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[54] N-BENZOYLSACCHARIN AS PEROXYGEN
BLEACH ACTIVATOR

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QP 1 B4.

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252/186
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[56] References Cited
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3,177,148 4/1965 Bright et al. 252/99

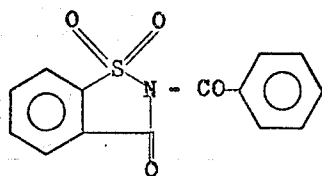
[57] ABSTRACT

N-Benzoylsaccharin is a bleach activator of high activating strength and good storage stability in bleaching compositions comprising hydrogen peroxide-releasing compounds, such as alkali metal perborates and percarbonates.

6 Claims, No Drawings

N-BENZOYLSACCHARIN AS PEROXYGEN BLEACH ACTIVATOR

This invention relates to bleaching compositions. More particularly, it relates to activated bleaching compositions comprising hydrogen peroxide or a hydrogen peroxide-releasing compound such as an organic peroxide or inorganic peroxygen salt and N-benzoylsaccharin (I) as bleach activator.



(I)

The use of various N-acyl compounds as bleach activators has been disclosed in the art. For example, U.S. Pat. No. 3,177,148 discloses the use of various N-acyl compounds, including N-acyl derivatives of saccharin, such as N-acetylsaccharin, as peroxygen salt activators in bleaching compositions for textile materials. Thus, a perborate bleaching composition containing N-acetylsaccharin as activator removes a much greater percentage of tea stain on cotton textile than the same bleaching composition without the N-acetylsaccharin.

However, as a perborate bleach activator, N-acetylsaccharin has not proved satisfactory because of its poor storage stability. Thus, when used as an activator in a dry perborate bleaching composition, the N-acetylsaccharin tends to lose its activity during the time period between the manufacture of the bleaching composition and its eventual use. Thus, the time consumed in the shipping of the commercial product, plus warehouse storage and standing time on the supermarket shelf before it is purchased and used, often amounts to several months or more. Accordingly, to be practicably useful, a bleach activator should maintain its activity over this time period.

It has now been found that N-benzoylsaccharin is equally effective to N-acetylsaccharin as a bleach activator in freshly prepared bleaching compositions and, in contrast to the N-acetylsaccharin, maintains a relatively high degree of its activity over a prolonged period in such compositions.

An additional advantage of the N-benzoylsaccharin is that it provides dry bleaching compositions which not only exhibit good bleaching activity at relatively low water temperatures, but which are safer and easier to handle than liquid bleach products. Also, the N-benzoylsaccharin compositions are relatively safe for all fabrics.

The amount of the N-benzoylsaccharin to be incorporated in the bleach composition may vary over wide limits, depending on the application for which the composition is designed. Usually, the amount used will be that to provide a mole ratio of the N-benzoylsaccharin to peroxide-releasing compound in the composition of from about 1:1 to about 1:10, with the preferred ratio being about 1:1.1.

As hydrogen peroxide releasing compounds, examples of organic peroxides are: urea peroxide, benzoyl peroxide and methyl ethyl ketone peroxide. Exemplary of inorganic peroxygen bleaching compounds are: al-

kali metal perborates, percarbonates, perphosphates, persulfates, monopersulfates, and the like. Although various peroxide releasing compounds as mentioned above may be used in the compositions of the invention, preferred peroxide releasing compounds are sodium perborate (for economic considerations) and sodium percarbonate (for ecological reasons).

The activated bleach compositions of the invention are useful for application to various substrates, including fabrics, particularly when incorporated in detergent compositions for household or commercial laundering purposes. Thus, due to the presence of the bleaching composition, the detergent composition, in addition to its regular cleansing action, will have the ability to remove stains, including food stains, such as those of coffee and tea, as well as maintain purity of white in uncolored textiles.

In addition to the detergent, peroxygen-releasing compound and peroxygen bleach activator, such detergent compositions may contain other optional additives such as germicides, fungicides, enzymes, optical brighteners, colorants, perfumes, thickeners, emulsion or suspension stabilizers and the like.

