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PROCESS FOR MAKING TEXTILE FIBERS

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The present invention relates in general to textile fibers which are composed in whole or in part of a cellulose ether and in particular to a process for the conversion of cellulose textile fibers into textile fibers composed in whole or in part of a cellulose ether which is soluble in water or aqueous alkali solutions.

In the prior specification Serial No. 218,220 filed July 8, 1938, there is disclosed for the first time a method of preparing textile fibers formed in whole or in part of a cellulose ether. Among the uses of such fibers are many valuable technical applications in the textile and cordage arts in which it is necessary to card, comb and spin the fibers. For such uses it is essential that the fibers are capable of being freely separated by the carding and combing operations. The fibers of the present invention are prepared by etherifying cellulosic textile fibers in the presence of alkali. The alkali tends under certain conditions of high concentration and low temperature to cause the etherified fibers to gelatinize sufficiently to stick together which interferes with their purification, drying and subsequent use. In particular the coalesced fibers cannot be readily separated by carding, combing and the like. Further in the production of textile fibers comprising alkali-soluble cellulose ethers it is desirable to obtain a product which is readily soluble in dilute caustic alkali solution of 2 to 3 percent strength when a mixture of the fibers and caustic alkali is cooled to 0° C. so that the use of cellulose ether fibers in admixture with rayon fibers which are not resistant to strong alkalies is rendered difficult or impracticable.

Accordingly, the chief object of the present invention is to convert cellulosic fibers of textile-making length superficially, partially or completely into a cellulose ether in the form of loose fibers which can be readily separated by carding, combing and the like.

According to the present invention, natural or artificial cellulosic fibers of textile-making length are superficially, partially or completely etherified in the presence of alkali and also in the presence of a substantial quantity of an added water-soluble inert salt, the reaction being carried out until the cellulose of the fibers is converted to a water-soluble or alkali-soluble cellulose ether and the fibers are then purified, preferably by washing the fibers with a solution of a water-soluble inert salt and then drying the fibers. The alkali remaining in the reaction mass is preferably neutralized by dumping the product into a large quantity of a dilute solution of any

suitable inorganic or organic acid. The product is then washed and dried. The proportion of reactants and the conditions of reaction are selected so that there is a minimum of degradation of the cellulose and the process is carried out so as to avoid breaking the fibers below a textile making length, as by avoiding shredding, cutting, etc. The presence of a substantial quantity of a water-soluble inert salt retards or substantially prevents the gelatinization and/or solution of the product in the reaction medium and/or in the washing liquors and renders the etherification more uniform. The individual fibers of the product can be readily separated by carding, combing and the like.

The expression "cellulosic fibers" as used herein, is intended to designate both natural and artificial fibers and filaments of all kinds, which are suitable for conversion in the process of the invention.

By way of illustrating, but not by way of limiting the invention, the following are examples of suitable cellulosic fibers which may be treated in the present invention: naturally-occurring cellulose fibers such as those derived from cotton, kapok, flax, hemp, jute, sisal, ramie, coconut, straw, wood and the like; cellulose hydrate fibers as derived from any source such, for example, as from the regeneration of cellulose xanthates, de-esterification of cellulose esters, or by precipitation from solutions of cellulose in organic or inorganic solvents; textile fibers comprising cellulose conversion products such, for example, as oxy-cellulose, hydro-cellulose, and the like; cellulose ethers such, for example, as cellulose ether xanthates of a low degree of etherification, cellulose mixed ether-esters, and the like.

In the preparation of the cellulosic raw material for treatment in the process, the materials may be subjected to any suitable purification treatment, if necessary, for the removal of non-cellulosic constituents, or for bleaching or other conditioning, as for example, by pretreatment with alkali. The excess alkali may be removed by pressing or centrifuging the treated material in a known manner.

Among the alkalies which may be used are the organic or inorganic bases such, for example, as sodium hydroxide, potassium hydroxide, amines, quaternary alkyl ammonium hydroxides and the like.

The conversion of the cellulosic raw material into a cellulose ether may be accomplished by the use of alkylating agents such, for example, as an alkyl ester of an inorganic acid, for ex-

ample, alkyl halides such as methyl chloride, ethyl chloride, ethyl bromide and di-methyl sulphate, and alkyl half esters of sulphuric acid; also an hydroxy-alkylating agent such, for example, as a halogen derivative of a polyhydric alcohol, such as ethylene chlorhydrin, propylene chlorhydrin, epichlorhydrin, and alkylene oxides, such as ethylene oxide, propylene oxide and glycidic; also a halogenated fatty acid or its salt or ester which converts the cellulosic material into a hydroxy acid ether of cellulose, suitable agents being chloroacetic acid, chloropropionic acid, and the like.

The expression "cellulose ether," as used herein, is intended to include any cellulose ether produced by reacting on cellulose with one or more of the mentioned reagents or their chemical equivalents.

The salt employed in the present process may comprise any water-soluble inert salt of an alkali metal with an inorganic or organic acid such, for example, as sodium or potassium chloride, sodium or potassium sulphate, sodium or potassium nitrate, and the like, sodium or potassium acetate, sodium or potassium citrate, sodium or potassium lactate.

