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BLEACHING FIBERS OF VEGETABLE ORIGIN WITH FORMAMIDINE SULFINATE AND ALKALINE PEROXIDE

James H. Young, Niagara Falls, N. Y., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware

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This invention relates to a method of bleaching fibers of vegetable origin, particularly cotton goods.

Cotton goods have long been scoured with a solution of a strong alkali and then bleached with an alkaline peroxide solution. Both treatments are usually carried out at an elevated temperature either with the goods immersed in the treating solution or merely saturated or dampened therewith.

Methods have been devised for carrying out such scouring and bleaching treatments continuously, but whether practiced batchwise or continuously each treatment has usually required a treating time of the order of 1 hour or longer, necessitating the use of costly kiers or J-boxes for storing the goods the required time in contact with the hot treating solution. The prior methods degrade the cotton significantly, and such degradation, and a somewhat impermanent final brightness, are normally expected of such methods. Bleaching to a high permanent brightness without degradation of the goods but with good absorbency and complete mote removal would be highly desirable. The present invention relates to a practical method whereby these objectives may be largely achieved.

It is an object of the invention to provide an improved method for bleaching fibers of vegetable origin. A further object is to provide a method for bleaching cotton goods whereby high brightnesses of improved permanency are readily obtained with excellent absorbencies and mote removal and with relatively little degradation of the cotton. Another object is to provide a method whereby the above desirable results can be readily obtained in a shorter time than is possible by methods now used. Still further objects will be apparent from the following description.

The objects of the invention are accomplished by extracting the vegetable fibers with an aqueous solution of an alkali metal hydroxide containing dissolved therein a formamidine sulfinate, then bleaching the fibers with an aqueous alkaline peroxide solution.

Any water-soluble formamidine sulfinate can be used for the present purpose but sodium formamidine sulfinate is preferred. Other weaker reducing agents such as the sulfites and sulfides are ineffective for the present purpose.

In experimenting with formamidine sulfinic acid, it was discovered that aqueous sodium hydroxide solutions thereof, in which the acid is of course present as sodium formamidine sulfinate, reduce and solubilize dyes which are similar to the color bodies naturally occurring in vegetable fibers such as cotton and wood pulp. It was further found that a substantial part of such color bodies can be solubilized and extracted from such fibers by means of the above solutions with the result that the extracted fibers are more readily bleached with an alkaline peroxide solution.

The present extraction treatment also appears to swell the cellulose fibers somewhat and softens and opens up the mote structure so that subsequent bleaching with an alkaline peroxide solution is facilitated.

The alkali component of the extracting solution should be an alkali metal hydroxide. Other alkaline materials such as soda ash are too weakly alkaline. The concentration of the alkali metal hydroxide, such as potassium or sodium hydroxide, in the solution may be varied considerably, e. g. from about 0.5 to 10%, or even up to

20% by weight, but most generally will range from about 2 to 6%. The use of sodium hydroxide is preferred for cost reasons.

The concentration of the reducing agent can also be varied considerably, but for cost reasons, concentrations greater than about 5% by weight of the solution will not generally be used. The preferred concentrations range from about 0.1 to 1.0%, although a worthwhile effect can be obtained at a concentration as low as about 0.01%.

The extraction treatment can be carried out at ordinary temperatures but higher temperatures are preferred since they accelerate the solubilizing action of the alkaline formamidine sulfinate solution on the color bodies. As a general guide, an overnight steep of the fibers in the extracting solution at about 25° C. results in effective solubilization of the color bodies, while treatment times of 1 to 30 minutes at 90 to 100° C. or 20 seconds to 2 minutes at 110 to 130° C. are effective. After suitable solubilization of the color bodies has resulted, the solution containing the solubilized bodies should be separated from the fibers. Preferably, the fibers are then thoroughly rinsed before being bleached with alkaline peroxide.

The invention is illustrated by the following examples. Concentrations of reagents represent percentages by weight, based on the weight of the treating solution, unless indicated to be otherwise. Brightness (percent reflectances) were determined using a Hunter multipurpose reflectometer. The absorbency values reported represent the time in seconds for the absorption on the taut surface of the fabric (held in embroidery hoops) of one drop of water delivered from an eye dropper held 2 inches above the surface.

Example 1

Strips of unbleached cotton muslin were wet-out for 2 to 3 minutes at 70 to 100° C. with a 0.2% solution of "Duponol" ME (a sodium alkyl sulfate wetting agent) which also contained 0.3% sodium pyrophosphate. The wet-out strips were rinsed, then extracted by treatment for 1 to 8 minutes at the boiling temperature of the solution with one of the following solutions:

(*a*) A 3% aqueous solution of sodium hydroxide.