The detergent component of such activated bleach composition may be any of the conventional types such as anionic, cationic, nonionic or amphoteric. Examples of typically suitable anionic detergents include the alkali metal or alkaline earth metal salts of higher alkylbenzene sulfonates, olefin sulfonates, higher alkyl sulfates and higher fatty acid monoglyceride sulfates.

Examples of typically suitable cationic detergents include tetraalkyl ammonium salts in which one of the alkyl groups contains approximately 12 to 18 carbons such as dodecyltrimethylammonium chloride and ethyldimethyloctadecylammonium methosulfate.

Examples of suitably typical amphoteric detergents are those detergent compounds possessing both cationic and anionic sites and include, for example, amino fatty acids, such as dimethylaminopropionic acid, and iminodifatty acids, such as methyliminodilauric acid.

Examples of typical nonionic detergents include polyglycol ethers of alkanol amides of higher fatty acids and also polyglycol ethers of higher alkanols and higher fatty acids.

The detergent chosen in a given instance is generally governed by the use anticipated for the formulation.

The bleaching compositions of the invention may also be used for their germicidal properties in various applications for control of microbial growth. Application may be made to any surface or substrate where such control is desired.

The treatment of swimming pool water and swimming pool surfaces with the compositions of the invention is especially efficacious since the usually lower temperatures of these environments prevent effective use of other antimicrobial agents. A related utility is the treatment of water supplies to render the same fit for human consumption or for industrial use, such as the sanitization of field water for consumption by military personnel or the treatment of industrial process water so it can be reused in industrial processes or by the surrounding community. The compositions also may be employed in admixture with detergents for use as home or industrial germicidal detergents.

The following examples will serve to illustrate the invention.

EXAMPLE 1

Preparation of N-Benzoylsaccharin

To 14.1 grams (0.1 mole) of benzoyl chloride and 18.3 grams (0.1 mole) saccharin suspended in 100 ml. of benzene there is added with stirring 10.0 grams (0.1 mole) of triethylamine dropwise, and this then refluxed over night. The reaction mass is clarified hot and the benzene filtrate concentrated to 50 ml. and cooled to give 22.4 grams (78.1% yield) of white solid. Recrystallized from acetone, m.p. 165°C.

EXAMPLE 2

Evaluation of N-Benzoylsaccharin as Activator for Sodium Perborate

A bleaching composition was prepared using N-Benzoylsaccharin and sodium perborate in a molar ratio of 1:1.1 and the bleaching effectiveness of this composition determined by the following test procedure: Five-gram swatches of desized cotton fabric are stained with tea in the following manner. Five tea bags are placed in one liter of water and boiled for 5 minutes. The swatches are then immersed in the tea and the boiling is continued for another 5 minutes. The swatches are then removed from the tea, wrung out, dried at 200°-215°F., rinsed in cold water and again dried.

Two of the stained cotton swatches are placed in a stainless steel Terg-O-Tometer vessel manufactured by the U.S. Testing Company. A solution of 2.0 grams of "Tide" (anionic detergent), 0.33 grams of sodium perborate and 0.86 grams of N-benzoylsaccharin in one liter of distilled water is then added to the vessel (pH is 9-9.5). Cut-up pieces of white terry cloth toweling are then added to provide a typical household washing machine water-to-cloth ratio of about 20 to 1. The Terg-O-Tometer is operated at 100 cycles per minute for fifteen minutes at a temperature of 50°C. At the end of the cycle, the swatches are removed, rinsed with cold water, and ironed dry.

Reflectance readings of the swatches are taken both before and after the cycle using a Hunter Model D-40 Reflectometer with a blue filter. Each swatch is read twice (warp and fill) on each side, with a backing of three similarly stained swatches. Fluorescent effect is excluded from all readings.

The reflectance readings are averaged and the percent stain removal determined by the following formula wherein R represents reflectance.

% Stain Removal = $\frac{[R \text{ (bleached)} - R \text{ (stained)}]}{[R \text{ (unstained)} - R \text{ (stained)}]} \times 100$

The test results showed that the bleaching composition, prepared as above, removed an average of 71.5% of the stain from the swatches, whereas in comparative tests in which no N-benzoylsaccharin was employed along with the sodium perborate only 45.1% of the stain was removed.

