents used for converting inexpensive fibers into wool-like fibers are materially decreased.

A still further object of the invention is to control the loss of weight of the inexpensive fiber such as jute or the like by regulating the conditions of treatment. Our invention provides a process by which the loss of weight of the vegetable fibers, i. e., the amount of non-cellulosic components left in the fiber, can be controlled by controlling the factors of the initial treatment including caustic soda solution, time, temperature, etc.

It is also within the contemplation of our invention to provide the use of an oxidizing bath having a pH value of less than about 9 for use in converting vegetable fibers to fibers resembling natural wool.

By the use of our process it is possible to produce a converted jute fiber resembling natural wool which has a light color and which requires only about 10% caustic soda solution for effecting the crinkling instead of a 17% solution as needed heretofore for crinkling vegetable fibers. It is thus possible to economize materially on the consumption of caustic soda in the crinkling bath.

Broadly stated we have discovered that we can improve the process of converting vegetable fibers into fibers of wool type and obtain an improved product by subjecting jute and similar fibers to (I) an initial oxidation and hydrolysis treatment, (II) a crinkling treatment and (III) a bleaching treatment.

We base the present invention on the fact that the yellow color obtained when jute is treated with an alkali is due to neither the cellulose nor the lignin but to another type of compound, perhaps a glucoside, which contains within it, or has

mixed with it an oxygen type of dye color. In fact, there is, apparently, more than one such coloring material. These dyes are nearly colorless when in neutral or acid solution but in the presence of alkali, become colored (yellows and browns) and at the same time tend to fix themselves to the fiber. Accordingly, we remove these materials, leaving, as nearly as possible, only cellulose and lignin, and we accomplish this removal under conditions which are approximately neutral (i. e. a pH value of about 5 to about 9) but in no case strongly alkaline. Of course after we have washed out these coloring materials, we can use a strong caustic soda without permanently discoloring the fiber.

We accomplish removal of the coloring materials by oxidizing and hydrolyzing them in a neutral or slightly acid solution and washing out the resultant products. In practice we find it best to carry out this process with a pH of 5 to approximately 8.

A specific example is given for purposes of illustration but it is to be understood that none of the specific substances, temperatures, concentrations or the like are to be taken as limitations upon the invention.

Other objects and advantages of the invention will become apparent from the following description of a typical run of this process. Jute fibers are preferably, but not necessarily, cut into lengths as desired (say about 5") and are opened up by means of a "card" or "picker" in order that the solutions employed in the treatment of the fibers can easily and thoroughly penetrate to the individual fibers.

After the fibers are cut and opened they are preferably washed to remove adhering foreign matter, such as dust, dirt and water soluble materials associated therewith. This washing is, however, not essential. In carrying out this operation, we prefer to add a small amount of sulfonated oil, such as "Monopole" or "Turkey red" oil, to the water with which the washing is effected. Any excess of sulfonated oil is removed by rinsing with fresh water, either hot or cold, before the fiber is given the initial oxidation treatment. The washing operation, in addition to removing foreign matter, "wets out" the fibers in such a manner that a uniform and thorough action can be obtained during subsequent treatments with chemical reagents.

Step I

When the jute fibers have been prepared as described hereinabove they are ready for the

initial oxidation treatment. A nearly neutral solution which simultaneously gives some hydrolysis is used. In practice, we prefer to effect this treatment in a solution which has been buffered so that the pH has a value of about 6 to 8. A typical bath for this step may be made up as follows:

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| Potassium permanganate ----- | 8 pounds |
| 10 Tri-sodium phosphate ----- | 14 pounds |
| Phosphoric acid (85%) ----- | 3 pounds |
| Water ----- | 3500 gallons |

Such an oxidizing bath is capable of treating about 1000 pounds of jute. A temperature of about 25° C. (about room temperature) is suitable for this step. At this temperature a period of about 15 to 20 minutes in the oxidizing bath is required.

During this oxidizing treatment some manganese dioxide is precipitated on the jute fibers. This precipitate can be removed in any appropriate manner. For instance, we may first press out the excess of liquid and then treat the jute fibers with a solution containing a small percentage of acid sodium sulfite (NaHSO_3) at a temperature of approximately 60° C. A few minutes treatment is sufficient at the aforementioned temperature. In case lower temperatures are used a longer time of treatment is necessary. The jute fibers are preferably washed free of the acid sodium sulfite preparatory to the subsequent operations.

A single bath in which the "wetting out", oxidation, and hydrolysis occur simultaneously may be used instead of the above series. Such a bath may consist of

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| Monopole oil ----- | 30 pounds |
| 40 Tri-sodium phosphate ---- | 20 pounds |
| Phosphoric acid (85%) --- | 9.3 pounds |
| Hydrogen peroxide (100 volume) ----- | 10 pounds or more |
| Water ----- | 3500 gallons |

In this case temperatures up to 50° C. may be used and 20 minutes is then a sufficient time. There is no precipitated manganese dioxide, of course, in this bath and therefore no acid sodium sulfite is required.

