PRODUCTION OF ARTIFICIAL PROTEIN FIBERS


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5 Claims. (Cl. 18—34)

1. This invention relates to the production of artificial protein fibres.

In the production of fabrics, it is known to form the fabric from a mixture of fibres and subsequently to remove one of the constituent fibres, for example by destroying or dissolving out such fibre, in order to obtain fabrics having special characteristics. Thus, British patent specification No. 547,583 describes tufted yarns for incorporation in a pile fabric comprising yarns made from gelatin, alginites, nitrate cellulose, casein or other destructible fibre in admixture with other yarns made for example of cotton fibres; after the tufted yarn has been made into pile fabric the destructible yarn is removed to give a pile fabric having a chenille surface. British patent specification No. 550,528 describes a process for the manufacture of textile fabrics characterised by producing a fabric, at least in part, from yarn consisting wholly or partially of soluble alginic fibres and thereafter modifying the fabric by dissolving or gelatinising some or all of the alginic fibres.

It is the object of the present invention to produce a cheap, soluble, protein fibre having the requisite strength for use in textile processes.

According to the present invention a process for the production of protein fibres soluble in dilute alkaline solutions comprises extruding an alkaline protein solution into a coagulant bath containing one or more metal salts, such as sulphuric acid may also be included in the coagulant bath. The coagulant bath may be used at ordinary temperatures, namely about 15° to 25° centigrade, or may be used at elevated temperatures, for example at 35° to 40° centigrade.

The stretching according to the invention may be effected by means of stretching rollers, reels or godets. The stretching is preferably effected in the presence of a coagulant bath, for example by using rollers immersed in the coagulant bath or preferably by using rollers in a subsequent coagulating bath which may be the same as or different from the coagulant bath: the stretching may be effected outside the coagulant bath by leading the thread on withdrawal from the bath to stretching rollers by suitably placed guides. The coagulant baths in which stretching is effected are preferably heated for example to 40° to 70° centigrade; if desired two or more baths of increasing temperatures may be used. The stretching should be at least 50 per cent but is preferably much higher for example from 300—700 per cent.

In accordance with the present invention, the fibres may be partially hardened with chloral or the like before they are stretched and the hardening is then completed in a subsequent chloral or like bath. For example, chloral may be added to the coagulant bath, to the subsequent coagulant liquid baths in which the stretching takes place or to both these baths.

The treatment with chloral or similar halogen-substituted aldehydes according to the invention to render the stretched threads resistant to cold water may be effected by passing the threads through an aqueous bath of chloral or the like for example by means of rollers or thread advancing reels, or may be effected by allowing the fibers to accumulate in a relaxed condition within an aqueous bath containing chloral or the like. In this latter form of the invention, the treatment is preferably effected by the method described in U. S. Patent No. 2,383,358, which corresponds to British specification No. 565,011. The aqueous chloral or like bath preferably also contains one or more metal salts such as for example sodium sulphate, magnesium sulphate, aluminium acetate, calcium chloride or mixtures of such salts, to assist in the hardening of the thread. The thread may also be stretched further after the treatment with the halogen-substituted aldehyde. The chloral or the like hardening bath may also be heated if desired, for example from 40° to 70° centigrade.

The fibres obtained according to the present
invention generally have dry tenacities of from 1 to 2 grams per denier and are thus suitable for use either as warp or weft in the normal textile processes such as knitting and weaving. The fibres, however, when placed in weak alkaline solution such as a dilute ammonia-soap bath readily swell and become dispersed in the alkaline solution. This action normally takes place within a few minutes at room temperature and may be speeded up by warming the alkaline solution. The fibres are therefore particularly suitable for use as a soluble yarn in the production of effect textile materials; for example, the fibre may be used as a binder for twistless yarns or may be twisted with wool fibres to produce yarns for making fabrics which on removal of the protein fibre give worsted fabrics with very fine yarns normally incapable of being woven or knitted. When used in conjunction with wool fibres the protein fibres according to the invention are readily removed from the fabric by the normal scouring process.

