PATENT SPECIFICATION (11) 1 588 647

1 588 647

(21) Application No. 46527/77 (22) Filed 8 Nov. 1977 (19)
(31) Convention Application No. 741173 (32) Filed 12 Nov. 1976 in
(33) United States of America (US)
(44) Complete Specification Published 29 Apr. 1981
(51) INT. CL.[3] C08J 5/00
C08L 1/24 5/04
(52) Index at Acceptance
C3M 163 200 XX
C3U 12B1B3A 13 2C2
C3Y B120 B127 B284 F200 G240 G315

(54) IMPROVEMENTS IN OR RELATING TO HIGH FLUID-HOLDING FIBRE

(71) We, AVTEX FIBERS INC., a corporation organised and existing under the laws of the State of New York, United States of America, of 580 East Swedesford Road, 9 Executive Mall, P.O. Box 880, Valley Forge, Pennsylvania 19482, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention is directed to alkaline alloy fibers having high fluid-holding capacity, to shaped articles comprising such fibers and a method of preparing them.

Known in the art are alloy fibers consisting of a matrix of regenerated cellulose and an additive imparting a fluid-holding capacity to these alloy fibers which is greater than that of conventional regenerated cellulose fibers. This advantage is at least partially offset by their higher manufacturing costs.

As employed throughout the description and claims, the terminology "alloy fibers" refers to cellulose fibers having an additive uniformly dispersed through their regenerated cellulose matrix. Similarly, "fluid-holding capacity" is a measure of liquid absorbed into the fibers of a mass of alloy fibers, together with the liquid retained within the interstices of such fiber mass.

Herein, the term "fluid-holding capacity" is used in the sense defined by the "Syngyna Test" in Specification No. 1,517,398 as published.

According to the first aspect of the present invention there is provided an alkaline alloy fibre comprising a regenerated cellulose matrix and dispersed therein from 5% to 30%, based on the weight of the cellulose, of an additive capable of increasing the fluid-holding capacity of the fibre, the proportion of the additive being such that the fluid-holding capacity of the fibre is at least 4.79 cc/g, at least the majority (by weight) of the additive consisting of alginic acid alkali metal salt(s). Advantageously, at least 90% by weight of the additive consists of alginic acid alkali metal salt(s), preferably the additive substantially consists of alginic acid alkali metal salt(s) and most preferably the sole additive is an alginic salt of an alkali metal.

According to the second aspect of the present invention there is provided a shaped article which comprises a compressed mass of the fibres in accordance with the first aspect of the present invention in staple form.

According to the third aspect of the present invention there is provided a method of preparing alkaline alloy fibre in accordance with the first aspect of the invention, wherein filament-forming viscose is mixed with the alkali metal alginic acid salt(s), the mixture is formed into fibres, the fibres are coagulated and regenerated, the lubricating finish, if desired, is applied to the fibres, and the fibres are dried in an alkaline state, the fibre tow being cut into staple form, if desired, before drying, to obtain alkaline alloy fibre. The fibre is preferably prepared by mixing an alkaline aqueous soltuion of an alkali metal alginate with a filament-forming viscose, shaping the mixture into fibers, coagulating and regenerating the shaped fibers, and thereafter drying the same. Viscose constitutes the major portion of the mixture and the shaped alloy fibers are coagulated and regenerated by known means, and preferably in an acid bath containing sulfuric acid and sodium sulfate. Zinc sulfate is often incorporated in the bath as well as other coagulation modifiers, as desired. No special finishes and/or drying procedures are required to render the alloy fibers in a form which can be carded without difficulty.

The viscose which is employed in making the alloy fibers is, desirably, of a composition as is used in making conventional regenerated cellulose fibers.

Suitable alkali metal salts of alginic acid give viscosities in the range of from 20 to 150 seconds and preferably in the range of from 40 to 80 seconds (measurement of viscosity by time for a 3 mm diameter steel ball to fall 20cm through the solution) in dilute NaOH or aqueous solutions of an alginate concentration of from 2 to 15%, preferably in the range of from 8 to 10 %. The pH of the solution is preferably about or above seven.

