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3,528,115

**BLEACHING FABRICS WITH PERACETIC ACID
FORMED IN SITU THEREON**

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No Drawing. Filed Dec. 22, 1967, Ser. No. 692,691

Int. Cl. D06l 3/02

U.S. Cl. 8—111

7 Claims

ABSTRACT OF THE DISCLOSURE

Textile fabrics which are amenable to bleaching with peracetic acid are saturated with an alkaline solution of hydrogen peroxide, the saturated fabric is contacted with acetic anhydride vapor, whereby peracetic acid is formed in situ on the fabric, and the fabric is then bleached with the peracetic acid.

The bleaching of textile fabrics with alkaline hydrogen peroxide solutions is widely practiced. Most commonly, relatively concentrated and highly alkaline peroxide solutions are employed at about atmospheric steam temperatures in order to achieve bleaching in short times. Under such rather severe conditions, efficient use of the peroxide is not realized and some of the more sensitive fabrics are damaged. Peracetic bleach solutions are known to effectively bleach most fabrics even under much milder bleaching conditions. However, peracetic acid bleach solutions heretofore proposed have been solutions formulated prior to their use. Thus, such peracetic acid bleach solutions have been made from more concentrated purchased peracetic acid solutions, or they can be made as proposed in Reichert et al. U.S. Pat. 2,377,038 by the addition of acetic anhydride to an alkaline solution of hydrogen peroxide. Performed solutions of peracetic acid have noxious odors and are potentially hazardous to prepare and handle because of their explosive tendencies.

The present invention is based upon the discovery of a very practical way of forming peracetic acid solutions in situ upon the fabric that is to be bleached, thus overcoming the hazards and inconvenience of preparing and handling performed peracetic acid solutions.

SUMMARY OF THE INVENTION

Textile fabrics which are amenable to bleaching with peracetic acid are saturated with an alkaline hydrogen peroxide bleaching solution, the saturated fabric is contacted with acetic anhydride vapor to effect absorption of said vapor on the fabric and reaction of the absorbed acetic anhydride with the hydrogen peroxide thereby producing peracetic acid in situ on the fabric, and the fabric is bleached with the peracetic acid so produced.

Alternatively, the fabric can be partially bleached with the alkaline hydrogen peroxide solution with which it is initially saturated, following which, peracetic acid is formed in situ on the fabric as indicated above and bleaching is completed with the peracetic acid so formed.

**DETAILED DESCRIPTION AND
PREFERRED EMBODIMENTS**

The alkaline hydrogen peroxide solution with which the fabric to be bleached is saturated and which serves as the precursor of the peracetic acid solution formed in situ in accordance with the invention, will generally con-

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tain from around 0.1 to 5%, preferably 0.4 to 2%, H₂O₂ by weight and an alkali which will be present in an amount sufficient to render the solution distinctly alkaline, e.g., have a pH of at least 9.0. Preferably, the solution will have a pH of at least 10.5 e.g., 10.5 to 13, and will contain sufficient alkali so that when it is reacted on the fabric with the acetic anhydride vapor to produce the peracetic acid, the alkalinity of the solution will be reduced to a pH not lower than 4.5, most preferably not lower than pH 5.5.

The alkaline hydrogen peroxide solution most generally will be formulated using commercial aqueous hydrogen peroxide products as the source of the hydrogen peroxide, although other products which yield hydrogen peroxide in solution can be used, examples of such products being sodium peroxide, sodium perborate, sodium percarbonate, urea peroxide, sodium phosphate perhydrates, and the like. The alkalinity of the peroxide solutions will generally be derived mainly from caustic soda or caustic potash although other alkaline-reacting materials such as sodium silicate, sodium carbonate, borax, and the like, may also be used. Most generally, the alkaline peroxide solution will contain caustic soda in combination with buffer materials such as sodium silicate and tetrasodium pyrophosphate. Other materials commonly employed in peroxide bleach solutions may also be present, examples of which are heavy metal ion sequestering agents, such as the polycarboxyamine compounds, e.g., ethylenediaminetetraacetic acid, diethylenetriaminepentaacetic acid, nitrilotriacetic and the hydroxy-alkylethylenediamine triacetic acids, and the sodium salts of such acids. Wetting agents may also be present in the solution, examples of which are the conventional anionic and non-ionic detergents such as the common soluble soaps and the sodium and potassium alkyl sulfates and alkyl benzene sulfonates, the alkoxyated aryl polyether alcohols, and the like.

The fabric to be bleached will generally be dampened, impregnated, or saturated with an amount of the alkaline hydrogen peroxide solution equal to about 50 to 150%, preferably, 80 to 120%, of the dry weight of the fabric. Following saturation of the fabric with such an amount of the solution, the saturated fabric can then be contacted directly with acetic anhydride vapors. Alternatively, the saturated fabric can be partially bleached with the alkaline peroxide solution by storing it for a sufficient time at ordinary or elevated temperatures to effect a partial bleaching of the fabric. At that point, the partially bleached fabric, which will contain a substantial amount of unused peroxide, can be contacted with acetic anhydride vapor to effect absorption of the anhydride on the fabric. The reaction of the absorbed anhydride with the residual peroxide will form peracetic acid in situ on the partially bleached fabric. Thereafter, bleaching is permitted to continue with the peracetic acid as the bleaching agent. The partial bleaching with the peroxide solution can, of course, be omitted and the fabric initially saturated with the peroxide solution can be immediately contacted with the acetic anhydride vapor and then bleached with the peracetic acid formed in situ as indicated above.