Step II

The fibers are now ready for their alkali treatment. This may be carried out in two stages. In the first stage the fibers are given a preliminary treatment in a bath of very dilute caustic soda (about 0.1%) at a temperature of about 60° C. to about 80° C. At this temperature a treatment for about an hour has been found sufficient to remove the desired amount of non-cellulosic components such as lignin. By increasing the caustic strength larger amounts of non-cellulosic materials may be removed and by controlling caustic content, temperature, time, etc., the loss of weight may be varied from about 5% to as high as 30% or more. The quality and character of the finished product vary with the loss of weight as will be readily understood. In practice, we prefer to operate limiting the loss to 5 to 10% in the weight of the fiber. Before proceeding with the second stage of alkali treatment the excess of dilute caustic solution is pressed from the fibers, e. g., by passing them through rolls. The pressed fibers are then treated in a second caustic soda

bath which is of such a strength, that, together with the water present in the fibers, the resulting solution will preferably have approximately a 10% caustic soda content. With a solution of this character a few minutes, say ten minutes, in the bath is ample time of contact.

In commercial practice the strength of the second caustic soda bath is determined by the amount of crinkle desired in the finished fiber. Solutions having a caustic soda content lower than 10% can be used in the second stage of the alkaline treatments although we prefer to use a solution having a caustic soda content of about 10% or somewhat higher. As a general rule, the stronger the caustic soda bath or the lower the temperature thereof, the more crinkle is produced in the finished fibers.

The same result may be accomplished, but at a sacrifice of caustic soda, by using only a single bath of the strength of the second, or crinkling, bath. A longer time in the strong alkali bath may then be needed.

After the fibers have been subjected to the strong alkali treatment they are washed thoroughly to free them from alkali. This is preferably accomplished by a series of washings. The last washing bath may desirably contain a small amount of an acid such as phosphoric acid or an acid salt of phosphoric acid such as monosodium phosphate (NaH_2PO_4) or other acid salts of similar character. A washing bath of this character insures the complete neutralization of alkali contained in or adsorbed on the fibers.

Step III

The washed, crinkled fibers are then ready for bleaching and softening. The said fibers have a brown color at this stage of the process but this brown color is of such a character as to be easily removable by the succeeding bleaching operation.

The bleaching may be accomplished with a variety of solutions, for instance, solutions containing a permanganate or hydrogen peroxide or perborates, or similar bleaching agents. In practice, we have found that excellent results are obtained when the bleaching solution has a pH value of less than about 8. Under some conditions, it has been found best to bleach in a series of solutions using the same or different bleaching agents in each solution. Bleaching in stages usually gives the whitest product, although a single bleach will produce a fiber which has a color even lighter than that of natural wool. The double bleaching, moreover, not only gives a whiter product but a more uniform product.

We prefer to use a double bleaching treatment. Such a treatment may be as follows: The washed crinkled fibers are immersed in a solution containing about 0.1% by weight of hydrogen peroxide (H_2O_2) which has been buffered by means of sodium phosphate and phosphoric acid to give a pH value of about 8. An amount of sulfonated oil equal to about 1% of the weight of the fiber is added to the bath to insure wetting of the fibers. The sulfonated oil, although not essential, facilitates the bleaching and is an aid to uniformity of the product. The fibers are maintained in the bath about 16 hours, the exact time depending on the temperature and shade desired in the finished product. The excess of bleaching solution is removed by passing the fibers through rolls or the like or by washing or both.

The second stage of the bleaching operation in-

volves the treatment of the fibers with a very dilute solution (about 0.02%) of a permanganate, say potassium permanganate, buffered with sodium phosphate and phosphoric acid to give a pH value of approximately 5.5. In a typical run, the amount of potassium permanganate was about 1.7% of the weight of the fiber treated. The fibers remain in the permanganate solution until bleaching is accomplished, approximately 1/2 hour at room temperature. A small amount of manganese dioxide is precipitated on the fibers in the aforesaid bleaching operation and is removed by soaking the fibers in a solution of acid sodium sulfite at a temperature of about 50° C. for a period of about an hour. The weight of acid sodium sulfite employed is about three times the weight of permanganate taken and is sufficient to dissolve any manganese dioxide which may have been precipitated on the fibers.

In the event that the apparatus, chemicals or water used contain iron the fibers are likely to absorb some iron. In cases of this sort, the iron can conveniently be removed by treating the fibers with an oxalic acid solution containing about 0.01% of oxalic acid. Of course, other acids having properties similar in this respect to oxalic acid can also be employed, for example, hydrochloric acid. If treated with oxalic acid or the like, the fibers must be washed free of this acid preparatory to the subsequent operations.

