8-111 SR
FIP8309 OR 1,767,543
CATION OF TEXTILES & FIBERS,
111

Patented June 24, 1930

1,767,543

UNITED STATES PATENT OFFICE

RALPH H. McKEE, OF JERSEY CITY, EARLE H. MORSE, OF NUTLEY, AND PHILIP EDWARD ROLLHAUS, OF EAST ORANGE, NEW JERSEY, ASSIGNORS TO PILOT LABORATORY, INC., OF ARLINGTON, NEW JERSEY, A CORPORATION OF NEW JERSEY

PROCESS OF BLEACHING CELLULOSE PRODUCTS

No Drawing. Application filed February 15, 1928. Serial No. 254,607.

This invention relates to a process of bleaching cellulose products such as artificial silk, cellulose ester fibers, artificial horsehair, cellophane, and the like, but particularly artificial silk, commonly known as "rayon".

The principal object of the invention is to provide a process of bleaching cellulose products without substantial deterioration or lowering of the grade of the products treated.

Another object of the invention is to provide a rapid, efficient, and relatively inexpensive process of bleaching rayon.

A further object of the invention is to provide a process of producing rayon of superior quality and improved luster.

A further object of the invention is to provide a process of bleaching cellulose products wherein a peroxidized organic acid is employed as the bleaching agent.

Other objects and advantages of the invention will become apparent from the following description.

In the methods of bleaching rayon now commonly practiced wherein alkaline solutions, such as solutions of sodium hypochlorite, hydrogen peroxide, calcium hypochlorite and the like, and in processes wherein acid solutions, such as solutions of permanganates, are employed as bleaching agents, there is a substantial loss in the market value of the product treated due to the deterioration and lowering of the grade of the product resulting from the deleterious action of the bleaching agent on the material treated. For example, in the alkaline processes the strength and elasticity of the rayon are materially decreased due to the modifying action of the alkaline material on the rayon. Moreover, in the acid processes various salts are deposited on the rayon and all of these salts cannot be or are not removed in the subsequent washing treatment. In addition, these processes are relatively expensive and involve a great amount of handling of the product being treated.

We have discovered that we can treat cellulose products and particularly rayon with organic bleaching agents in neutral, alkaline, or acid solutions, and preferably neutral or slightly acid solutions, with substantially no injury to the quality of the product, and at the same time accomplish the effective bleaching of the product more cheaply and easily than by employing any of the methods heretofore used for bleaching similar materials. Moreover, by our process we are able to produce commercially, highly satisfactory white or bleached rayon of a high degree of purity.

In the preferred practice of our process we prefer to employ as an organic bleaching agent peroxidized organic acids such as organic peracids, notably acetic peracid, and organic peroxids such as benzoyl peroxid. The organic peroxids although but slightly soluble in water have been found to be effective bleaching agents for cellulose products particularly when employed at a temperature slightly in excess of ordinary room temperature. However, we prefer to employ in the practice of our process organic peracids and especially acetic peracid in a neutral or slightly acid solution. An alkaline solution of an organic peracid may be employed but we do not recommend this procedure because of the deleterious effect of alkaline solutions on the strength and elasticity of rayon. Of the organic peracids we have found that the following are particularly adapted for use in connection with our process: benzoyl peracid, phthalyl peracid, acetyl peracid, succinyl peracid, maleic peracid and fumaric peracid.

Of the organic peroxids we have found that benzoyl peroxid, phthalyl peroxid, acetyl peroxid, succinyl peroxid, succinyl peroxid acid, benzoyl acetyl peroxid, maleic peroxid, and fumaric peroxid are particularly adapted for use in the practice of our process. Although the theory by which the bleaching of cellulose products takes place when these bleaching agents are employed is not definitely known, it is believed that the organic peroxids hydrolyze in an aqueous solution to form organic acid and an organic peracid and that the organic peracid serves as the bleaching agent. For example, benzoyl peroxid when treated with

water probably hydrolyzes in accordance with the following equation to give a mixture of benzoic acid and benzoic peracid:

$$(C_6H_5CO)_2O_2+H_2O\rightarrow C_6H_5COOH+C_6H_5COOOH$$

The amount of organic bleaching agents to be used in any particular case will vary according to the condition of the material to be treated, its initial color, the process of producing the material, the temperature and time of the bleaching operation, and upon the exact color or finish desired in the bleached material. However, the amount of bleaching agent to be employed is generally very small. For example, we have found 3 grams of benzoyl peracid in 10 liters of water is more than sufficient to bleach one kilogram of air-dried, unbleached viscose rayon. When using this amount of bleaching agent, the bleaching action will be complete in about one hour, the bleaching solution being maintained at approximately ordinary room temperature. It will be obvious that the bleaching process may be accomplished in a shorter period of time if the temperature of the bleaching solution is slightly raised. It has been found that rayon may be immersed in a bleaching solution of the character referred to for about twelve hours without detrimentally affecting to any substantial extent the finish or the elasticity of the bleached rayon. By employing a temperature of 35° to 65° C., more rapid penetration of the rayon by the bleaching agent may be obtained and the time of bleaching may be shortened. However, at these temperatures there is a slight decomposition of the bleaching agent in such manner that the decomposed portion does not serve to bleach the material and hence the use of higher temperatures is not always advisable.