(*b*) A 3% aqueous solution of sodium hydroxide containing the sodium salt of formamidine sulfinic acid, $HN{=}C(NH_2)SO_2H$, in an amount equivalent to 0.2% of the acid.

The strips were then thoroughly rinsed with water and finally immersed for 5 minutes in a boiling aqueous bleach solution containing 0.2% hydrogen peroxide (H_2O_2) and 1.5% of a 42° Bé. sodium silicate solution (having the approximate composition: 10% Na_2O, 25% SiO_2 and 65% H_2O). In some instances, the hydrogen peroxide concentration of the bleach solution was increased to 0.4%. The results are tabulated below:

BLEACHED WITH 0.2% H_2O_2 SOLUTION

Extraction Treatment		Percent Reflectance	
With Solution (*a*) or (*b*)	Min.	After Extraction	After Bleach
(*a*)	1	58.0	77.8
(*b*)	1	70.2	81.4
(*a*)	2	63.5	79.6
(*b*)	2	70.8	82.7
(*a*)	4	64.5	80.2
(*b*)	4	75.2	85.4
(*a*)	8	65.2	81.2
(*b*)	8	75.3	86.1

BLEACHED WITH 0.4% H_2O_2 SOLUTION

(*b*)	1		85.6
(*b*)	2		86.6
(*b*)	4		88.6
(*b*)	8		89.5

In those samples bleached with the 0.2% peroxide solution, the samples of the (*a*) series were distinctly poorer than the corresponding samples of the (*b*) series both with respect to final brightness and the completeness of mote removal. Mote removal was substantially complete in all samples of the (*b*) series which were bleached at the higher peroxide concentrations.

Example 2

Strips of unbleached cotton muslin were wet-out, rinsed then extracted with solution (*a*) or (*b*) as described in Example 1, except that the extraction time in each instance was 5 minutes. The strips were then rinsed, dampened with an amount of a bleach liquor corresponding to a weight ratio of liquor to cloth of about 1:1, bleached under pressure at 116° C. for 30 to 120 seconds and finally washed and ironed. The bleach liquor contained 0.4% hydrogen peroxide and 2.8% sodium silicate (42° Bé. solution). Results were:

Extraction with Solution (*a*) or (*b*)	Time for Peroxide Bleach (seconds)	Final, Percent Reflectance
(*a*)	30	78.4
(*b*)	30	84.6
(*a*)	60	82.3
(*b*)	60	86.6
(*a*)	90	83.2
(*b*)	90	87.5
(*a*)	120	85.4
(*b*)	120	88.1

Mote removal was incomplete in all of the above bleached samples of the (*a*) series while the corresponding samples of the (*b*) series were mote-free.

Example 3

Strips of unbleached cotton muslin were wet-out and rinsed as described in Example 1, extracted 3 to 5 minutes at the boiling temperature with an aqueous solution of 3% sodium hydroxide and sodium formamidine sulfinate equal to 0.2% of the acid, rinsed, then bleached at the boiling temperature for 3 to 30 minutes with a solution containing 1.5% sodium silicate (42° Bé. solution) and and 0.4% hydrogen peroxide. Results were:

Extraction Time (min.)	Bleaching Time (min.)	Percent Reflectance	Absorbency (seconds)	Cuam Fluidity (Rhes)	Percent Brightness Loss in Aging
3	3	87.0	0.5	1.7	--------
5	5	89.7	0.5	2.0	--------
5	15	90.3	0.5	------	--------
5	30	90.8	0.5	------	3.0
[1] 60	[1] 60	[1] 86.5	[1] 2.5	[1] 4.9	[1] 5.6

[1] These are average values for 23 cotton fabrics bleached in a continuous commercial system in which fabric was scoured with a caustic soda solution, washed, then bleached with alkaline peroxide solution. Both the scouring and the bleaching treatments involved saturating the goods with the treating solution, heating the staurated goods with live steam to about the boiling point, then storing the heated goods in an insulated J-box for 1 hour.

It is seen from the above data that the samples treated by the present method were distinctly superior to the commercially bleached goods in every respect, and that these superior results were obtained in much shorter times.

Cuam fluidity (rhes) is well recognized as a measure of the degree of cellulose degradation, the higher the Cuam fluidity the greater being the degradation. The "percent brightness loss in aging" represents the loss in brightness (percent reflectance) resulting when a sample in exposed for 1 hour to saturated steam at 15 p. s. i. g.

