

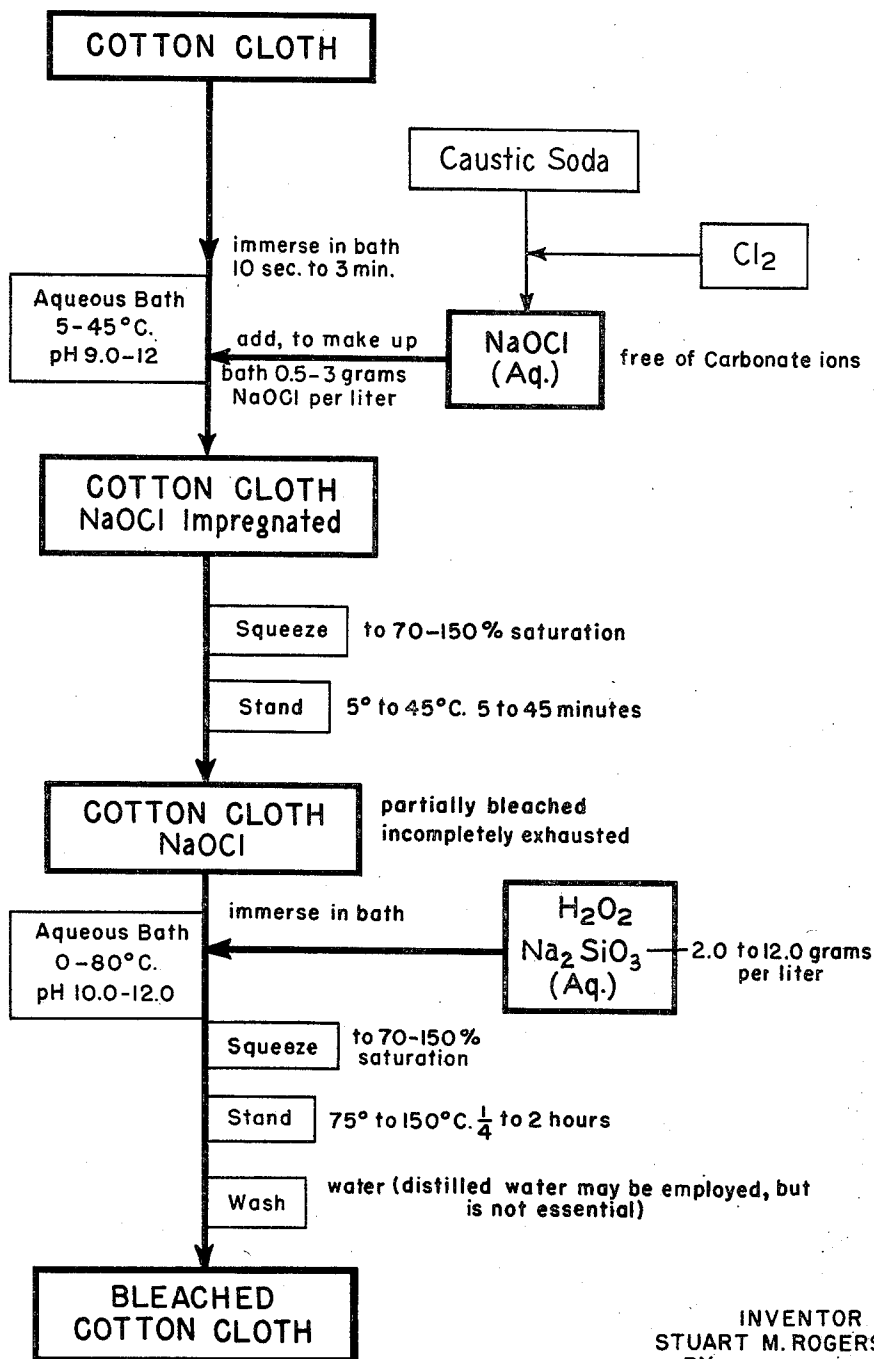
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 $\text{NaOCl-H}_2\text{O}_2$ COTTON CLOTH BLEACHING

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NaOCl—H₂O₂ COTTON CLOTH BLEACHING

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This invention relates to bleaching of cotton textiles and more particularly refers to the production of bleached cotton cloth of superior properties by a new and improved process of bleaching.