The washed, bleached, and crinkled fibers now resemble wool fibers. To give them the desired softness, they are treated with any of the well known softening materials commonly used for this purpose in the textile industry, for instance, materials such as soaps, sulfonated oils, glycerine and the like may be employed. After softening and drying, the fibers are ready for use as an article of commerce. These fibers can be carded and spun in the same way as natural wool fibers can be carded and spun. The converted fibers can be used alone or mixed with natural wool for the production of a great variety of articles and materials, including textile fabrics, rugs, blankets and the like as one skilled in the art will readily understand.

In the present specification and claims when the term "oxidizing" is used, it is to be understood to mean a treatment with an agent giving oxygen directly as, for example, potassium permanganate, hydrogen peroxide, sodium perborate, etc. The term "oxidizing" as used herein does not refer to treatment with such agents as, for example, sodium hypochlorite, which in the alkaline condition is both an oxidizing agent and a chlorinating agent for jute and similar fibers and in acid solutions is primarily a chlorinating agent.

We claim:

1. The process of treating vegetable fibers to convert the same into fibers resembling natural wool which comprises subjecting the vegetable fibers to oxidizing treatment at a pH between 5 and 8, then crinkling the thus treated fibers by subjecting them to the action of a caustic soda solution of approximately 10% strength.

2. The process of treating vegetable fibers to convert the same into fibers resembling natural wool which comprises subjecting the vegetable fibers to an oxidizing bath having a pH of about 5 to approximately 8, then crinkling the thus treated fibers by subjecting them to the action of a caustic soda solution of approximately 10% strength and finally bleaching said fibers.

3. The process of treating vegetable fibers to

convert the same into fibers resembling natural wool which comprises subjecting the vegetable fibers to an initial oxidizing and hydrolyzing treatment at a pH of about 5 to about approximately 8, then crinkling the thus treated fibers by subjecting them to the action of a caustic soda solution of approximately 10% strength and finally bleaching said fibers.

4. The process of treating vegetable fibers to convert the same into fibers resembling natural wool which comprises treating the fibers with an aqueous bath containing a small amount of sulfonated oil, subjecting the vegetable fibers to an oxidizing and hydrolyzing treatment at a pH of about 5 to approximately 8, then crinkling the thus treated fibers by subjecting them to the action of a caustic soda solution of approximately 10% strength and finally bleaching said fibers.

5. The process of treating vegetable fibers to convert the same into fibers resembling natural wool which comprises washing the fibers, wetting the washed fibers with an aqueous vehicle containing a sulfonated oil, subjecting said thus treated fibers to an oxidation treatment in an approximately neutral solution, washing said fibers with water, crinkling said fibers by contacting them with caustic soda solution of about 10% strength, washing said fibers free of caustic, and bleaching said crinkled fibers by subjecting them to the action of a bleaching solution having a pH value of less than about 8.

6. The process of treating fibers of the jute type which comprises cutting said fibers into short lengths, opening up said fibers to permit penetration by the solutions, washing said cut fibers, rinsing said washed fibers with water, immersing about 1000 parts of said fibers in a bath containing about 30 parts of sulfonated oil, 20 parts of trisodium phosphate, 9.3 parts phosphoric acid (85%) about 10 parts hydrogen peroxide, and about 30,000 parts of water, washing the fibers with water to free them of the aforesaid bath, treating the washed fibers with a caustic soda solution of about 10% strength to give the desired amount of crinkling, washing the crinkled fibers free of alkali, and bleaching the said fibers with a bleaching solution having a pH value of less than about 8.

7. The process of treating fibers of the jute type which comprises cutting said fibers into short lengths, opening up said fibers to permit penetration by solutions, washing said cut fibers, rinsing said washed fibers with water, immersing about 1,000 parts of said fibers in a bath containing about 30 parts of sulfonated oil, 20 parts of trisodium phosphate, 9.3 parts of phosphoric acid (85%), about 10 parts of hydrogen peroxide and about 30,000 parts of water, washing the fibers with water to free them of the aforesaid bath, treating the washed fibers with a caustic soda solution of about 10% strength to give the desired amount of crinkling, washing the alkali from the crinkled fibers, and bleaching the said fibers with a bleaching solution having a pH value of less than about 8, washing the fibers free of the bleaching agent and immersing the fibers in a softening fluid to give the crinkled converted fibers the desired softness.

8. The process of treating fibers of the jute type which comprises cutting said fibers into short lengths, opening up said fibers to permit penetration of the solutions, immersing about 1,000 parts of said fibers in a bath containing about 30 parts of sulfonated oil, 20 parts of trisodium phosphate, 9.3 parts of phosphoric acid

(85%), about 10 parts of hydrogen peroxide and about 30,000 parts of water, washing the fibers with water to free them of the aforesaid bath, treating the washed fibers with a caustic soda solution of about 10% strength to produce the desired amount of crinkling, controlling the aforesaid treatment including the caustic soda content,

the temperature and duration to vary the loss in weight of the fibers from about 5% to about 30%, washing the crinkled fibers free of alkali, and bleaching the said fibers with a bleaching solution having a pH value of less than about 8.

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