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NITROGENOUS TEXTILE FIBER

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5 Claims. (Cl. 18—54)

This invention relates to a process for the production of wool-like artificial textile fibers, in which nitrogenous raw (starting) material is dissolved in alkalis and thereafter spun in a coagulating bath.

In known processes of this kind the threads obtained must be subjected after coagulation to a special fixing bath, for instance a formaldehyde bath, to prevent the proteid substances contained therein, that is their nitrogen content, from being lost in the ordinary aftertreatments to which the thread is subjected in the production of textile fibres (desulphurising bleaching, boiling in 1-2% acid baths).

As distinct therefrom, it is a feature of this invention that the fixing substances is admixed to the mass before spinning, preferably as the raw materials are dissolved in alkalis.

This process has the advantage that a special after-treatment of the threads for fixing the nitrogen content becomes superfluous and the fixing substance is present throughout the thread mass and not only on the outer surface thereof, so that, for instance, it is not objectionable in dyeing.

According to this invention it is possible to employ fixing substances which undergo chemical alterations in the dissolving processes, as well as fixing substances which behave indifferently in said processes. Phenols and its homologues and derivatives, for instance creosote are more particularly advantageous fixing agents inasmuch as they have a cleaning action on the raw material, making it possible for instance to employ vegetable casein from commercial glutens as starting material.

When phenols etc. are employed, an excess of alkali, for instance caustic soda solution, is required, to secure besides the sodium phenate C_6H_5ONa formed, $NaOH$ enough for dissolving the nitrogenous raw materials. On coagulation of the threads spun, which may be carried out in the acid bath containing inorganic salts as usually employed in the production of viscose silk, phenol is formed back from the sodium phenate and remains in the thread mass as a fixing agent for the proteid substances. Phenol may be replaced in part by a borate, for instance sodium borate, which is a fixing agent that does not take part in the chemical reactions occurring in the process or only does so to a small extent. Phenols and their homologues, such as creosote, have only a very low acidity, which, moreover, is neutralized by caustic soda or other alkali. Therefore, all these substances have no

precipitating effect on casein, which is not pure, nor on viscose. They may be used in processes which employ commercial casein and in which viscose is added to the nitrogenous mass.

Spinning may be carried out by the same methods and on the same machines as usually employed for the production of artificial silk threads.

The threads obtained, which may be produced also in the form of staple fiber, are similar to animal wool and have substantially the same properties as regards strength, easiness of dyeing, and heat insulation, as well as with regard to the nitrogen content, which is directly fixed therein and is not lost when the threads are desulphurised, bleached and boiled in 1-2% acid baths.

The nitrogenous mass dissolved in caustic soda solution and containing the fixing agent may be admixed with 10-75% cellulose solution suitable for spinning, for instance viscose (cellulose xanthogenate), cellulose acetate, cuprammonium cellulose. In this case, the nitrogen content in the threads, which on spinning nitrogenous mass alone may reach the value of the nitrogen content in animal wool, is reduced in accordance with the quantity of cellulose solution added.

It is advisable to employ vegetable casein obtained from glutens as nitrogenous starting material for carrying out the process according to this invention. It is known that a number of cereals and leguminous plants have a high albuminoid content and are therefore easily transformable into glutens. Vegetable casein for carrying out the process according to this invention may be derived for instance from commercial wheat gluten, rice gluten, maize gluten, soja bean gluten, hopper glutens.

As further raw materials we may mention for instance gelatinous materials, such as commercial gelatine, fish-glue, joiner's glue, as well as slaughter-house residues such as meat residues, hair, skins, horny matter, bones, etc.

Two examples of the process according to this invention shall now be described. They relate to the production of gluten mass apt to be spun alone and a gluten mass mixed with cellulose xanthogenate, respectively.

1. Production of a mass suitable for spinning from glutens transformed into vegetable casein

- (a) About 2,500 kgs. ordinary glutens transformed into vegetable casein;
- (b) About 0.600 kgs. phenol and
- (c) About 2,500 kgs. water,

Phenol may be in part replaced by sodium borate.

All these ingredients are brought to 50° C. and stirred together over 20 minutes. The following further ingredients are then added

- (a) 1.50 liters 18% soda and
- (b) 9.00 liters water.

The mixture is stirred during two hours cooling to a temperature between 24 and 28° C., whereupon it is filtered and evacuated. Sodium caseinate and sodium phenate have been formed. Ripening is carried on for about 64 to 70 hours at 22-24° C. Viscosity remains thus constant at 15-18 for about 74 hours and more; then spinning may be effected on the usual spinning machines employed for rayon and staple fiber.