The alginate containing solution is incorporated directly into a viscose and is employed in relatively large quantities, advantageously ranging from 5% to 30%, preferably at least 10%, based upon the weight of the cellulose in the viscose. Fibers formed from a viscose containing less than about 5% of a useful alginate do not necessarily differ appreciably from conventional regenerated cellulose fibers in their fluid-holding capacity.

The aqueous solutions of alkali metal salts of alginic acid may be injected into the viscose as it is pumped to spinnerets for extrusion or, alternately, aqueous solutions of such alginates and viscose may be passed through a blender or homogenizer if it is necessary to secure a more uniform dispersion. After the spinning, coagulation and regeneration stages, the shaped continuous tow of filaments undergoes the usual processing, which may include stretching, if desired, and is then dried by conventional means. Generally, before drying, the continuous tow of filaments is cut into staple of a desired length.

In general, the resulting alloy fibers experience no bonding during drying, and can be subsequently carded with no difficulty by the manufacturer of absorbent articles incorporating such fibers. Alternatively, after coagulation and at least partial regeneration, the fibers are stretched, if desired, conventionally wet processed and treated with an aqueous lubricating finish composition. The fibers are then dried to an alkaline, cardable product. In the preferred method of this invention, the sodium alginate dispersed in viscose, during processing into fibers, is alternately in the alkaline state, the acid state and again the alkaline state. During passage of the modified viscose through the acidic coagulating and regenerating bath, the sodium alginate is acidified. In order to obtain alkaline fibers containing sodium alginate, as required by the invention, the wet gel fibers are made alkaline preferably in a bath preceding the finish bath or, if desired, in the finish bath.

Sodium salts of alginic acid, suitable for use in this invention, are commercially available from the Kelco Division of Merck & Co., under the designation "Keltex" alginates, sold in various grades as determined by viscosity and concentration in aqueous dispersions. The words "Kelco" and "Keltex" are Trade Marks.

EXAMPLE I

Using conventional rayon spinning equipment, an alkaline aqueous solution of sodium alginate was separately injected by a metering pump into a viscose stream during its passage through the blender, and the blend was thereafter extruded into a spinning bath.

Preparation of a 10% concentration of sodium alginate solution was as follows: 300 g. of sodium alginate (KELTEX) high viscosity technical grade "Kelco" alginate were added to 300 ml of 18% concentration of aqueous sodium hydroxide plus 90 ml of 30% concentration hydrogen peroixde. The peroxide serves to reduce molecular weight and viscosity. This system, after mixing with a spatula, was added to 2250 ml of water, while stirring, and mixed for two hours.

After overnight standing at room temperature, it was mixed again for two more hours. The ball-fall viscosity after 48 hours was 24 seconds. The polymer was added to the viscose and spunn by injection, as described above. During this stage, the blend was subjected to high mechanical shearing. The viscose composition was 9.0% cellulose, 6.0% sodium hydroxide and 32% carbon disulfide (based upon weight of cellulose). The viscose ball-fall viscosity was 61 and its common salt test was 6.6.

The mixtures of viscose and sodium alginate were extruded through a 980 hole spinneret into an aqueous spinning bath consisting of 7.5% by weight of sulfuric acid, 20% by weight of sodium sulfate, and 1.5% by weight of zinc sulfate. After passage through the spinning bath, the resulting continuous tow was washed with water, desulfurized with an aqueous solution of sodium hydrosulfide, and again washed with water. The still wet multifilament tow was cut into staple fibers and finished with a solution comprising 1% $NaHCO_3$, 0.5% "Span 20" sorbitan monolaurate surfactant and water. The fiber was then dried. The word "Span" is a Trade Mark.

The fluid-holding capacity of sample fibers, made with various approximate proportions (tabulated below) of cellulose and sodium alginate in the spinning solution, was determined by the following test procedure.

