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TREATMENT OF FIBROUS CELLULOSE FOR THE PRODUCTION OF HYDRATED DERIVATIVES.

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To all whom it may concern:

Be it known that we, CHARLES FREDERICK CROSS and EDWARD J. BEVAN, both subjects of the King of Great Britain, both residing in London, England, have jointly invented a certain new and useful Treatment of Fibrous Cellulose for the Production of Hydrated Derivatives, of which the following is a specification.

The action of the alkaline hydrates in strong solution on fibrous celluloses determines structural changes in the fibres, especially marked by shrinkage, and in the case of selected cotton textile fabrics the action, with certain mechanical aids, is applied to produce the effects known as "mercerization". In practice the mercerizing process consists in applying under the suitable conditions, an aqueous solution of caustic soda of 15–17.5 per cent, (NaOH) at ordinary temperatures. The effects are diminished as the concentration of the caustic soda is diminished below 15 per cent; with solutions of 12.5 per cent to 10.5 per cent (NaOH) the diminution of effect is rapid, and it may be said that at strengths below 12.5 per cent caustic soda solution has no substantial mercerizing effect. Thus cotton or cotton fabric treated with an alkaline lye of 9 per cent caustic soda (NaOH) is not sensibly affected in the direction of mercerization.

For the production of alkali-cellulose in the synthesis of water soluble cellulose derivatives (by subsequent action of carbon disulphide) as described in specification of British Letters Patent No. 8700 of 1892, it is known that the cellulose must be taken to the stage of full mercerization and therefore the fibrous cellulose must be treated with a solution containing the equivalent of upwards of 15.0 per cent of caustic soda (NaOH). Alkali-celluloses for industrial production of viscose, which implies complete conversion to water soluble derivatives in the subsequent reaction, are prepared by treating the air-dry cellulose with caustic soda lye of 17 per cent (NaOH) strength and pressing the product so that the fibrous mass retains solution amounting to 3 times the weight of the cellulose, inclusive of absorbed alkali. On the other hand, when a less concentrated solution is used the formation of soluble derivatives on subsequent or simultaneous treatment with carbon disulphide is increasingly imperfect. Thus, when cotton or other cellulose is uniformly impregnated with solution of 9 per cent (NaOH) in the proportion of twice the weight of the fibre or fabric, and then exposed to carbon disulphide, only a small fraction of the material is converted into water soluble derivatives.

The attendant formation of water-insoluble xanthates is also fractional in amount. But, as we have discovered, fundamental reaction occurs, and the cellulose is profoundly modified. The new characteristics are evidenced in further reaction with water; structural changes in the fibre are developed, with large increase of diameter, and thickening of the cell wall, and in the mass there are visible effects of hydration. The fibrous mass swells considerably, and retains a very much increased percentage of water, after having been pressed or centrifuged. The fibres, however, though much distended remain free, and may be readily and quickly washed free from excess of alkali, and the small proportion of soluble cellulose compounds. For subsequent removal of residual alkali and decomposition of fractional residues of water-insoluble xanthates, a treatment with dilute acid may be desirable.

It is this fully hydrated condition of the cellulose and the implied corresponding plasticity of the substance, which gives rise to the novel technical results of the present invention, which consists in modifying the cellulose for subsequent full hydration by treatment of the cellulose with alkali solution of a strength between 6 and 11 per cent (NaOH), and simultaneously or subsequently, with carbon disulphide.

As an example of the treatment, raw cotton fibre may be impregnated with twice its weight of caustic soda, of 9 per cent strength and introduced into a closed chamber together with carbon disulphide amounting to say 15 per cent of the weight of the original fibre. After a period of 6–10 hours, at the ordinary temperature, the mass is transferred to a convenient apparatus, in which the product is now washed with water at ordinary temperature for removal and recovery of alkali and to determine or develop the effects of hydration.

The structural modifications produced in the cotton fibre and the changes accompanying the swelling of the fibre generally resemble those produced by saline solvents of cellulose in the earlier stages of action, that is mainly in the thickening by swelling of the cell wall. A more special effect is the exaggeration of the central canal defined by symmetrical sinuous outlines. In extreme treatment a definite continuous spiral appears in the mass of swollen fibre substance. This development of the ultimate spiral differentations of the external tissues of the cotton is more marked if the preparatory treatment with alkali is carried out at higher temperatures, say 80° to 90° C.