The expression "inert" as applied to the salt, indicates that a salt is to be selected which is inert with respect to alkali or the etherifying agent employed in the process, and which does not swell or gelatinize the cellulosic raw material or the cellulose ether produced therefrom under the conditions of reaction. The concentration of the salt to be used in a particular case will depend, inter alia, upon the quantity of alkali and water present during the steeping or reaction, and upon the nature and character of the cellulosic fibers and of the product produced. It is merely necessary that a sufficient quantity be present to retard or substantially prevent gelatinization of the cellulosic raw material or of the product produced, and to inhibit or prevent the solution of the product in the reaction medium and/or in the washing liquors. In the preferred embodiment of the invention, the salt is used in an amount of from 10 per cent to 20 per cent, by weight, of the cellulose. It is to be understood that the salt may be recovered by known means, since it is not decomposed or used up in the process.

The salt may be added to the cellulosic raw material prior to or during the steeping or mercerizing of the initial raw cellulose with alkali, or may be dissolved in the alkali which is employed for such steeping or mercerizing; and/or the salt may be present during the reaction by which the cellulosic fibers are converted to the cellulose ether. When the salt is present during steeping or mercerizing, the quantity which remains, after pressing or centrifuging, may be sufficient to effect the novel results during the chemical reaction but, if not, additional salt may be added to the reaction mixture. In addition, the salt may be added to the liquors which are employed for purifying and washing the product.

It is to be understood that the conversion of the cellulosic raw material to the cellulose ether is carried out under suitable conditions of concentration and quality of reactants, temperature, pressure and other variables as known to those skilled in the art, and such processes, per se, when not employing a salt as herein described form no part of the present invention.

When a cellulosic material is etherified with

an etherifying agent derived from an acid in a reaction medium containing an inorganic alkali, it is obvious that an inorganic salt may be produced as a by-product. It is to be understood that the salt thus produced as a by-product is not sufficient to prevent the gelatinization or solution of the product and, therefore, does not effect the new and novel results obtained by the use of additional quantities of a water-soluble inert salt as provided in the present invention.

To prepare a fibrous material suitable for use in the textile and cordage arts, a vegetable material in the form of loose separable fibers of textile making length is converted into alkali-soluble cellulose ether by alkalinizing the vegetable fiber with a dilute alkali solution and etherifying the alkalinized fibers with a suitable etherifying agent in an alkaline medium in the presence of a water-soluble inert salt. The concentration of the alkali and the temperature of the alkalinizing treatment should be so correlated as to soften but not to gelatinize the fibers. An alkali concentration of from 10 to 20 per cent at a temperature of from 15 to 20° C. is suitable when the water-soluble inert salt is present in an amount of from 10 to 20 per cent based on the weight of the cellulose. With lower concentration of alkali the quantity of salt may be reduced. During the step of etherification, the mole ratio by weight of dry cellulose ($C_6H_{10}O_5$) to solid alkali calculated as sodium hydroxide preferably should not exceed 1:1.5, the water present during reaction being such that the concentration of alkali is not above about 20 per cent, preferably not above 10 per cent when etherifying cellulose hydrate fibers and not above 20 per cent when etherifying natural-occurring cellulose fibers, and the concentration of salt being sufficient to prevent the gelatinization or sticking together of the fibers during reaction.

By way of illustrating, but not by way of limiting the scope of the invention, there may be given the following examples but the invention is not limited to the precise proportion or ingredients, the times and temperatures and sequence of steps set forth. The parts are by weight.

Example I.—420 grams of air-dried cotton fibers of textile making length are steeped for one hour at 20 to 23° C. in a solution containing 18 per cent sodium hydroxide and 15 per cent sodium chloride, the mass then being extracted in a centrifuge until there is obtained a weight of 1470 grams. The alkali cellulose thus produced is aged for 16 hours at from 20 to 21° C. and then placed in an autoclave. To the autoclave 82 grams (20 per cent by weight) of ethylene chlorhydrin are added, the autoclave then being rotated for about 5 hours at a temperature being maintained at from 20 to 26° C. After about 5 hours, the cellulose ether fibers are removed from the autoclave and washed in boiling water until free of alkali and salt and thereafter treated with hot dilute oxalic acid to remove color due to iron and finally washed until free of acid, extracted in a centrifuge and dried. The product is a hydroxy ethyl ether of cellulose in the form of a textile fiber which is readily soluble in 3% caustic alkali at 0° C.