The contacting of the fabric saturated with the alkaline peroxide solution with the acetic anhydride vapor can be carried out at various temperatures for periods of time sufficient to provide absorption on the fabric of at least

about 0.2% acetic anhydride, based upon the dry weight of the fabric. Any desirable temperature can be employed providing it is not sufficiently high to adversely affect the fabric. Temperatures from room temperature up to about the boiling temperature of water are generally satisfactory. Absorption of from about 0.2 to 3%, preferably 0.8 to 1.5%, acetic anhydride based upon the fabric weight will generally be adequate and give good performance.

Reaction of the alkaline peroxide solution upon the fabric with the absorbed acetic anhydride vapor to produce peracetic acid proceeds quite readily even at room temperatures and, of course, the reaction proceeds more rapidly the higher the temperature. Temperatures from around room temperature, or even lower, up to around 300° F. may be employed, although in general, temperatures of around 100 to 200° F. will most generally be preferred. Following absorption of the anhydride, bleaching with the peracetic acid formed may be effected at any desired temperature, e.g., from room temperature up to 300° F., the lower temperatures requiring longer times than the higher temperatures. In most instances, temperatures from room temperature to about 212° F. will be used and those from about 150 to 212° F. will be preferred.

The method of the invention is illustrated by the following examples in which all composition percentages are by weight.

In the experiments of Example 1, the results of which are reported in Table 1, 6" x 16" fabric samples of heavy all-cotton twill (1 yd./lb.) were bleached. All of the fabric samples had been freshly scoured by saturating them with a scouring solution and steaming the saturated fabric at 212° F. for 2 minutes. The scouring solution contained 10% caustic soda, 0.5% tetrasodium pyrophosphate decahydrate and 0.2% of a commercial sodium dodecylated oxydibenzene disulfonate surfactant.

In the experiments of Example 2, the results of which are reported in Table 2, 6" x 16" fabric samples of a nylon/cotton twill (15.85, nylon:cotton) having a weight of 1 yd./lb., which had been freshly scoured as indicated above, were bleached.

The bleaching procedure for the experiments of both Example 1 and 2 was as follows: the freshly scoured fabric sample for a given experiment was saturated with an equal weight of the alkaline hydrogen peroxide bleach solution, following which the fabric was either bleached directly (in the controls) by steaming the fabric at a desired temperature or by allowing it to stand at a temperature lower than the normal steaming temperature, or by first suspending the saturated fabric for a short period of time in an agitated acetic anhydride vapor bath maintained over liquid acetic anhydride at 212° F., to effect absorption of from about 1 to 1.5% acetic anhydride on the fabric. Bleaching was then carried out as indicated above. In all cases, the bleached fabric was rinsed well with 500 ml. of warm water and the pH of the total rinse water was determined as well as its hydrogen peroxide and peracetic acid contents.

The compositions of the various hydrogen peroxide

bleach solutions employed in the experiments of Examples 1 and 2 were as follows:

Solution A

- 1.4% H₂O₂
- 2.0% sodium silicate
- 0.05% epsom salt
- 0.1% sodium salt of diethylenetriaminepentaacetic acid
- 0.1% sodium dodecylated oxydibenzene disulfonate
- 0.5% TSPP

Solution B

- 1.4% H₂O₂
- 1.0% caustic soda
- 2.0% sodium silicate
- 0.05% epsom salt

Solution C

- 1.4% H₂O₂
- 1.5% caustic soda

Solution D

- 0.7% H₂O₂
- 1.0% caustic soda
- 2.0% sodium silicate
- 0.5% TSPP
- 0.05% epsom salt

Solution E

- 0.7% H₂O₂
- 2.0% Na₂CO₃
- 0.5% TSPP

Solution F

- 0.5% H₂O₂
- 1.0% caustic soda
- 0.5% TSPP
- 0.05% epsom salt

Solution G

- 0.5% H₂O₂
- 4.0% TSPP
- 0.05% epsom salt
- 0.1% sodium dodecylated oxydibenzene disulfonate

In the above compositions, the abbreviation TSPP stands for tetrasodium pyrophosphate decahydrate, and the designation "sodium silicate" stands for a commercial 40° Bé. sodium silicate solution whose approximate composition was 10% Na₂O, 25% SiO₂ and 65% H₂O.

In Tables 1 and 2, which report the results of the bleaching experiments, the initial pH reported is that of the starting hydrogen peroxide bleach solution while the final pH is that of the rinse water following bleaching. The whiteness values reported are percent whitenesses representing the percent light reflected from the samples as measured using a Hunter Reflectometer with a blue filter for which magnesium oxide gives a reflectance of 100%. Under the heading "MOTES" in Table 2, a "+" designation means the bleached fabric was free or virtually free of motes; and a "-" designation means that motes were barely visible; and a "—" designation means that motes were distinctly visible. In the column heading "SECONDS IN AC₂O VAPOR," "Ac₂O" designates acetic anhydride.