Similar results are obtained when "All" (a non-ionic detergent) is used in the tests instead of "Tide".

EXAMPLE 3

Evaluation of N-Benzoylsaccharin as Activator for Sodium Percarbonate

The effectiveness of N-benzoylsaccharin as an activator for sodium percarbonate was tested by the same procedure as in Example 2. In this case, the formulation was such as to provide 2.0 grams of "Tide" deter-

gent, 0.38 gram (1.1M) of sodium percarbonate and 0.86 gram (1.0M) of N-benzoylsaccharin per liter of solution. The test results showed that the bleaching composition removed 71.5% of the stain from the fabric.

EXAMPLE 4

Evaluation of N-Acetylsaccharin as Bleach Activator

A dry bleaching composition was prepared by mixing 1 mole proportion of N-acetylsaccharin with 1.1 mole proportions of sodium perborate. The composition was tested by the same procedure described in Example 2. In this case, the bath contained 2.0 grams of "Tide" detergent, 0.33 grams of sodium perborate and 0.68 gram of N-acetylsaccharin per liter of solution. The test results showed that the bleaching composition removed 71.4% of the stain.

EXAMPLE 5

The storage stabilities of N-acetylsaccharin and N-benzoylsaccharin in a dry bleaching composition were compared by testing the bleaching effectiveness of typical compositions containing each of the activator compounds after storage in an open container at 80°F. and 80% relative humidity and noting the change of bleaching effectiveness due to storage. The bleaching test procedure in all cases was the same as that described in Example 2, except, of course, that the bleaching formulation was prepared dry and stored for the time period indicated and then added, as such, to the bleach bath water at the time of the bleaching test. The test results are given in Tables I and II.

TABLE I

Bleach Bath		% of Stain Removed from Tea-stained Cotton
2.0 g/l Tide Detergent	control	41.0
0.33 g/l sodium perborate	freshly prepared	
no activator		
2.0 g/l Tide Detergent		71.9
0.33 g/l sodium perborate	freshly prepared	
0.68 g/l N-acetylsaccharin		
2.0 g/l Tide Detergent	stored 3 months in open container 80°F., 80% RH ⁽¹⁾	47.2
0.33 g/l sodium perborate		
0.68 g/l N-acetylsaccharin		

⁽¹⁾Refers to the dry formulation.

TABLE II

Bleach Bath		% of Stain Removed from Tea-Stained Cotton
2.0 g/l Tide Detergent	control	41.4
0.33 g/l sodium perborate	freshly prepared	
no activator		
2.0 g/l Tide Detergent		71.5
0.33 g/l sodium perborate	freshly prepared	

TABLE II-Continued

Bleach Bath		% of Stain Removed from Tea-Stained Cotton
0.86 g/l N-benzoylsaccharin		
2.0 g/l Tide Detergent	stored 4 months	55.5
0.33 g/l sodium perborate	in open container	
0.86 g/l N-benzoylsaccharin	80°F., 80% RH ⁽¹⁾	

⁽¹⁾Refers to the dry formulation.

The test results shown in Tables I and II illustrate the markedly superior storage stability of N-benzoylsaccharin over N-acetylsaccharin. Thus, it is seen that while the activity of the N-acetylsaccharin in a freshly prepared composition is comparable to that of the N-benzoylsaccharin in a freshly prepared composition, the N-acetylsaccharin retained only 20% of its original activity after 3 months storage, while the N-benzoylsac-

charin retained 47% of its activity after 4 months storage.

I claim:

1. A bleaching composition comprising hydrogen peroxide or a hydrogen peroxide-releasing compound and an activating amount of N-benzoylsaccharin.
2. A composition according to claim 1 wherein the mole ratio of N-benzoylsaccharin to hydrogen peroxide-releasing compound is from about 1:1 to about 1:10.
3. A composition according to claim 2 wherein the mole ratio of N-benzoylsaccharin to hydrogen peroxide-releasing compound is about 1:1.1.
4. A composition according to claim 1 wherein the hydrogen peroxide-releasing compound is sodium perborate.
5. A composition according to claim 1 wherein the hydrogen peroxide-releasing compound is sodium percarbonate.
6. A composition according to claim 1 containing a detergent.

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