Hydrogen peroxide for bleaching in the textile industry was employed for at least 35 years partially supplanting competitive bleaches, such as hypochlorites. The commercial continuous hydrogen peroxide system for bleaching cotton textiles adopted by the industry involves saturating the cotton cloth with caustic soda, steaming, washing, saturating with hydrogen peroxide plus additives, steaming and washing. The additive usually added to the hydrogen peroxide is sodium silicate whose function is to control the release of oxygen.

Bleaching of cotton cloth is not a matter simply of improving the whiteness of the cloth, but also involves other features which are as important as whiteness. Cloth can be bleached to a high degree of whiteness by drastic treatment, but good quality cloth should have low "fluidity," a measure of degradation of the cloth. Absorbency is another important property of bleached cloth. Absorbency determines the suitability of a fabric for particular uses, as it is also important in fabrics which are to be dyed since the dyeability can be effected by the absorbency. Another feature in the bleaching of cloth is the silicate content of the finished cloth. The presence of silicate in cloth is objectionable. Unfortunately, in conventional hydrogen peroxide bleaching operations, there is a permanent absorption of silicate from the peroxide vat. As a matter of fact, in hydrogen peroxide bleaching operations, highly purified water, such as distilled water, must be employed as wash water or else the salts normally carried by tap water or hard water will be picked up by the cloth. Silicate, salts and other inorganic materials increase the ash content of the cloth and have a detrimental effect on its properties. While the use of distilled water will reduce the ash content of the bleached cloth, distilled water in quantities required for washing adds considerably to the overall cost of bleaching cloth.

One object of the present invention is to provide a process of bleaching to produce cotton cloth of improved quality having high absorbency and brightness and low ash and fluidity.

Another object of the invention is to provide a bleaching process employing hydrogen peroxide containing sodium silicate as an additive wherein the sodium silicate is not absorbed by the cotton cloth.

A further object is to provide a bleaching process for cotton cloth involving treatment with hydrogen peroxide wherein washing the bleached cloth with distilled water is unnecessary and the cloth may be washed with ordinary tap water without the detrimental effect of hardness in the wash water on cloth absorbency.

A still further object of the present invention is to provide an efficient process for effecting bleaching of cotton cloth with a reduced amount of hydrogen perox-

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ide. Other objects and advantages will be apparent from the following description.

In accordance with the present invention, bleaching may be accomplished to produce cotton cloth of high absorbency, high whiteness and low fluidity and low ash content by treating cotton cloth in sequential manner under regulated conditions involving immersing cotton cloth in an aqueous solution containing 0.5–3, preferably 1–2, grams per liter NaOCl and having a pH value of 9.0–12.0, preferably 10.0–11.5, said solution being substantially free of carbonate ions, maintained at a temperature between 5° C. and 45° C., preferably within the range of 15–30° C., for a period of time of less than 3 minutes, preferably about 10–60 seconds, squeezing the cloth impregnated with the sodium hypochlorite solution to 70%–150%, preferably 80–120%, saturation and retaining the thus squeezed cloth at a temperature between 5–45° C., preferably within the range of 15–30° C., for a period of about 10–30 minutes, preferably about 10–20 minutes, thereafter without washing and without other treatment immersing the cloth in a hydrogen peroxide solution containing sodium silicate and having a pH of 10.0–12.0, preferably 10.0–11.0, at a temperature within the range of 0–80° C., preferably within the range of 10–40° C., squeezing the cloth impregnated with the hydrogen peroxide solution to 70–150%, preferably 80–120%, saturation and retaining the thus squeezed cloth at a temperature within the range of 75–150° C., preferably within the range of 90–120° C., for a period of ¼–2 hours, preferably ¾–1½ hours, and washing the thus treated cloth with water.

The accompanying drawing diagrammatically illustrates the process of the present invention.

