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PROCESS OF BLEACHING AND DESIZING COTTON GRAY GOODS WITH ORGANIC SOLVENT DEGREASING AND DEWAXING AND PEROXYGEN BLEACHING

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This invention relates to a process for the bleaching of cellulosic goods.

It is known that goods containing cotton may be bleached by a continuous process in which the cotton goods must first be desized by passing through an enzyme bath, are subsequently padded with aqueous caustic soda solution and steamed, and are finally impregnated with an aqueous alkaline solution of a peroxygen compound such as hydrogen peroxide, and steamed. This process calls for retention of the goods after padding in a suitable device such as a J-box for a considerable period and calls for retention for a further period in a suitable device of the goods after treatment with the aqueous alkali solution of the peroxygen compound. Such procedures are time consuming and particularly, when, as is often the case, the goods are in rope form creasing of the goods occurs.

In my copending application Serial No. 402,132, filed January 4, 1954, now U. S. Patent No. 2,790,699, there is described a multi-stage process for the preparation of cotton goods for dyeing, printing or other finishing processes which comprises the steps of continuously and rapidly passing the goods, preferably in open width, through a bath of a hydrocarbon or a chlorinated hydrocarbon solvent where they are degreased and dewaxed and then passing the goods through a bath of an aqueous alkaline solution of a peroxygen compound where they are desized and bleached. Since the contact time of the goods with the bath liquors is short in each case the process is convenient for running continuously at open width and for combination with continuous dyeing operations.

In this process the desizing operation is carried out in alkaline solution, preferably at a pH of 11 to 12 and sodium peroxide, which gives particularly satisfactory results, is used in aqueous solution in its unneutralised state. The concentration of the peroxygen compound in the solution is necessarily dependent upon whether the cloth is to be treated by immersion or whether it is to be impregnated and then steamed. When the former method is used and sodium peroxide is the peroxygen compound a concentration of a little as 3 grams per litre corresponding to approximately 3.1 grams per litre of sodium hydroxide gives very good results. The steaming process of course requires a stronger solution and it is found that in order to obtain a satisfactory product the concentration of peroxide should be in this case at least 7 grams per litre, corresponding to approximately 7.2 grams per litre of sodium hydroxide for a material composed entirely of cotton, while it is preferred to use 10 to 15 grams per litre of peroxide, corresponding to approximately 10.3 to 15.4 grams per litre of sodium hydroxide. If solutions of other per-compounds are to be used an equivalent concentration calculated on the active oxygen present should be employed, the pH of the solution and alkalinity being adjusted, if necessary by addition of the appropriate amount of alkali.

The bleach effected by the second stage of this process is adequate when the cloth is subsequently to be dyed, but it is not equivalent to a full bleach. A fully bleached material is necessary, for example, for production of a pure white finish and may also be required when the cloth is to be printed with a white background. A further treatment of the cloth may thus be necessary in order to achieve a full white.

According to the present invention an improved three-stage process for the bleaching of cellulosic goods comprises (*i*) continuously and rapidly passing the the goods, preferably in open width, through a bath of a hydrocarbon or a chlorinated hydrocarbon solvent where they are degreased and dewaxed, then (*ii*) passing them through a bath of an aqueous alkaline solution of a peroxygen compound at a pH of 11 to 12 with an alkalinity corresponding to at least 7.2 grams per litre of sodium hydroxide and subsequently steaming them to promote desizing and partial bleaching and finally (*iii*) passing them through a second bath of an aqueous alkaline solution of a peroxygen compound and again steaming to develop a full bleach, this second bath of alkaline peroxygen compound being such that its alkalinity is less than that corresponding to 7.2 grams per litre of sodium hydroxide and the average pH value of the solution while in contact with the goods is at least 9.5 but less than 11.