Two and one-half grams of the different fibers prepared as described above were separately made into vaginal tampons by the following procedure: The fibers were carded into webs, each having a length of about 15 cm and being of variable thickness and width. Each of these webs was individually rolled in the direction of its width to provide a 15 cm roll and a string was looped about the center thereof. Each such roll was then folded on itself at the string loop and drawn into a 12mm tube within which it was compressed by a clamp and pluger. After compression, the resulting tampons were removed, allowed to stand for a period of about 30 minutes during which the tampons recovered to a bulk density of about 0.4 g/cc. and were than evaluated for their capacity to hold water by the *Syngyna* test Method, as described by G.W. Rapp in a June 1958 publication of the Department of Research, Loyola University, Chicago, Illinois (and as described in Specification No. 1,517,398). The results of such test are set forth in the Table for fibers made with various approximate proportions (as tabulated in the Table of cellulose and sodium alginate in the spinning solution.

EXAMPLE II

An aqueous solution of sodium alginate (prepared from a granular form supplied by Kelco) was made by dissolving "Keltex" in water to give 3% solution. The solution was injected into viscose, whereby the spinning solution contained 11.1% sodium alginate, based on cellulose. The fibers produced were subsequently processed in different ways and evaluated for fluid-holding capacity.

A portion of the resulting rayon tow was treated with 1% aqueous Na_2CO_3 and 1% "Span 20" in one solution, and dried. Sample staple fibers were carded, or otherwise well opened, and then conditioned at 24°C and 58% relative humidity. Two grams of such alloy fibers were placed in a 25 mm diameter die, pressed to a thickness of 3.2mm, and maintained in this condition for one minute. This compressed pellet of fibers was removed from the die and placed on a porous plate of a Buchner funnel. The upper surface of the pellet was then engaged with a plunger which was mounted for free verticel movement, the plunger having a diameter of 25mm and a weight of 1.1kg.

The funnel stem was connected by a flexible hose to a dropping bottle from which water was introduced into the funnel to wet the pellet of fibers. Control over the water flow was exercised by the position of the dropping bottle. After an immersion period of two minutes, the water was permitted to drain from the fiber pellet for three minutes, after which the still wet pellet was removed from the funnel and weighed. One-half of the weight of water in the sample pellet is a measure of the fluid-holding capacity of the fibers, expressed in cc/g. This measurement is defined as the potential ratio.

A blend of equal parts of this sample fiber with a three denier crimped rayon (U.S. Specification No. 3.046,983) had a potential ratio for 4.28 cc/g. The rayon control sample (no alginate loading) had a potential ratio of 2.7 cc/g.

The aqueous alkaline lubricating finish is often a bath containing an aqueous solution of sodium carbonate and sorbitan monolaurate; however, other alkaline agents and lubricating agents may be employed as taught in the art for ordinary rayon yarn. Alternatively, good control of product and process is maintained by separating the alkalizing and finishing treatment steps.

Some examples of finishes for cellulose fibers include partial higher fatty acid esters of sorbitan or mannitan and their polyoxyethylene derivatives, sodium oleate and oleic acid. Some examples of alkaline agents for alkalizing the fibers include dibasic ammonium phosphate, dibasic sodium phosphate, tribasic sodium phosphate, and sodium tetraborate.

The alloy fibers are adapted for use in a variety of articles, such as sanitary napkins and tampons, in which high fluid retention is an essential characteristic. In the manufacture of such articles, the alloy fibers necessitate no special techniques or equipment and they may be blended with other fibers which may or may not enhance the absorbent properties of the resulting articles.

TABLE

SAMPLE	*CELLULOSE*	*SODIUM ALGINATE*	*FLUID-HOLDING CAPACITY cc/g*
A	100	0	4.0
B	90	10	4.79
C	80	20	5.34
D	70	30	4.92
E	60	40	4.51

Reference is directed to British Patent Application No. 46127/75 (Serial No. 1,517,398) and no claim is made herein to matter claimed in Claims 1, 14 and 27 thereof.