We have discovered that when employing bleaching agents such as benzoyl peroxid which are only slightly soluble in water, the peroxid may be used in a fine suspension in water, preferably in a soap emulsion. The efficiency of the bleaching process is enhanced by the use of such an emulsion and rayon having a particularly soft finish is produced. In bleaching rayon according to this method we have successfully used approximately 1.5 grams of benzoyl peroxid suspended in approximately 5 liters of water to treat approximately one kilogram of rayon.

We also have found that succinyl peroxid acid is particularly adapted for use since this compound is a peroxid which is moderately soluble in water and that the peracid produced therefrom is relatively stable in an aqueous solution.

An acetic peracid solution containing acetic peracid in an amount equal to 0.4 per cent of the weight of the rayon to be bleached, the solution having been rendered distinctly acid either by the use of acetic acid or sulfuric acid, has been found to produce an excellent, rapid bleach for rayon either at ordinary room temperature or at a materially higher temperatue as, for example, at a temperature of approximately 50° C. After bleaching and rinsing, no special treatment is required and the goods may be taken directly to the driers or they may be subjected to any supplementary treatment.

Although we prefer to use organic bleaching agents in acid solutions for bleaching rayon, it is to be understood that satisfactory bleaches may be obtained by using the bleaching agents in alkaline solutions. If alkaline solutions are employed we prefer to use solutions which are only slightly alkaline, employing, for example, solutions of sodium phosphate or sodium bicarbonate rather than the more alkaline solutions of sodium carbonate or caustic soda. However, by exercising special care in handling the rayon we have obtained satisfactory results when employing solutions of strongly alkaline materials such as caustic soda. Soap solutions and soluble oils such, for example, as Turkey red oil, may advantageously be used in the bleaching bath, and in case the fibers are greasy or oily, such solutions have distinctly advantageous properties.

As will be readily understood, rayon bleached by any of the above mentioned organic bleaching agents may be removed from the bleaching bath, rinsed in warm or cold water, soaped, dyed, or dried in any usual or desired manner or otherwise submitted to any finishing treatment.

It is to be understood that we may employ a single peroxidized organic acid or mixture of several of such compounds. However, we have found that the use of a solution of a single peroxidized organic acid produces wholly satisfactory results in bleaching cellulose products.

The term "rayon" as employed is intended to include all types of artificial silk whether such silk be made by the viscose process, the cellulose acetate process, or by any other process for the production of artificial silk.

The term "peroxidized organic acids" as employed in the claims is meant to include both organic acid peroxids and organic peracids. The peracids may be used alone or may be used as produced in the process from the organic acid peroxids by the hydrolytic action of the solvent.

By the term "solution" as employed in the claims is meant a true solution or a suspension or emulsion so miscible with the solvent employed as to form in effect a solution.

While we have described in detail the preferred practice of our process and the materials which we prefer to employ, it is to be understood that we are not limited

8. BLEACHING & DYEING; FLUID TREATMENT & CHEMICAL MODIFICATION OF TEXTILES & FIBERS,
111

thereto except as set forth in the subjoined claims.

We claim:

1. The process of bleaching cellulose products which comprises subjecting the material to be treated to the action of a peroxidized organic acid.

2. The process of bleaching cellulose products which comprises subjecting the material to be treated to the action of a mixture of peroxidized organic acids.

3. The process of bleaching cellulose products which comprises subjecting the material to be treated to the action of an organic peracid.

4. The process of bleaching cellulose products which comprises subjecting the material to be treated to the action of a solution of a peroxidized organic acid.

5. The process of bleaching cellulose products which comprises subjecting the material to be treated to the action of an aqueous solution of a peroxidized organic acid.

6. The process of bleaching cellulose products which comprises subjecting the material to be treated to the action of a neutral solution of a peroxidized organic acid.

7. The process of bleaching cellulose products which comprises subjecting the material to be treated to the action of an aqueous neutral solution of a peroxidized organic acid.

In testimony whereof we affix our signatures.

RALPH H. McKEE.
EARLE H. MORSE.
PHILIP EDWARD ROLLHAUS.