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3,796,629

SELF-BONDING RAYON

James E. McMaster, Secane, Pa., assignor to
FMC Corporation, Philadelphia, Pa.

No Drawing. Filed Oct. 15, 1970, Ser. No. 81,178

Int. Cl. D21h 1/30

U.S. Cl. 162-157 C

6 Claims

ABSTRACT OF THE DISCLOSURE

A non-woven web is prepared from viscose comprising 1 to about 6.5% cellulose, from about 0.6 to about 5% sodium hydroxide based on the weight of viscose, and from about 27-40% carbon disulfide based on the weight of cellulose. The said viscose is extruded into an aqueous bath containing 9 to about 25% sulfuric acid, from 0 to about 0.15% zinc sulfate and from about 0.3 to about 23% sodium sulfate to form filaments. The said filaments are stretched from 0 to 30% of their spun length, cut into staple fibers while in the wet gel state and formed into a non-woven web.

Various methods have been proposed for the manufacture of self-bonding regenerated cellulose fibers which may be used in the manufacture of paper and other non-wovens. One method involves the preparation of flat hollow filaments of regenerated cellulose wherein a gas is incorporated in the spinning solution using special techniques, such as described in U.S. Pat. 3,156,605.

Another method involves the partial derivatization of the cellulose to provide a fiber product having a greater swelling capacity and consequently better bonding properties such as disclosed in U.S. Pat. 3,525,735.

Still another method involves a special and expensive viscose process wherein a viscose of very high viscosity is spun into a low acid bath to obtain a highly swollen incompletely regenerated fibrous product such as disclosed in U.S. Pat. 3,320,117.

It is an object of the present invention to provide a method of producing a non-woven from self-bonding regenerated cellulose filaments and fibers which method employs viscose preparation and spinning steps.

This and other objects are accomplished in accordance with this invention which comprises extruding filaments of viscose of from 1 to 6.5 cellulose, from about 0.6 to about 5% sodium hydroxide, said percents based on the weight of the viscose, and from about 27 to 40% carbon disulfide, based on the weight of the cellulose, into an aqueous bath containing from 9 to about 25% by weight of sulfuric acid, from 0 to about 0.15% zinc sulfate and from about 0.3 to about 23% by weight of sodium sulfate to coagulate and regenerate said filaments, stretching said filaments from 0 to 30% of their spun length, cutting the filaments into staple fiber while in the wet gel state and forming a non-woven web from the wet-gel filaments.

The viscose employed herein preferably contains from 2 to 4% cellulose based on the weight of the viscose. As the cellulose content of the viscose is decreased, the strength of non-woven sheets made from fibers prepared from the viscose increases. However, spinnability of the viscose becomes more difficult as the cellulose content is decreased. The lower cellulose content viscose is readily made from higher cellulose containing viscose by dilution, for example, by injecting water into the viscose at the spinning machine. The required viscose may also be specifically prepared, if desired.

In general, the viscose used has a D.P. (degree of polymerization) of from 250 to 900, a ball fall viscosity of about 1 to 300 seconds, and a common salt test index of about 3 to about 11. The higher D.P. celluloses provide fibers which in turn produce higher strength papers or

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non-woven webs. The cellulose preferably has a D.P. of at least 400 to provide optimum bonding strength.

The viscose can also include agents which will provide higher strength non-wovens, e.g., starch, sodium silicate and wet strength agents including, for example, a polyamide-epichlorohydrin resin as described in U.S. Pat. 2,926,116. Such agents are usually incorporated in the viscose during the mixing stage in amounts ranging from 1 to 100% based on the weight of the viscose. The viscose is conventionally extruded from a multi-hole spinneret to form fibers consisting of a plurality of filaments whose cross-sectional shape is not critical.

The aqueous spinning bath for this invention must not contain any appreciable amount of zinc salt. The zinc salt, e.g., zinc sulfate content, is preferably below 0.1%. Sulfuric acid and sodium sulfate concentrations are not critical as long as the acid content is at least 9%. If a starch additive has been added to the viscose, somewhat higher acid (2-4% higher) should be used.

Immersion distance of the filaments in the spin bath is not particularly critical, a distance of from 7 to 40 inches is a suitable range for practical reasons.