Following the present extraction treatment the goods can be bleached by any desired method employing an alkaline peroxide bleach solution. The concentration of hydrogen peroxide (H_2O_2), or equivalent peroxygen compound such as sodium peroxide or sodium perborate,

in such solutions can be varied considerably depending upon the results desired but generally will range from about 0.05 to 1.5% or somewhat higher. Hydrogen peroxide concentrations of at least about 0.3% are preferred when complete removal of motes is desired. The solution also will usually contain from about 0.5 to 3% or more of sodium silicate (42° Bé. solution or equivalent commercial silicate), but other commonly used alkaline agents, such as sodium hydroxide, the alkaline phosphates and borax, or mixtures theerof, can also be used, either alone or in combination with the silicate. Stabilizers such as soluble magnesium compounds and the pyrophosphates can also be added.

Bleaching can be effected at ordinary temperature but higher temperatures, e. g. 80 to 100° C. (or even up to 130° C. or higher), are preferred. However, because bleaching and mote removal are facilitated by the present extraction treatment, the bleaching time required at any given temperature will generally be substantially shorter than that required when the extraction treatment is omitted or is replaced by the usual caustic scouring treatment. This reduction in bleaching time is important since it frequently will make unnecessary the use of costly storage facilities such as kiers and J-boxes.

The present method is applicable to wood pulps and other fibers of vegetable origin besides cotton. Thus, chemical wood pulps such as the kraft and sulfite pulps can be extracted with the present alkali metal hydroxide solution of a formamidine sulfinate, then bleached with alkaline peroxide with resultant advantages similar to those obtained with cotton.

When bleaching cotton fabrics, it is generally preferred to wet-out the fabric with a solution of a surface active wetting agent prior to the extraction treatment, since such a wet-out treatment improves the absorbency of the bleached fabric. Anionic, cationic and nonionic wetting agents can be used for this purpose. Examples are the usual soaps and the synthetic surface active wetting agents such as the alkyl aryl sulfonates, the fatty alcohol sulfates, quaternary ammonium salts such as cetyl pyridinium chloride, fatty alcohol amine condensates, alkoxylated aryl polyether alcohols and polyethylene oxide condensates. The wet-out solution most preferably will also contain a water-soluble phosphate, e. g. a molecularly dehydrated phosphate such as sodium pyrophosphate, sodium tripolyphosphate or sodium hexametaphosphate. The wet-out treatment is preferably carried out at an elevated temperature, e. g. 50 to 100° C., with immersion times ranging from 1 to 30 minutes.

I claim:

1. In a method of bleaching vegetable fibers of the group consisting of cotton, kraft wood pulp and sulfite wood pulp with an alkaline peroxide solution, the improvement comprising extracting naturally occurring color bodies from said fibers with an aqueous alkali metal hydroxide solution of a formamidine sulfinate prior to bleaching said fibers with the alkaline peroxide solution.

2. A method of bleaching vegetable fibers of the group consisting of cotton, kraft wood pulp and sulfite wood pulp comprising extracting naturally occurring color bodies from said fibers with an aqueous alkali metal hydroxide solution of a formamidine sulfinate and then bleaching said fibers with an alkaline solution of hydrogen peroxide.

3. The method of claim 2 wherein both the extraction and the bleaching treatments are carried out at a temperature in the range 80 to 130° C.

4. The method of claim 2 employing sodium formamidine sulfinate.

5. A method of bleaching cotton fabric comprising extracting naturally occurring color bodies from said fabric with an aqueous alkali metal hydroxide solution of a formamidine sulfinate and then bleaching the fabric with an aqueous alkaline solution of hydrogen peroxide.

6. The method of claim 5 wherein both the extraction

and the bleaching treatments are carried out at a temperature in the range of 80 to 130° C.

7. The method of claim 5 employing sodium formamidine sulfinate.

8. The method of claim 5 wherein the fabric is wet-out with a solution of a surface active wetting agent prior to extraction of color bodies from said fabric with the alkali metal hydroxide solution of a formamidine sulfinate.

9. The method of bleaching cotton fabric comprising (*a*) wetting-out said fabric with a solution of a surface active wetting agent, (*b*) rinsing the fabric, (*c*) extracting naturally occurring color bodies from the fabric with an aqueous sodium hydroxide solution of sodium formamidine sulfinate, (*d*) rinsing the fabric, then (*e*) bleaching the fabric with an aqueous alkaline solution of hydrogen peroxide.

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