The invention also includes a modification of the process wherein either the second stage, which produces desizing and partial bleaching, or the third stage, which develops the full bleach, or both the second and third stages, are carried out by immersing the goods in the bath at elevated temperature for a prolonged period, instead of by passing them rapidly through the bath and subsequently steaming, the baths used for such prolonged immersion treatments having, for the second stage, an alkalinity corresponding to at least 3.1 grams per litre of sodium hydroxide, and for the third stage, an alkalinity less than that corresponding to 3.1 grams per litre of sodium hydroxide.

The steaming procedure is preferred since the volume of liquid in use is considerably reduced. Also this procedure is better adapted to continuous bleaching of cotton goods since the preliminary dewaxing and desizing process can be carried out in a continuous manner and the goods can then merely be saturated with the bleaching liquor, the time of contact being not more than a few seconds, and then steamed and held for a suitable period in a retention device. In contradistinction the immersion technique results in some hold-up of the goods since the latter are soaked in the bath for a considerable period of time.

The peroxygen compound is suitably aqueous sodium peroxide solution but in its unneutralised state sodium peroxide is to alkaline and partial neutralisation with acid or an acid salt (which on reaction with the peroxide gives a salt which has substantially no detrimental effect on the goods) is necessary to bring the sodium hydroxide content of the solution below the said amounts. When the steaming procedure is used the sodium hydroxide content is preferably in the range 1 to 5 grams per litre and when the immersion procedure is used the sodium hydroxide content is preferably in the range 0.2 to 1.5 grams per litre. A peroxide solution of suitable alkalinity for use when the steaming procedure is used can be prepared by adding an acid such as sulphuric acid to an aqueous solution of sodium peroxide to such an extent that the caustic alkalinity of the latter is approximately 60% neutralised but this figure is not to be taken as critical. When sodium peroxide which has been partially neutralised is employed the actual weight of peroxide relative to the dry weight of goods is suitably of the order of 1% to 2% by weight.

It is also possible to use solutions of other peroxygen compounds such as sodium perborate, or sodium percarbonate and hydrogen peroxide, the alkalinity being then adjusted to the desired value if necessary by addition of alkali.

In the present invention in order to effect the full bleach of the cloth the time of immersion or steaming of the cloth will depend on the quantity of bleaching agent, say, the peroxide, present per litre of the solution and the type of material under treatment. Generally, however, a rather long period of treatment is required and when using the immersion treatment a period of 2 to 4 hours at a temperature of, for instance, 60° C. to 90° C., gives useful results. When using the preferred steaming treatment 15 to 30 minutes' exposure to steam is adequate. Alternatively the goods may be steamed for only a few minutes and then kept hot, say at a temperature of 90° C. to 100° C. in a J-box or similar retention device so that the total time of steaming and retention of the hot goods is 15 to 30 minutes. Subsequently the goods are washed with water and dried. In the whole process of degreasing and dewaxing, desizing and partial bleaching, followed by the final full bleaching treatment only one retention device need be employed.

Since the cloth is in contact with the peroxide solution for fairly long periods of time the pH of the solution decreases with time and it must be understood that it is the average pH value of the solution while in contact with the goods using the immersion or steaming techniques which should be at least 9.5 but less than 11. When the peroxide solution is in contact with the cloth during the steaming process and when using a retention device the pH of the solution may be determined by squeezing the solution from the cloth from time to time and testing the same. It is quite possible that the pH of the original solution as made-up may be 11 or over but as aforesaid the average pH will be essentially between 9.5 to 11. Preferably the average pH is in the range 10 to 10.8 and particularly good results are obtained when the average pH is in the range 10.5 to 10.7.

Preferably an alkali metal silicate, such as sodium silicate is present in the bleaching solution since this substance prevents unduly rapid decomposition of the peroxygen compound. The quantity of silicate added to the solution should be sufficient to slow down the decomposition of the per-compound at the temperatures in use, an amount equivalent to one third to an equal weight of the peroxygen compound is usually adequate.

Preferably the bleaching solution also contains a small amount of wetting agent of neutral or anionic character. The choice of wetting agent is not of vital importance but it is preferable to employ a wetting agent which has also a detergent action.