The hydration effects are enhanced by increasing the proportion of alkali to fibre in the first stage of treatment, for instance by using an amount of caustic soda solution equal to 2.5 to 3.0 times the weight of the fibre. The effects may also be controlled by varying the temperature of the alkali solution, the action of the latter in determining hydration effects being greater the lower the temperature; on the other hand, raising the temperature alters the structural characteristics of the hydrated product, and particularly in the sense that when formed into a mass, or in sheets and subjected to pressure it yields a parchment-like material.

The finally reverted air dry product generally retains 10 to 11 per cent of moisture as compared with the 6 to 7 per cent normal to the raw cotton fibre, which is correlative with a generally increased dyeing capacity.

Instead of washing with water the material which has been attacked by carbon disulphide, it may be washed with a strong solution of a neutral alkali salt, for instance brine, to remove excess of alkali; the hydration effect is thus suspended and can be subsequently produced by treatment with water. This brine treatment is useful in connection with the recovery of the alkali, which is thereby obtained in more concentrated state, as it is in furnishing a product which can be subsequently hydrated by washing away the brine.

For the purposes of this invention the term "fibrous cellulose" includes the well-known industrial forms of cellulose: cotton, flax, hemp, esparto, wood cellulose, etc. Also, raw cottons which contain only small proportions of alkali-soluble constituents; otherwise, as in the case of flax, it is advantageous to operate on the alkali-scoured material; also papermakers' half stuff, or pulps in the bleached or the unbleached state, in which they retain residues of non-cellulose components.

The invention may also be applied to compound celluloses of the type of jute fibre, in treating which the upper limit of concentration of the alkali used in the preliminary treatment is required.

In regard to the technical uses of the product, its use in paper-making has been noted.

It is to be noted that the hydration effects give the necessary papermaking quality, and are independent therefore of any hydration effects such as produced in the ordinary Hollander or beater. Therefore, also fibrous cellulose of any length of fibre may be brought into the state necessary for wet manipulation with conservation of length. Hence, fine felted sheets of say 15 grammes per sq. meter of area can be produced, showing considerable adhesion in the final reverted state.

The hydrated fibre in admixture with ordinary paper-making cellulose, that is, as a mixed fibre, produces its proportionate effect, and it is characteristic of the reversion that in the final form the fibre retains elasticity and bulk.

In treating textile materials the process may be applied to roving, to be afterwards spun and drawn by the wet process to yarn. Yarns and cloth may also be treated with or without the tension necessary to oppose shrinkage of the material which occurs in the hydration process.

In this way a range of finishes and effects are obtained with some of the characteristics of mercerization, but differentiated by reason of the relative elasticity and bulk of the fibre or material in the finished state.

Having thus described the nature of the same invention and the best means we know of carrying the same into practical effect, we claim:—

1. A process of treating fibrous cellulose which consists in subjecting the cellulose to the action of alkaline solution of strength between 6 and 11 per cent (NaOH), and of carbon bisulphide at the ordinary temperature.

2. A process of treating fibrous cellulose which consists in subjecting the cellulose to the action of alkaline solution of strength between 6 and 11 per cent (NaOH) and then to the action of carbon bisulphide at the ordinary temperature.

3. A process of treating fibrous cellulose which consists in subjecting the cellulose to the action of alkaline solution of strength

between 6 and 11 per cent (NaOH) and of carbon bisulphide at the ordinary temperature, and finally washing the product with a strong solution of a neutral alkali salt.

4. A process of treating fibrous cellulose which consists in subjecting the cellulose to the action of alkaline solution of strength between 6 and 11 per cent (NaOH) and then to the action of carbon bisulphide at the ordinary temperature, and finally washing the product with a strong solution of a neutral alkali salt.

5. As a new product, hydrated fibrous cellulose having a thickened cell wall and an exaggerated central canal defined by symmetrical sinuous outlines.

In testimony whereof we have signed our names to this specification.

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