Preliminary to bleaching cotton cloth, the cloth is prepared in the usual manner by singeing, desizing and caustic treatment. It may be desirable to wash the fabric with hot water to remove impurities which adhere to the cloth prior to desizing. Desizing may be accomplished by treatment with dilute sulfuric acid and with a solution of enzymes. The desizing operation may be followed by water washing and then by caustic treatment to effect further removal of impurities from the cloth. The caustic treated cloth may then be washed and subjected to souring, which is the action of a very dilute solution of a mineral acid on the fabrics to neutralize residual caustic remaining in the fabrics and to dissolve foreign matter. In some instances, either the caustic treatment or souring, or both, may be dispensed with in preparing cloth for bleaching depending upon the nature of the impurities. The operations for preparing the cotton goods for bleaching is preferably carried out in continuous manner in conventional equipment employed in the industry for this purpose, such as saturators, washers, J-boxes and squeezers.

The cloth prepared for bleaching is first subjected to treatment with hypochlorite under regulated conditions which will permit production of a bleached cloth of improved properties. Into a saturator is introduced an aqueous solution of sodium hypochlorite having a preferable concentration of 1–2 grams per liter or of sufficient concentration so that the cloth leaving the saturator after squeezing will contain .1–.2% NaOCl basis weight of cloth. A concentration below 0.5 gram sodium hypochlorite per liter would be ineffective and a concentration above 3 grams sodium hypochlorite per liter would deleteriously affect the properties of the cloth. The hypochlorite bath should be at a low temperature, below 45° C., preferably about room temperature, and the cloth should not remain immersed in the hypochlorite bath for more than 3 minutes, preferably less than one

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minute, in order to avoid deterioration of the cloth. Another factor of great importance is the avoidance of carbonate ion in the sodium hypochlorite bath. We discovered that the presence of carbonate ion in the hypochlorite solution resulted in a cloth of appreciably lower absorbency and also was instrumental in causing the cloth to absorb silicate and other inorganic salts which depreciate the properties of the cloth. Consequently the use of soda ash or any carbonate salt as is common practice in treating with hypochlorite should be avoided in the practice of this invention. Sodium hypochlorite for use in the saturator of the present invention may be prepared by chlorinating caustic soda. The best results may be obtained by maintaining the pH of the sodium hypochlorite solution between 9.0-12.0, preferably 10.0-11.5.

The cloth is removed from the saturator and squeezed to a dampened condition, about 100% liquor saturation, i.e. a cloth-liquor ratio of about 1:1. The dampened cloth is then transferred to any suitable container, such as a J-box, wherein it is held for a short period of time of the order of 10-20 minutes. A retention time appreciably less than 10 minutes would not effectively treat the cloth and a period of time much in excess of 30 minutes would deteriorate the cloth. The temperature during retention should be below 45° C., preferably substantially atmospheric temperature.

It is important that cloth treated with hypochlorite in the manner as described above be directly, without washing or other treatment, immersed in a hydrogen peroxide solution. Retention of the residual hypochlorite in the cloth which is carried over into the hydrogen peroxide bath appears to impart desirable properties to the finished cloth, and its neutralization by the peroxide liberates oxygen which is beneficial in bleaching. The hydrogen peroxide bath may contain 1.0-15.0, preferably 2.0-5.0, grams per liter hydrogen peroxide and 2.0-12.0, preferably 4.0-6.0, grams per liter sodium silicate. The pH of the hydrogen peroxide solution may be held within the range of 10-12, preferably 10.0-11.0, by the addition of sodium hydroxide. The concentration of hydrogen peroxide, the amount of sodium silicate and the temperature of the hydrogen peroxide solution may be varied over a considerable range since the sensitivity of the cloth is much lower with hydrogen peroxide than with sodium hypochlorite. The cloth need be immersed in the hydrogen peroxide bath only for sufficient length of time to saturate it, usually a matter of minutes. The wet cloth is squeezed to approximately 100% liquor saturation and then passed to a J-box wherein it is heated to a temperature of approximately 212° F. by direct or indirect contact with steam for a sufficient length of time to effect the desired bleaching, usually of the order of between ½ to 1½ hours. The thus treated cloth is washed with water.

The whiteness of bleached cloth can be determined on a General Electric reflectometer with a blue filter. The results obtained are expressed as percentage reflectance as compared with reflectance of magnesium carbonate. A whiteness of 86% or higher is considered good for medium weight fabric.