In one form of the process the cloth is first passed at open width through one or more enclosed vessels containing a chlorinated hydrocarbon solvent such as trichloroethylene or perchloroethylene maintained at or near to its boiling point. Where there are several vessels the solvent is allowed to flow from one to the other in the opposite direction to the direction of travel of the cloth, and the rate at which the latter passes through is adjusted according to the contact time desired. After being dewaxed the cloth is freed from solvent; this may be carried out by any known means, for example, by drying with hot air or by steaming or by passage through hot water whereby the solvent is flashed off and can be recovered and returned to the dewaxing unit. Excess water is then removed from the cloth conveniently by means of mangles, and it is passed directly through a desizing bath containing an aqueous solution of unneutralised sodium peroxide which contains also sodium silicate and a wetting agent. From this the impregnated material passes through a chamber where it is steamed for a very short period of time, for example, 1 to 2 minutes. The cloth is then rinsed in water and passed through a further

bath containing a partially neutralised solution of sodium peroxide. Preferably this solution contains from 0.75% to 1.5% of partially neutralised peroxide and the degree of expression is so arranged that the cloth retains approximately 1½ times its own weight of liquor. Subsequently the cloth passes through a steaming chamber and then through a retention device such as a J-box of sufficient dimensions to retain the material for the desired period at open width. It is finally rinsed with water and is then in a fully bleached absorbent condition.

The process of the invention is advantageous in that cotton goods may be continuously bleached at open width with practically no trouble due to creasing to give a fully bleached material of excellent absorbency and in contradistinction to some continuous bleaching processes successfully removes adventitious oil stains and renders possible the use of waxy lubricants in warp sizing.

The following example illustrates, but does not limit the invention:

Example

The procedure described hereunder was carried out entirely at open width on specimens of the following cotton cloths: pillow cotton weighing 3 ozs. per sq. yd., a basecloth for leathercloth weighing 3 ozs. per sq. yd., cambric weighing 2½ ozs. per sq. yd., a typewriter cloth weighing 3 ozs. per sq. yd., and a basecloth for leathercloth weighing 7 ozs. per sq. yd. These materials in the grey loom state were first passed through two vessels containing trichloroethylene maintained at its boiling point, the total immersion time for each sample of material being 40 seconds. The cloths were then passed through boiling water for 20 seconds in order to flash off the solvent, after which they were padded in an aqueous solution containing 15 grams per litre of unneutralised sodium peroxide, 5 grams per litre of sodium silicate and 0.5 gram per litre of a wetting agent consisting of a condensation product of octylated cresol with ethylene oxide. The nip was adjusted so that the cloth retained 1½ times its weight of liquor. The impregnated cloth was then steamed for 2 minutes, rinsed in hot water and mangled. The average reflectance of the cloths in light of wavelength 4,700 angstrom units was, at this stage, 80%.

The next step was to impregnate the cloth with an aqueous solution containing 15 grams per litre of sodium peroxide, 13 grams per litre of 96% sulphuric acid and 10 grams per litre of sodium silicate, the nip being adjusted so that the cloth retained 1½ times its weight of liquor. The impregnated cloth was then steamed for 20 minutes, the average pH of the solution during the time of treatment being between 9.5 and 11. The steamed cloth was given two rinses with hot water and one rinse with cold water. After drying it was found that all the various materials were bleached to a full white, the average percent reflectance determined in the presence of light of wavelength 4,700 angstrom units being 91%. The materials were of low fluidity, a 0.5% solution of the fully bleached 7 oz. cloth in standard cuprammonium solution having a fluidity of 4.3. The cloths were also free from starch and motes and were extremely absorbent.

By way of comparison the 7 oz. cloth was bleached by a known continuous process which comprised the steps of enzyme desizing, padding with caustic soda and steaming in a retention device followed by impregnating with a solution of hydrogen peroxide and sodium hydroxide and steaming in a retention device, the total time of treatment including enzyme desizing being about 1½ hours. The finished sample was less absorbent than those treated by the process of the present invention and a 0.5% solution of the treated cloth in standard cuprammonium solution had a fluidity of 5.3.

