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PROCESS OF HYDROLYZING CYANOETHYLATED COTTON TEXTILES WITH ALKALINE HYDROGEN PEROXIDE AND ACIDIC HYDROLYSIS

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This invention relates to cellulosic fabric or yarn having beta-carboxyethyl groups and more particularly to an improved process for producing such carboxyethyl substituted products from cyanoethylated cotton textile fibers containing a plurality of cyanoethyl groups and from 3% to 6% of nitrogen, by weight, of said fibers, the nitrogen being nitrile nitrogen in the cyanoethyl groups thereof, wherein cyanoethyl groups thereof are converted into carboxyethyl groups by a 2-step hydrolysis, including (1) an alkaline hydrogen peroxide hydrolysis, and (2) a mild, acidic hydrolysis, in that order, without destroying the fibrous structure thereof, all as more fully hereinafter described and claimed.

There is a considerable demand for cellulosic fibers and fabrics containing carboxyethyl groups. These fabrics retain the fibrous structure of cellulose, such as cotton, and at the same time have a high degree of affinity for water with a resultant high moisture regain and ready solution in dilute alkali. The first property permits fabrics which are used to absorb water, such as certain towelings, and the second property permits the use of such material as a backing for lace or other material which requires support but from which finally the backing has to be dissolved. Other uses for fibers and fabrics of this type are also known.

It has been proposed in the past to hydrolyze cyanoethylated cellulose fibers and fabrics, such as for example cyanoethylated cotton, in which the degree of cyanoethylation is below that which results in loss or serious weakening of the fiber structure of the cotton. Ordinarily this means cyanoethylation corresponding to from about 3% nitrogen to 6 or more percent. Previously proposed processes involve an alkaline hydrolysis, for example a treatment with caustic alkali solutions. It is, of course, possible to hydrolyze the nitrile groups under these conditions, particularly at slightly elevated temperatures. However, such drastic swelling often takes place that the nature of the fiber or fabric is drastically altered and far reaching disintegration or weakening takes place.

According to the present invention, I have found that if a hydrolysis is effected in two steps utilizing: (1) basic hydrogen peroxide then (2) mild acidic condition it is possible to hydrolyze a considerable portion of cyanoethylated cellulose, such as cyanoethylated cotton, without excessive swelling, resulting in weakening or disintergration of the fibers and fabrics. In general, the first hydrolysis step should be effected in the presence of a base such as ammonium hydroxide. After this step has been completed, the hydrolysis is continued by an acidic medium under conditions which wil hydrolyze the amide group to the carboxyl group but will not appreciably hydrolyze the glucosidic linkages of cellulose.

The invention will be described in greater detail in conjunction with the following specific examples, which are illustrative only. It should be understood that the invention is not limited to the use of cyanoethylated cotton, but is equally applicable to the hydrolysis of any other cyanoethylated alphacellulose material.

Example 1

A cotton cloth cyanoethylated to a nitrogen percentage of 3.2 is treated with an aqueous hydrogen peroxide solution, the proportions being 67 parts of cotton cloth to 130 parts of 35% hydrogen peroxide diluted with water to 375 parts. 12 parts of concentrated ammonium hydroxide is then added at room temperature and the reaction allowed to proceed with no attempt to reduce the temperature resulting from the exotherm of the reaction. This temperature rises to about 80° C., whereupon the mixture is cooled and allowed to stand at about 35° C. until reaction has proceeded as far as it will at this temperature. In general, the period of standing is about twice as long as the period during which the exotherm causes a gradual rise to about 80° C. The cloth is then removed, washed with water and then 0.1 N HCl followed by treatment with 2 N HCl at 60° C. until a maximum hydrolysis of $CONH_2$ to COOH groups takes place without material hydrolysis of other parts of the molecule. The cloth is then washed well past neutral with water, dried, and contains 1.59% nitrogen and about 0.69 molar equivalent of carboxyethyl groups per gram. The analysis corresponds to about 0.2 mol of nitrile or amide group and 0.1 mol of carboxy group per anhydro glucose unit. The hydrolysis does not result in transforming all of the nitrile groups to the carboxy form. There is in general some production of the intermediate hydrolysis phenomenon transforming nitrile groups into carboxamide groups.

The cloth shows a high moisture regain and can be used for various absorbing purposes. It is also readily disintegrated in dilute aqueous sodium hydroxide, and is therefore useful as a removable backing for lace.

Example 2

The procedure of Example 1 is repeated, substituting cotton yarn for the cotton fabric. Hydrolysis to substantially the same degree results and the yarn retains its fiber structure and can be woven into fabric having substantially the same properties as the partially hydrolyzed fabric of Example 1.

We claim:

1. As a process of producing carboxyethyl substituted cellulose products having a fibrous structure from cyanoethylated cotton textile fibers containing a plurality of cyanoethyl groups and from 3% to 6% of nitrogen, by weight, of said fibers, the nitrogen being nitrile nitrogen in the cyanoethyl groups thereof, the improved process which comprises reacting said fibers with an alkaline aqueous solution of hydrogen peroxide and ammonium hydroxide until a part of the nitrile groups are converted into amide groups, washing the so-reacted fibers with water and dilute aqueous hydrochloric acid to remove excess reagents, reacting the so-treated fibers with an aqueous solution of 2 N hydrochloric acid until the amide groups are converted into carboxylic acid groups to produce a carboxyethyl substituted cellulose product having a fibrous structure and containing about 0.1 mol of carboxy group and about 0.2 mol of nitrogen per anhdyroglucose unit thereof, the said aqueous alkaline and acidic treatments of said fibers being insufficient to destroy the fibrous structure thereof and insufficient to appreciably hydrolyze the glucosidic linkages thereof, and recovering the fibrous carboxyethyl substituted cellulose product so obtained.

2. The process of claim 1 wherein the said cayno-ethylated cotton textile fibers employed as the starting material therein is a cyanoethylated cotton cloth.

3. The process of claim 1 wherein the said cyanoethylated cotton textile fibers employed as the starting material therein is a cyanoethylated cotton yarn.

4. The process of claim 1 wherein the said alkaline hydrogen peroxide hydrolysis is effected at temperatures between 80° C. and 35° C.

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