The invention is illustrated by the following examples, in which the percentages are by weight; weights of sodium sulphate are based on the anhydrous salt, Na₂SO₄:

**Example 1**

A solution containing 30 per cent of peanut protein, 0.65 per cent of caustic soda and 0.33 per cent of sodium sulphide was extruded into a bath maintained at 30° centigrade and containing 500 grams of crystalline aluminium sulphate (Al₂(SO₄)₃·18H₂O) per litre. The extruded threads were withdrawn from the bath and stretched 600 per cent in a similar bath but heated to a temperature of 60° centigrade. The threads were then placed in the relaxed condition in a bath containing, in each litre, 150 grams of sodium sulphate, 450 grams of crystalline magnesium sulphate (MgSO₄·7H₂O), and 31 grams of chloral hydrate. The threads were kept in this bath, maintained at a temperature of 40° centigrade and a pH of 7.0 for 7 hours, and were then washed with water, stretched 200 per cent in water and dried under tension. The threads obtained had a dry tenacity of 1.6 grams per denier and were soluble in a bath containing in each litre 40 grams of ammonium carbonate (soda ash) and 10 grams of sodium stearate.

**Example 2**

Threads were extruded, stretched and treated exactly as described in Example 1 except that they were left in the chloral hydrate solution for only 2 hours at 30° centigrade. The finally dried threads were insoluble in a bath containing in each litre 10 grams of sodium carbonate (soda ash) and 10 grams of sodium oleate.

**Example 3**

A solution containing 20 per cent of peanut protein, 0.45 per cent of caustic soda and 0.33 per cent of sodium sulphide was extruded into a bath maintained at 30° centigrade and containing 500 grams of crystalline aluminium sulphate per litre. The extruded threads were withdrawn from the bath and stretched a total of 550 per cent by first stretching the threads 300 per cent in a bath at 40° centigrade containing in each litre, 500 grams of crystalline aluminium sulphate and 12 grams of chloral hydrate and then completing the stretching in a similar bath at 70° centigrade. The threads were then placed in the relaxed condition in a bath containing in each litre, 150 grams of anhydrous sodium sulphate, 450 grams of crystalline magnesium sulphate and 10 grams of chloral hydrate. The threads were kept in this bath, maintained at a temperature of 40° centigrade and a pH of 7.0 for 16 hours, and were then washed.

**Example 4**

The procedure described in Example 3 was repeated using a 20 per cent solution of lactic casein in 1.0 per cent aqueous caustic soda in place of the peanut protein solution. The product was a lactic casein fibre which was soluble in a bath containing in each litre 40 grams of ammonium and 10 grams of sodium stearate.

**Example 5**

The procedure described in Example 3 was repeated with the only exception that the peanut protein solution was extruded into a bath maintained at 38° centigrade and containing in each litre, 400 grams of sodium sulphate and 10 grams of sulphuric acid. The threads obtained were soluble in a bath containing in each litre 40 grams of ammonium and 10 grams of sodium stearate.

A similar coagulant bath may also be used in Example 4 to give a similar alkali-soluble lactic casein fibre.

**Example 6**

The procedure described in Example 3 was repeated with the only exception that the peanut protein solution was extruded into a bath maintained at 38° centigrade and containing in each litre, 400 grams of sodium sulphate, 450 grams of crystalline magnesium sulphate and 10 grams of sulphuric acid. The threads obtained were soluble in a bath containing in each litre 40 grams of ammonium and 10 grams of sodium stearate.

**Example 7**

The procedure described in Example 3 was repeated with the only exception that the peanut protein solution was extruded into a coagulant bath at 25° centigrade containing in each litre, 150 grams of sodium sulphate, 450 grams of crystalline magnesium sulphate and 10 grams of sulphuric acid. The threads obtained were soluble in a bath containing in each litre 40 grams of ammonium and 10 grams of sodium stearate.