What I claim is:

1. In a multi-stage process for the bleaching and desizing of substantially gray goods containing cotton which comprises (1) degreasing and dewaxing said goods

by continuously and rapidly passing the same through a bath of solvent selected from the group consisting of trichloroethylene and perchloroethylene at approximately the boiling point thereof, the total necessary immersion time in the bath being not more than three minutes, (2) removing solvent from the goods by contacting them with an aqueous medium held at an elevated temperature sufficient to flash-off said solvent, the improvement which comprises the combination of (3) then passing the goods through a desizing and partial bleaching bath of an aqueous alkaline solution of a desizing peroxygen compound, said solution having a pH of 11 to 12 with an alkalinity corresponding to at least 7.2 grams per liter of sodium hydroxide and subsequently subjecting the goods to the action of steam for a period requiring not more than three minutes to desize and partially bleach the goods, (4) thereafter passing said steamed goods through a second bath of an aqueous alkaline liquor of a peroxygen bleaching compound, said liquor having an alkalinity less than that corresponding to 7.2 grams per liter of sodium hydroxide and the average pH value of the liquor while in contact with said goods being at least 9.5 but less than 11, and (5) then developing a full bleach of said liquor-containing goods by steaming the same, the total time involved in developing the full bleach being a period requiring not more than one half hour.

2. The process of claim 1 wherein at least one of said aqueous alkaline baths employed in steps (3) and (4) contains an alkali metal silicate as a stabilizer.

3. The process of claim 1 wherein said substantially gray goods are treated in open width.

4. The process of claim 1 wherein the pH of the liquor of step (4) is in the range 10.5 to 10.7.

5. The process of claim 1 wherein the average pH of said aqueous alkaline liquor of a peroxygen bleaching compound in step (4) is in the range from 10 to 10.8.

6. The process of claim 1 wherein said aqueous alkaline solution of a peroxygen compound in step (3) has an alkalinity corresponding to from 10 to 15 grams per liter of sodium hydroxide and said aqueous alkaline liquor of a peroxygen bleaching compound in step (4) has an alkalinity corresponding to from 1 to 5 grams per liter of sodium hydroxide.

7. The process of claim 1 wherein said full bleach is developed by steaming said goods and then retaining said steamed goods at substantially the temperature of steam in an enclosed zone for said required period.

8. In a continuous multi-stage process for the bleaching and desizing of substantially gray goods containing cotton in open width which comprises (1) degreasing and dewaxing said goods by continuously and rapidly passing the same through a bath of solvent selected from the group consisting of trichloroethylene and perchloroethylene at approximately the boiling point thereof, the total necessary immersion time in the bath being not more than three minutes, (2) passing the goods through an aqueous medium held at an elevated temperature sufficient to flash-off said solvent, the improvement which comprises the combination of (3) then passing said goods through a desizing bath containing an aqueous solution of unneutralized sodium peroxide and sodium silicate as a stabilizer, said solution having a pH of 11 to 12 and an alkalinity corresponding to at least 7.2 grams per liter of sodium hydroxide, subjecting the goods to the action of steam for one to two minutes, washing the goods, (4) then passing them through a bath of an aqueous alkaline liquor containing 0.75% to 1.5% of sodium peroxide partially neutralized with a mineral acid to an extent that the alkalinity of the liquor corresponds to a content of 1 to 5 grams of sodium hydroxide per liter, and sodium silicate as a stabilizer, said liquor having a pH from 9.5 to 11, withdrawing the goods from said bath and expressing the liquor therefrom to an extent that the goods retain approximately one and one-half times their own weight of the liquor, and (5) thereafter subjecting said liquor-containing goods to the action of steam for a period of time from fifteen to thirty minutes, and finally washing the goods.

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