Composition of the "viscose":

- Glutens about 15 to 16%
- NaOH (soda) about 1.30 to 1.60%
- Phenols about 4%.

Coagulating bath formed by:

- About 250 grams acid per litre and
- About 150 grams zinc sulphate per litre
- At the temperature of about 55-60° C.

2. Preparation of a "viscose" formed by glutens transformed into vegetable casein with the addition of viscose in the form of cellulose xanthogenate

- (a) About 100 kgs. cellulose xanthogenate containing 8.20% cellulose and 6.50% soda;
- (b) About 8.00 kgs. ordinary glutens transformed into vegetable casein;
- (c) About 2.40 kgs. phenol and
- (d) About 8.00 liters water.

Phenol may be replaced in part by sodium borate.

The above mentioned quantities of phenol and water mixed together and the mixture is heated to the temperature of 55° C.; the glutens (kgs. 8.00) are added little by little stirring about 20 minutes until a voluminous coagulation product is formed which swells.

9 liters water mixed with 7 liters soda containing 18% NaOH are added, stirring further 20 minutes and cooling to 25° C.

To this mass comprising sodium caseinate and sodium phenate 100 kgs. viscose in the form of cellulose xanthogenate are added at a temperature of about 20-25° C., stirring for thirty minutes or longer till the two viscoses are well incorporated in each other, whereupon the mass is filtered as in the case of rayon viscose.

The "viscose" thus obtained before filtration shall have a coagulation degree of 7 to 7½ and a viscosity of 60-65.

After filtering, "viscose" is treated under vacuum during at least 10 hours in order to evacuate it, maintaining a temperature not lower than about 20° C.

Before addition of the mass from gluten compounds, the cellulose viscose must have ripened during 54-56 hours after starting sulphuration.

After ten hours treatment under vacuum, viscose obtained as above shall have a coagulation degree of 4¼ to 4½ and a viscosity of 32-36.

These qualities are stable for over 15 hours.

The material can now be fed to the spinning machines and be spun with the usual rayon spinning nozzles and coagulated with the same bath ordinarily employed for normal rayon and staple fibre at a temperature of about 50-60° C.

In this bath, temperature and concentration shall be increased in small proportions as the percentage of glutens in viscose increases.

After spinning no further baths, such as formaldehyde or other baths, are required for fixing nitrogen, and the fibre is successively treated like staple fiber or rayon. It may be desulphurated, boiled in an acid bath, bleached as usual with hydrogen superoxide without the smallest loss of the nitrogen incorporated and fixed therein in the percentage determined before starting the treatment.

What I claim is:

1. A process for the manufacture of artificial nitrogenous textile fibers consisting in dissolving a proteid soluble in caustic alkali with an excess of alkaline solvents, adding a substance selected from the group consisting of phenol and its homologues, in such manner as to form a compound selected from a group consisting of alkaline phenates and their homologues and ripening the nitrogenous mass thus obtained.

2. An artificial nitrogenous textile fiber, comprising throughout its mass a substance selected from a group consisting of phenols as a fixing agent opposing the tendency of the protetin present in the fiber to dissolve.

3. In a process for the manufacture of artificial nitrogenous textile fibers the steps consisting in mixing together in water about 15% to 16% of a proteid soluble in caustic alkali, about 4% of a phenol, in heating said mixture to about 50° C., stirring the mixture for about 20 minutes, adding to the mixture about 1.30% to 1.60% caustic soda dissolved in water, mixing for about two hours while cooling to about 24° to 28° C., and ripening the mass for about 64 to 70 hours at about 22° to 24° C.

4. In a process for the manufacture of artificial nitrogenous textile fibers the steps consisting in mixing together in water about 15% to 16% gluten, about 4% phenol, heating said mixture to about 50° C., stirring for about 20 minutes, adding to the mixture about 1.30% to 1.60% caustic soda dissolved in water, mixing for about two hours while cooling to about 24° to 28° C. and ripening for about 64 to 70 hours at about 22° to 24° C.

5. In a process for the manufacture of artificial nitrogenous textile fibers the steps consisting in dissolving phenol in water, heating to about 55° C., adding gluten little by little to the mixture and stirring for about 20 minutes, adding an 18% caustic soda solution and stirring for a further 20 minutes while cooling to 25° C., so as to obtain a mass containing sodium glutenate and sodium phenate adding previously ripened cellulose xanthogenate to said mass while maintaining a temperature of 20 to 25° C. and mixing till the said mass and cellulose xanthogenate are fully incorporated with each other.

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