EXAMPLE 1

[TABLE I.—Bleaching heavy Cotton Twill]

Experiment	Bleach solution	Seconds in Ac ₂ O vapor	Steaming time at 212° C., min.	pH		Percent residual A. O.		Percent whiteness	Percent whiteness gain
				Initial	Final	As H ₂ O ₂	As peracid		
a (control)	A		2	10.1	9.8	90		80.2	
b (control)	A	10	2	10.1	5.4	83	5.0	83.1	2.9
c (control)	A plus 0.5% NaOH		2	10.9	10.4	81		81.0	
d	do	10	2	10.9	6.0	65	6.0	84.4	3.4
e (control)	A plus 0.9% NaOH		2	11.3	10.8	73		83.8	
f	do	10	2	11.3	8.3	52	2.0	87.0	3.2
g	do	10	1	11.3	7.7	51	5.0	83.8	
h	do	10	1+1	11.3	6.0	37	10.0	86.8	3.0

¹The fabric saturated with the bleach solution was steamed for 1 minute, then suspended in the acetic anhydride vapor bath then steamed for an additional minute.

EXAMPLE 2

[Table II.—Bleaching Nylon/Cotton Twill]

Experiment	Bleach solution	Seconds in Ac ₂ O vapor	pH		Bleach conditions	Percent residual A. O.		Motes	Percent whiteness	Percent whiteness gain
			Initial	Final		As H ₂ O ₂	As peracid			
a (control)	B		11.4	10.9	212° F./2 minutes	64		±	81.4	
b	B	10	11.4	9.75	do	47	0.6	+	85.3	3.9
c	B	10	11.4	5.2	212° F./2 minutes, then Ac ₂ O treatment, then 150° F./15 min.	18		1 +	82.8	1.4
d (control)	B		11.4		170° F./2 minutes	76		—	70.5	
e	B	10	11.4	8.0	do	41		5 +	79.0	8.5
f (control)	C		11.9	11.2	212° F./2 minutes	53		±	76.0	
g	C	10	11.9	6.0	do	16		5 ±	78.8	2.3
h (control)	D		12.4	10.8	do	6		—	80.5	
i	D	10	12.4	5.9	212° F./15 minutes	1		2 +	83.7	3.2
j (control)	E		10.6	10.6	212° F./2 minutes	4		—	72.8	
k	E	5	10.6	6.9	do	3.7	3.4	—	79.8	7.0
l	E	10	10.6	6.4	do	7	9.5	—	82.0	9.2

The experiments of Examples 1 and 2 involving the use of peracetic acid formed in situ illustrate application of the method of the invention to the bleaching of cotton and cotton/nylon fabrics. The method of the invention is also applicable in similar manner to the bleaching of other textile fabrics which are amenable to bleaching with peracetic acid. Such other textile fabrics include: cotton-polyester blends, rayon, and cellulose acetates and triacetates.

I claim:

1. In a method for bleaching a textile fabric which is amenable to bleaching with an aqueous peracetic acid solution, the improvement comprising forming said peracetic acid solution in situ on said fabric by saturating said fabric with an aqueous alkaline solution of hydrogen peroxide and thereafter contacting the fabric saturated with said hydrogen peroxide solution with acetic anhydride vapor whereby acetic anhydride is absorbed by the fabric and the absorbed acetic anhydride reacts with said alkaline hydrogen peroxide solution to produce peracetic acid in situ.

2. The method of claim 1 wherein the alkaline hydrogen peroxide solution contains 0.1 to 5% H₂O₂ by weight and has a pH of at least 9.0.

3. The method of claim 1 wherein the alkaline hydrogen peroxide solution contains 0.1 to 5% H₂O₂ by weight and has a pH of 10.5 to 13, and wherein the fabric saturated with said hydrogen peroxide solution is contacted with acetic anhydride vapor to effect absorption on the fabric of from 0.2 to 3% of said acetic anhydride, based upon the dry weight of the fabric.

4. The method of claim 3 wherein bleaching with the peracetic acid formed in situ is effected at a temperature from room temperature to about 300° F.

5. The method of claim 3 wherein bleaching with the peracetic acid formed in situ is effected at a temperature from room temperature to 212° F.

6. The method of claim 3 wherein bleaching with the peracetic acid formed in situ is effected at a temperature from 150 to 212° F.

7. The method of claim 3 wherein the fabric saturated with the alkaline hydrogen peroxide solution is partially bleached with said hydrogen peroxide solution with only partial utilization of the hydrogen peroxide, and the fabric is thereafter contacted with acetic anhydride vapor to effect absorption of acetic anhydride thereon and the formation of peracetic acid in situ by reaction of the acetic anhydride with the residual hydrogen peroxide present on the fabric, and bleaching of the fabric is completed with said in situ formed peracetic acid.

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U.S. Cl. X.R.

8—101; 252—95, 97, 103, 186