**Example 8**

A solution containing 20 per cent of peanut protein, 0.45 per cent of caustic soda and 0.33 per cent of sodium sulphide was extruded into a bath maintained at 35° centigrade and containing in each litre, 150 grams of sodium sulphate, 450 grams of crystalline magnesium sulphate and 10 grams of sulphuric acid. The extruded threads were withdrawn from the bath and stretched 600 per cent by first stretching the threads 350 per cent in a bath at 40° centigrade containing in each litre, 500 grams of crystalline aluminium sulphate and 15 grams of chloral hydrate and then completing the stretching in a similar bath at 70° centigrade. The threads were then placed in the relaxed condition in a bath containing in each litre, 150 grams of anhydrous sodium sulphate, 450 grams of crystalline magnesium sulphate and 10 grams of chloral hydrate. The threads were kept in this bath, maintained at a temperature of 40° centigrade and a pH of 7.0 for 16 hours, and were then washed.
with water and dried. The threads obtained were soluble in a bath containing in each litre 40 grams of ammonia and 10 grams of sodium stearate.

**Example 9**

A solution containing 20 per cent of peanut protein, 0.45 per cent of caustic soda and 0.33 per cent of sodium sulphide was extruded into a bath maintained at 50° centigrade and containing in each litre, 150 grams of sodium sulphate, 450 grams of crystalline magnesium sulphate and 10 grams of sulphuric acid. The extruded threads were withdrawn from the bath and stretched 600 per cent by first stretching the threads 350 per cent in a bath at 40° centigrade containing in each litre, 500 grams of crystalline aluminium sulphate and 15 grams of chloral hydrate and then completing the stretching in a similar bath at 70° centigrade. The threads were then collected in cake form in a rapidly rotating centrifugal box. The threads were hardened further in cake form by circulating through the cakes a bath containing in each litre, 150 grams of sodium sulphate, 450 grams of crystalline magnesium sulphate and 10 grams of chloral hydrate. The threads were then washed with water, dried and rewound onto bobbins as twisted threads. The twisted threads were then stretched 100 per cent at 50° centigrade in an aqueous chloral solution containing 5 grams of chloral hydrate per litre. The threads were washed with acetone and dried under tension. The threads obtained were soluble in a bath containing in each litre 40 grams of ammonia and 10 grams of sodium stearate.

What I claim is:

1. A process for the production of protein fibres soluble in dilute alkaline solutions comprising extruding an alkaline protein solution into a coagulant bath containing at least one metal salt to coagulate the protein, stretching the resultant thread at least 50 per cent, rendering the stretched thread resistant to cold water by treatment with an aqueous bath containing chloral and drying the thus hardened thread, the entire process being effected in the absence of formaldehyde.

2. A process for the production of protein fibres soluble in dilute alkaline solutions comprising extruding an alkaline protein solution into a coagulant bath containing at least one metal salt to coagulate the protein, stretching the resultant thread at least 50 per cent, rendering the stretched thread resistant to cold water by treatment with an aqueous bath containing chloral and drying the thus hardened thread, the entire process being effected in the absence of formaldehyde.

3. A process for the production of protein fibres soluble in dilute alkaline solutions comprising extruding an alkaline protein solution into a coagulant bath containing at least one metal salt to coagulate the protein, stretching the resultant thread at least 50 per cent in the coagulant bath, rendering the stretched thread resistant to cold water by treatment with an aqueous bath containing chloral and drying the thus hardened thread, the entire process being effected in the absence of formaldehyde.

4. A process for the production of protein fibres soluble in dilute alkaline solutions comprising extruding an alkaline protein solution into a coagulant bath containing at least one metal salt to coagulate the protein, stretching the resultant thread at least 50 per cent in a subsequent bath containing at least one metal salt, rendering the stretched thread resistant to cold water by treatment with an aqueous bath containing chloral and drying the thus hardened thread, the entire process being effected in the absence of formaldehyde.

5. A process for the production of protein fibres soluble in dilute alkaline solutions comprising extruding an alkaline protein solution into a coagulant bath containing at least one metal salt to coagulate the protein, stretching the resultant thread from 300 to 700 per cent, rendering the stretched thread resistant to cold water by treatment with an aqueous bath containing chloral and drying the thus hardened thread, the entire process being effected in the absence of formaldehyde.

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No references cited.