The determination of the fluidity of cellulose dissolved in cuprammonium hydroxide or cupriethylenediamine solution is a very sensitive and accurate method for measuring the effect of bleaching upon cotton. It is particularly effective in measuring the modification or degradation of the cellulose fiber due to chemical processing and is largely independent of the fabric construction. This is a very useful test since the modification or degradation can be used to predict the effect of chemical treatments on the tensile strength of fabrics and their wearing qualities. Fluidity values are measured in rhes (reciprocal poises). Values above 6 indicate degradation of the cloth fibers. Values below 4 indicate superior fiber strength.

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The absorbency of the fabric is tested by fastening ½" strips, without stretching, between prongs projecting from the edges of two disks, spaced about 6" apart by means of a supporting rod passing through the center of each disk. There are enough prongs so that several samples may be tested at one time. With the samples in place, the whole assembly is inserted in a vertical glass cylinder containing 1-1½" of distilled water. A stop watch is started simultaneously with this operation and at the end of five minutes the height of the water absorption is measured by means of a transparent scale attached to the outside of the cylinder by flexible bands. The zero point is adjusted to the water level. Each ¼" rise of water in the fabric denotes one point of absorbency. The test is conducted at 70° F. with a relative humidity of 65% on conditioned fabric. Absorbencies are taken on both warp and filling of the cloth and averaged. An absorbency of 25-30 is acceptable and an absorbency above 30 is considered excellent.

The ash content is determined by charring samples of the cotton in a platinum crucible using a Meker burner, then muffling to constant weight. Although an ash content of 0.20 is considered satisfactory, the lower the value of ash, the better the cloth is considered.

Silicon content of bleached cloth, as determined by quantitative analysis, varies from 150 to 500 p.p.m. Cloth having a silicon content below 150 p.p.m. is considered superior.

As will be appreciated, the difficulty in bleaching lies not with attaining a high value with respect to one of the properties of bleached cloth, but with respect to all of the properties. Specifically, a brightness of 86 may be readily attained for medium weight fabric. However, it is important that the absorbency also be high and that the fluidity be low and the ash content be low. Cotton cloth bleached in accordance with the present invention imparts to the cloth not only high brightness, but also higher absorbency, lower fluidity and lower ash, thereby producing a superior bleached cotton fabric.

The following are two comparative examples, the first representing conventional commercial operation for bleaching of cotton cloth with hydrogen peroxide in a textile bleachery and the second representing an operation in accordance with the present invention carried out on commercial plant scale.

Example 1

Cotton cloth having the following specifications: 80 x 80 thread count, 4.00 yards per pound weight, was prepared for bleaching by singeing, desizing and caustic treating in a continuous manner as follows: Singeing, desizing, caustic J-box treatment and washing were carried out in the conventional manner. After squeezing to about 100% liquor saturation, it was passed through a peroxide saturator containing liquor of the following composition: 6.0 g.p.l. H₂O₂, 5.1 g.p.l. silicate and 1.26 g.p.l. NaOH. The pH was 10.7 and the temperature about 28° C. Retention time of the cloth in the liquor was about 30 seconds. Thereafter the cloth was squeezed to about 110% liquor saturation and passed in a J-box where it was heated by direct steam and retained for one hour. Finally the cloth was washed and dried.

Example 2

Cotton cloth having the specifications recited in Example 1 was treated in the same manner described therein involving singeing, desizing and caustic treatment to prepare the cotton cloth for bleaching.

The thus prepared cloth was then passed through a bath of hypochlorite liquor made by chlorinating caustic soda, said bath comprising a vessel containing 100 gallons hypochlorite liquor at room temperature (25° C.). The hypochlorite liquor contains 1.5 grams per liter NaOCl and has a pH of 11.3. The time of immersion of the cloth in the hypochlorite bath is about 30 seconds. The

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cloth, saturated with hypochlorite solution, is squeezed to approximately 115% liquor saturation and retained in boxes at room temperature for about 20 minutes. The cloth was further squeezed to about 90% liquor saturation. Thereafter, the hypochlorite-treated cloth, without washing or other treatment, was passed through a peroxide liquor saturator, said liquor having the following composition: 4.0 g.p.l. H_2O_2 , 3.4 g.p.l. silicate and 0.84 g.p.l. NaOH. The cloth retention time in the peroxide liquor saturator at room temperature was about 30 seconds. The saturated cloth was squeezed to about 100% liquor saturation and then passed through a J-box wherein the cloth was retained for one hour and wherein it was heated to a temperature of 100° C. by introduction of steam in direct contact with the cloth. After bleaching, the cloth was washed with tap water and passed through the drying rolls.

