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PROCESSES FOR THE MANUFACTURE OF ARTIFICIAL TEXTILE FIBRES

Antonio Ferretti, Milan, Italy; vested in the Alien Property Custodian

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This invention concerns improvements in or relating to processes for the manufacture of artificial textile fabrics.

A process is described in co-pending U. S. Patent application Serial N. 96,470 of 1936, by which casein may be produced which is suitable for use in the manufacture of artificial textile fibres. Casein, however delivered, is soluble in all alkalies giving viscous colloidal solutions. The present invention provides in a process for the manufacture of artificial textile fibres the selection of sodium hydrate and/or potassium hydrate as solvents for casein in the production of a colloidal solution thereof. Hitherto the weaker alkalies such as sodium borate, sodium carbonate and ammonium hydrate have been employed for this purpose because they do not affect the casein. According to the present invention, however, sodium hydrate and/or potassium hydrate are selected from all known alkalies for the production of a colloidal solution of casein although they have hitherto been avoided because they attack casein more than any other alkalies. All other alkaline agents, even if combined together in the most varied proportions, produce colloidal solutions of casein which cannot be spun. This fact, which has been ascertained as the result of numerous experiments, applies not only to the textile casein produced by the process described and claimed in the specification of the patent application referred to above, but to all trade caseins whether lactic or acid. Thus despite the disadvantages normally associated with sodium hydrate and potassium hydrate, only these two alkaline agents can be employed, according to the present invention, for the production of a colloidal solution of casein suitable for use in the manufacture of artificial textile fibres.

In practice it is preferable to employ sodium hydrate because it is cheaper than potassium hydrate while the results are identical. A mixture of sodium hydrate and potassium hydrate can also be used. Also, small quantities of other alkaline agents may be added to the solution in order to modify slightly the properties of the colloidal solution. It is necessary, however, to confine these additions within narrow limits, because otherwise the colloidal solution of casein would lose its spinning properties. In other words, it is essential that the alkaline agent employed for the colloidal solution of casein should preponderately consist of sodium hydrate or of potassium hydrate or of a mixture thereof.

In the preparation of the solution casein and alkali are employed in the equivalent ratio of 23

litres of 35° Bé sodium hydrate to 100 kilogrammes of dry unwashed casein containing a normal quantity of moisture, with a tolerance on the quantity of alkali employed of 20% above or below the indicated value.

The amount of sodium hydrate preferably to be employed in order to obtain a colloidal solution of casein suitable for the manufacture of textile fibres has been calculated upon the assumption that a temperature of 24° C. is employed for the solution of the casein and for its subsequent maturing. However, the amount of sodium hydrate may be slightly reduced if the adopted temperature is higher than the temperature indicated above, and conversely the amount may be slightly increased for a lower temperature. No true reverse proportion, however, exists between the quantity of alkali and the temperature adopted, because the quantity of alkali generally influences the solubility of the casein, whereas the temperature generally influences the viscosity, volume and maturing of the colloidal solution.

If the amount of sodium hydrate employed remains unaltered an increase in temperature results in a greater volume and viscosity and more rapid saturation of the colloidal solution.

It is not advisable however to depart considerably from the mean temperature of 24° C. For instance, if the temperature of 35° C. is progressively exceeded, the volume progressively becomes excessive and the viscosity unduly great as compared with the viscosity necessary for spinning and, above all, the risk of seriously endangering the quality of the ultimate product is incurred. Conversely, with a temperature below 14° C., an unduly large and useless delay occurs in the maturing and it is difficult, if not impossible, to realize the desired volume of colloidal solution.

It is possible to increase the viscosity of the colloidal solution of casein without altering its spinning property (and even improving the same) by employing special diluting agents hereinafter specified. These diluting agents, however, always have the same effect upon the viscosity and the volume of the colloidal solutions whatever may have been the preparation and maturing temperatures, so that the differences of the colloidal solutions of the textile casein in viscosity and volume remain practically unaltered relatively to one another at the different temperatures.

Various examples of colloidal solutions of tex-

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tile casein will be given in order that the present invention may be clearly understood.

Example A

When a dried casein that has not been washed after coagulation is employed:

A predetermined quantity of dried and ground casein is placed in a suitable vessel fitted with a stirring device and with a double bottom and double walls for the circulation of the water necessary to regulate the temperatures during the dissolving, diluting and maturing of the colloidal solution of casein.

For each 100 kilos of dried casein containing a normal moisture percentage of 10%, 200 litres of water previously heated to a temperature of 24°C. are poured into the vessel, and uniformly distributed throughout the casein which has been previously placed in the vessel. However, it is also practicable to pour the water into the vessel before the casein has been introduced.

After the water and casein have been mixed for two or three hours there is introduced into the vessel a solution comprising 23 (twenty three) litres of 35° Bé. sodium hydrate and 77 (seventy seven) litres of water, previously heated to a temperature of 24° C. for each 100 kilos of dried casein referred to above. The solution is energetically stirred in order rapidly and uniformly to incorporate the sodium hydrate with the casein solution. When the mixture has become uniform the stirring may be slowed down or be effected only at intervals. The temperature is preferably maintained constant at 24° C. It may be mentioned that instead of employing prepared 35° Bé. sodium hydrate, a corresponding quantity of dry sodium hydrate may be dissolved in water. Likewise, the procedure is the same if instead of sodium hydrate, potassium hydrate is employed, bearing in mind that, the temperature of 24° C. remaining unaltered, the quantity used must correspond to the alkalinity or neutralising power of 23 litres 35° Be. sodium hydrate for each 100 kilos of dry casein containing 10% moisture. If the moisture percentage in the casein employed is higher or lower, the amount of water and alkali to be added to the casein must be varied accordingly.