Subject to the foregoing disclaimer,

WHAT WE CLAIM IS:-

1. Alkaline alloy fibre comprising a regenerated cellulose matrix and dispersed therein from 5% to 30%, based on the weight of the cellulose, of an additive capable of increasing the fluid-holding capacity of the fibre, the proportion of the additive being such that the fluid-holding capacity of the fibre is at least 4.79 cc/g, at least the majority by weight of the additive consisting of alginic acid alkali metal salt(s).

2. Alkaline alloy fibre comprising a regenerated cellulose matrix and dispersed therein from 5% to 30%, based on the weight of the cellulose, of an additive capable of increasing the fluid-holding capacity of the fibre, the proportion of the additive being such that the fluid-holding capacity of the fibre is at least 4.79 cc/g, at least 90% by weight the additive consisting of alginic acid alkali metal salt(s).

3. Alkaline alloy fibre comprising a regenerated cellulose matrix and dispersed therein from 5% to 30%, based on the weight of the cellulose, of an additive capable of increasing the fluid-holding capacity of the fibre, the proportion of the additive being such that the fluid-holding capacity of the fibre is at least 4.79 cc/g, the additive substantially consisting of alginic acid alkali metal salt(s).

4. Alkaline alloy fibre according to Claim 1, 2 or 3, which has a lubricating finish for cellulose thereon.

5. Alkaline alloy fibre comprising a regenerated cellulose matrix and dispersed therein from 5% to 30%, based on the weight of the cellulose, of an additive capable of increasing the fluid-holding capacity of the fibre, the proportion of the additive being such that the fluid-holding capacity of the fibre is at least 4.79 cc/g, the fibre preferably having a lubricating finish for cellulose thereon, the sole additive being an alginic acid salt of an alkali metal.

6. Alkaline alloy fibre according to any one of the preceding claims, wherein the alkali metal of the salt(s) is sodium and the salt(s) is/are present in an amount of at least 10% based on the weight of the cellulose.

7. Alkaline alloy fibre in accordance with Claim 5 and substantially as described in Example I.

8. Alkaline alloy fibre, in accordance with Claim 5 and substantially as described in Example II.

9. A shaped article which comprises a compressed mass of the fibres of any one of the preceding claims in staple form.

10. A shaped article according to Claim 9, which comprises a pad.

11. A shaped article according to Claim 9, which comprises a vaginal tampon.

12. A method of preparing alkaline alloy fibre in accordance with any one of Claims 1 to 6, wherein filament-forming viscose is mixed with the alkali metal alginic acid salt(s), the mixture is formed into fibres, the fibres are coagulated and regenerated, the lubricating finish, if desired, is applied to the fibres, and the fibres are dried in an alkaline state, the fibre tow being cut into staple form, if desired, before drying, to obtain alkaline alloy fibre in accordance with any one of claims 1 to 6.

13. A method according to claim 12, wherein an alkaline aqueous solution of an alkali metal alginate is mixed with a filament-forming viscose, the alginate being used in an amount in the range of from 5 to 30% based on the weight of cellulose in the viscose.

14. A method of preparing alkaline alloy fibre, in accordance with Claim 12 and substantially as described in Example I.

15. A method of preparing alkaline alloy fibre, in accordance with claim 12 and substantially as described in Example II.

16. Alkaline alloy fibre whenever prepared by the method of Claim 12, 13, 14 or 15.

FORRESTER, KETLY & CO.
Chartered Patent Agents,
Forrester House,
52 Bounds Green Road,
London N11 2EY
also
Rutland House,
148 Edmund Street.
Birmingham B3 2LD
and
Scottish Provident Building
29 St. Vincent Place,
Glasgow G1 2DT
Agents for the Applicants

Printed for Her Majesty's Stationery Office, by Croydon Printing Company Limited, Croydon, Surrey, 1981.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.