In the table below is a comparison of the properties of the cloth treated in a conventional manner, Example 1, as compared to the properties of the cloth when bleached in accordance with the present invention, Example 2:

	Example 1	Example 2
Cloth Properties:		
Absorbency.....	28.9	40.1
Fluidity.....	5.45	3.05
Brightness.....	86.8	88.5
Ash, Percent.....	0.17	0.13
Spectrographic Analysis:		
P.p.m. in Cloth—		
Cu.....	0.5	0.6
Al.....	6.1	3.6
Fe.....	6.1	5.1
Mn.....	1.0	0.9
Pb.....	1.±	nd<1
V.....	<1.8	nd<1.8
Cr.....	1.2.±	nd<1
Ni.....	1 nd<1.8	nd<1.8
Est. Ti.....	4-5	<1
Mg.....	30	20
Est. Ca.....	150-300	100-200
Quantitative Analysis:		
Si.....	341	49

¹ nd=not detected.

From the above data, it will be evident that the cloth bleached in accordance with the present invention is markedly superior to conventional hydrogen peroxide bleaching. Specifically, the absorbency was increased from 28.9 to 40.1; the fluidity was improved from 5.45 to 3.05; the brightness was higher, a value of 88.5 compared to 86.8; and the ash was lower, being reduced from 0.17 to 0.13. These results were attained with concentrations of peroxide, silicate and caustic in the bleach bath of $\frac{2}{3}$ of normal.

The spectrographic analysis was generally more favorable for the cloth bleached in accordance with the present invention and of special interest is the marked decrease in silicate.

Although certain preferred embodiments of the invention have been disclosed for purpose of illustration, it

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will be evident that various changes and modifications may be made therein without departing from the scope and spirit of the invention.

I claim:

1. A process of bleaching to produce cotton cloth of high absorbency, high whiteness, low fluidity and low ash content which comprises (a) treating cotton cloth in sequential manner by immersing the cotton cloth for a period of time from about 10 seconds to less than 3 minutes in an aqueous solution of NaOCl prepared solely by chlorinating caustic soda to avoid the introduction of carbonate ions, at a temperature between 5-45° C. containing 0.5-3 grams per liter NaOCl and having a pH value of 9.0-12.0, (b) squeezing the cloth impregnated with the sodium hypochlorite solution to 70-150% saturation and maintaining the thus squeezed cloth at a temperature between 5-45° C. for a period of about 5-45 minutes, (c) thereafter directly immersing the cloth in a hydrogen peroxide solution containing 2.0-12.0 grams sodium silicate per liter as an additive and having a pH of 10.0-12.0 at a temperature within the range of 0-80° C., (d) squeezing the cloth impregnated with the hydrogen peroxide solution to 70-150% saturation and maintaining the thus squeezed cloth at a temperature within the range of 75-150° C. for a period of $\frac{1}{4}$ -2 hours, (e) and washing the thus treated cloth with water.

2. A process of bleaching to produce cotton cloth of high absorbency, high whiteness, low fluidity and low ash content which comprises (a) treating cotton cloth in sequential manner by immersing the cotton cloth for a period of time in the range of 10-60 seconds in an aqueous solution of NaOCl prepared solely by chlorinating caustic soda to avoid the introduction of carbonate ions, at a temperature between 15-30° C. containing 0.5-3 grams per liter NaOCl and having a pH value of 10.0-11.5, (b) squeezing the cloth impregnated with the sodium hypochlorite solution to 80-120% saturation and maintaining the thus squeezed cloth at a temperature between 15-30° C. for a period of about 10-15 minutes, (c) thereafter directly immersing the cloth in a hydrogen peroxide solution containing 4.0-6.0 grams sodium silicate per liter as an additive and having a pH of 10.0-11.0 at a temperature within the range of 10-40° C., (d) squeezing the cloth impregnated with the hydrogen peroxide solution to 80-120% saturation and maintaining the thus squeezed cloth at a temperature within the range of 90-120° C. for a period of $\frac{3}{4}$ -1½ hours, (e) and washing the thus treated cloth with water.

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