Example II.—100 parts of dry staple cotton fiber of textile-making length, which has been purified by a suitable pretreatment of non-cellulosic ingredients are alkalinized with 1000 parts of an 18 per cent aqueous solution of sodium hydroxide containing 8 per cent sodium chloride

based on the weight of dry cellulose for $\frac{1}{2}$ hour at room temperature until the cotton mass is thoroughly saturated with the alkali. The treated cotton is then centrifuged until the weight of the alkali solution remaining is substantially equal to twice the weight of the dry cotton. The centrifuged material is etherified with 15 per cent by weight of ethylene oxide at 25° C. in a closed container in the presence of at least 10 per cent of sodium chloride based on the weight of dry cellulose. At the end of about 12 hours, the reaction is substantially complete. The excess etherifying agent is withdrawn and the product is washed with hot water until substantially free of alkali and salt. The purified fiber is dried by passing a stream of warm air upwardly there-through until the fibers are substantially dry to the touch. The product consists of a mass of fibers of textile-making length comprising alkali-soluble cellulose ether, the individual fibers being separable one from another by carding, combing, etc.

The fibers produced by the present invention may be used alone or in admixture with other textile making fibers, such as cotton, flax or wool in the formation of textiles including felts, yarns, threads, cords, fabrics and woven textile structures of all kinds. The products have a wool-like feel, warmth and appearance.

The textile fibers of the present invention are particularly adapted for use as coalescing fibers. For example, a yarn formed from the fibers of the invention may be treated with a solvent such as an aqueous alkali solution of sufficient concentration to superficially, partially or substantially gelatinize the fibers and render them adhesive, whereby the yarn may then be squeezed to cause the fibers to coalesce and the alkali neutralized with an acid to fix the fibers in the coalesced condition. Likewise the fibers of the present invention may be used as potentially adhesive fibers and thus after being admixed with non-adhesive textile fibers, they may be formed into yarns, threads, cords, fabrics and other textiles and the cellulose ether fibers rendered adhesive by a suitable solvent to produce stiffened textiles as disclosed and claimed in prior British Patent No. 501,436.

Having described my invention, what I claim and desire to be secured by Letters Patent is:

1. A process for the production of textile fibers which comprises treating a mass of loose cellulosic fibers of textile-making length with an etherifying agent in the presence of an aqueous solution of an alkali capable of forming an alkali cellulose thereof, continuing the etherification until the cellulose ether fibers produced are soluble in caustic alkali, the etherification being carried out in the presence of a quantity of a water-soluble inert salt of an alkali metal sufficient to prevent the sticking together of the fibers to obtain alkali-soluble cellulose ether fibers of textile-making length which fibers can be readily separated by carding, combing, and the like.

2. A process for the production of textile fibers which comprises treating a mass of loose cellulosic fibers of textile-making length with an alkaline swelling agent in the presence of a water soluble inert salt of an alkali metal and thereafter treating the swollen fibers with an etheri-

fying agent in the presence of an aqueous solution of an alkali capable of forming an alkali cellulose thereof, continuing the etherification until the cellulose ether fibers produced are soluble in caustic alkali, the etherification being carried out in the presence of a quantity of a water soluble inert salt of an alkali metal sufficient to prevent the sticking together of the fibers to obtain alkali-soluble cellulose ether fibers of textile-making length which fibers can be readily separated by carding, combing, and the like.

3. A process for the production of textile fibers which comprises treating a mass of loose cellulosic fibers of textile-making length with an etherifying agent in the presence of an aqueous solution of an alkali capable of forming an alkali cellulose thereof, continuing the etherification until the cellulose ether fibers produced are soluble in caustic alkali, the etherification being carried out in the presence of a quantity of a water-soluble inert salt of an alkali metal sufficient to prevent the sticking together of the fibers, washing the treated fibers with an aqueous solution of a water-soluble inert salt of an alkali metal in sufficient quantity to prevent gelatinization of the fibers to obtain alkali-soluble cellulose ether fibers of textile-making length which fibers can be readily separated by carding, combing, and the like.

4. A process for the production of textile fibers which comprises treating a mass of loose cellulosic fibers of textile-making length with an etherifying agent in the presence of an aqueous solution of an alkali capable of forming an alkali cellulose thereof, continuing the etherification until the cellulose ether fibers produced are soluble in caustic alkali, the etherification being carried out in the presence of a quantity of a water-soluble inert salt of an alkali metal sufficient to prevent the sticking together of the fibers, washing the treated fibers with an aqueous solution of a water-soluble inert salt of an alkali metal in sufficient quantity to prevent gelatinization of the fibers to obtain alkali-soluble cellulose ether fibers of textile-making length which fibers can be readily separated by carding, combing, and the like, and drying the fibers by passing a current of warm air upwardly through the mass of fibers.

5. A process for the production of textile fibers which comprises treating a mass of loose cellulosic fibers of textile-making length with an alkaline swelling agent in the presence of a quantity of water-soluble inert salt of an alkali metal sufficient to prevent the gelatinization of the fibers, treating the swollen fibers with an etherifying agent in the presence of an aqueous solution of an alkali capable of forming an alkali cellulose thereof, continuing the etherification until the cellulose ether fibers produced are soluble in caustic alkali, the etherification being carried out in the presence of a quantity of a water-soluble inert salt of an alkali metal sufficient to prevent the sticking together of the fibers and washing the fibers with an aqueous solution containing a water-soluble inert salt of an alkali metal and drying the fibers to obtain alkali-soluble cellulose ether fibers of textile-making length which fibers can be readily separated by carding, combing, and the like.

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