When the casein has been dissolved and the mass has gradually become more viscous (this being effected in a few hours depending upon the type of casein employed, namely, whether it has been coagulated with a greater or smaller quantity of acid, and a temperature of 24° C. has remained unaltered), a slow and gradual dilution is effected so as to compensate for the increasing viscosity of the colloidal solution whilst it matures.

The diluting agents hereinafter specified and previously heated to a temperature of 24° C. are preferably added little by little in order to avoid momentarily an unduly great dilution which would delay the regular and gradual development of the viscosity necessary to permit the subsequent additions of diluting agents until the desired volume of the colloidal solution is obtained and without causing this solution to lose its spinning property.

When the desired volume and viscosity of the colloidal solution have been obtained, the increase in the viscosity can be stopped by slightly lowering the temperature of the solution, the solution thus remaining stabilised for a certain time so that it can be transferred to the spinning machines with the properties and charac-

teristics previously established. This is of the greatest importance for regular spinning which otherwise would be difficult to obtain.

The time necessary for accomplishment of the above features, that is the time required for the dissolving, the maturing and the increasing of volume of the colloidal solution of casein may vary within wide limits, because the said time does not depend solely upon the desired volume and viscosity but also upon the types of the caseins, and diluting agents that are employed and upon the temperature employed during the alkaline dissolving and maturing of the casein.

A casein obtained from 250 cubic centimetres of 66° Bé sulphuric acid for each 100 litres of skimmed milk by the methods described above, dissolved in the manner indicated at a constant temperature of 24° C, and diluted with ordinary water, generally takes 48 hours to mature in order to reach a viscosity suitable for spinning and a volume of 550-600 litres for each 100 kilos dry casein used. When a smaller volume is desired, all other factors remaining unaltered, the time for maturing may be reduced somewhat. When a greater volume is desired, the time for maturing may be increased but only up to a certain limit because it has been found that after 96 hours, for instance, it is possible detrimentally to effect the quality of the textile fibres obtained.

Also the quantity of acid employed for coagulation of the casein, and to a slight extent also the types of acid employed, play an important part in determining the viscosity and volume of the alkaline solution of casein. The greater the amount of the acid employed for coagulating casein, the viscosity and volume of the subsequent alkaline colloidal solution are lower and are obtained less rapidly. Conversely, the smaller the quantity of acid employed so the viscosity and volume of the solution are higher and are obtained more rapidly. By an excessive variation of the quantity of acid in the one or in the other direction, solutions can be obtained which are difficult to spin, apart from the lower quality of the resulting fibres.

Accordingly, when caseins having the different properties indicated above are employed, it is preferable and—in the extreme cases mentioned essential—to modify the initial volume of casein solution.

In the example indicated above the initial volume after the addition of the sodium hydrate solution is 400 litres for each 100 kilos of casein employed. This volume may be slightly increased or reduced, but only between narrow limits because by increasing the volume by a greater addition of water, while the other factors remain unaltered, the complete dissolving of the casein and the development of the viscosity requisite for proceeding to the successive additions of diluting agent are delayed. If an excessive initial volume of casein solutions is employed, the regular development of the process may be irreparably spoiled. On the other hand, a reduction of the initial volume of casein solution renders an intimate mixture of the alkali and the casein, and consequently a perfect solution, more difficult and impossible, if the initial volume of casein solution is very small.

The volume referred to above is suitable for the type of casein indicated. For caseins that have been coagulated with a greater quantity of acid it is advisable to reduce slightly the volume of the initial solution, and conversely to increase

the same for caseins that have been coagulated with a smaller quantity of acid.

The casein that has been washed prior to drying is dissolved in the same manner as that which has not been washed, with the difference that the quantity of alkali employed is reduced according to the lesser quantity of acid left in the casein in consequence of washing.

Example B

The dissolving of casein which has not been dried, that is to say, casein which still contains a percentage of acid serum, is carried into effect with the following modifications.

The casein, coagulated and heated in its serum as indicated above and previously deprived of its excess moisture, is pressed so as to reduce its serum content, preferably to less than 200% of the weight of the dry casein, this being effected in order to facilitate the subsequent grinding of the pressed casein.

The casein is placed in a vessel constructed as previously described, without addition of water which in this case is replaced by the serum left in the casein. The 35° Be. sodium hydrate solution is then added, this solution having been previously diluted with the quantity of water nec-

essary to produce, as indicated in the preceding example, a volume of 400 litres for each 100 kilos of casein, always referred to the dry weight of casein including the normal moisture content of 10%, the procedure thereafter is the same as that indicated in Example A.

In order to increase the volume of the solution while maintaining the viscosity constant the solution is diluted during the maturing action, for example by means of the milk serum obtained after the coagulation of the casein or by a solution of sodium lactate, or sodium formate or sodium bisulphite or soap.

Substances of the type of sulphides and xanthogenates which accelerate the subsequent coagulating properties of the casein may be added to the alkaline solution. A suitable accelerating agent is carbon sulphide or sodium sulphide, or sodium sulphohydrate. Soaps, glycerine and the like, previously treated with carbon sulphide and sodium hydrate, or cellulose xanthogenate can be employed.

The colloidal solution of casein obtained by the process according to the present invention is suitable for spinning in the manufacture of artificial textile fibres.

ANTONIO FERRETTI.