The spinning speed of the filaments as they leave the spinning machine is preferably between about 30 and 50 meters per minute based on the somewhat higher strength of non-wovens produced at this speed, however, spinning speeds of from 10 to 70 meters per minute can be used quite successfully.

From the spin bath, the fibers are advantageously run through a cold water bath which can contain a small amount of sulfuric acid and sodium sulfate although this is not necessary. The temperature of the bath is preferably kept at room temperature or below as higher temperatures adversely affect self-bonding properties of the yarn.

No stretch or low stretch for the filaments is preferred as stretching tends to collapse the open structure of the swollen filaments and thus reduce their bonding properties.

The dry denier of the individual filaments of this invention generally ranges from about 0.5 to about 3 with a range of from about 1 to about 2 being preferred for the production of higher strength non-wovens. Bundles of fibers are conventionally produced from these filaments having a total denier ranging from about 100,000 to about 1,000,000. The fibers are usually cut while still in the wet-gel state into short staple fibers of from about $\frac{3}{16}$ " to 1 inch long, for the purpose of making paper-like non-wovens.

These fibers permit the production of a more uniform non-woven product using longer length staple fibers. They also produce non-wovens or papers having much higher dry elongation than is normally possible with wood pulp.

The fibers produced in accordance with this invention are highly swollen and must remain in the wet-gel state until they are formed into a nonwoven web. This may be conveniently accomplished by keeping wet skeins or staple slurries of the gel fiber in closed containers until it is needed for the manufacture of webs.

The term "wet-gel" as used herein designates the state of the wet fibers which have never been dried after manufacture.

Prior to or during the manufacture of the non-wovens, the wet-gel fibers can be impregnated with wet strength resins as described, for example, in U.S. Pat. 2,926,116, if desired.

Paper-like non-woven webs are prepared from the wet gel fibers in a conventional paper-making process. The fibers are dispersed to form a slurry and deposited on the screen of a paper-making machine. The formed sheet is pressed to obtain greater bonding at pressures ranging from at least 100 to about 800 p.s.i. and dried.

The sheets formed from the fibers of this invention have good dry strength and some wet strength. The papers are useful, for example, as covers or wrappers for sanitary napkins, diapers, etc., particularly of the disposable type.

The following examples of the preparation of the filaments and non-wovens of this invention are now set forth.

EXAMPLE I

A viscose of 3 wt. percent cellulose having a D.P. of about 400, 2 wt. percent sodium hydroxide and 30.5% carbon disulfide based on the weight of the cellulose was prepared and aged to a common salt test index of 8.4 and a ball fall viscosity of 2.5 sec. at 14° C. The viscose was spun by extruding it through a 300 hole spinneret into an aqueous bath containing 14 wt. percent sulfuric acid and 14 wt. percent sodium sulfate. The fiber traveled 15 inches through the bath at a speed of 53 meters per minute. From the spin bath, the fiber traveled through a cool water bath (15–25° C.) where it was stretched to 30% of its original length. The dry filament denier was 1.84.

The wet gel fiber was collected in skeins. The skeins were pressed to remove excess water, cut into one quarter inch lengths and dispersed in water in a Waring Blendor for 10 seconds. Sheets were then cast in a laboratory Noble and Wood sheet mold. The sheets were pressed at 400 p.s.i. between two layers of heavy cloth and dried on a photo dryer using a layer of cloth over the top of the sheets to slow the drying rate.

Physical properties of the sheets were determined by standard TAPPI test procedures including T410 M–45 and T220 M–60. The paper-type product produced as above had the following measured properties:

Breaking length, meters (BLM), dry	2071
Ream weight	13.3
Dry elongation, percent	2.69
Fiber strength, g./d.	1.20

EXAMPLE II

A viscose of 9 wt. percent cellulose having a D.P. of about 620, 6 wt. percent sodium hydroxide, 30.5% carbon disulfide based on the weight of the cellulose, and 1.5 wt. percent of an ethylene oxide (avg. 15 units) modified phenol was prepared and aged as in Example I. The viscose was pumped to the spinning machine and enough water injected therein to produce a spinning solution containing 3% cellulose and 2% sodium hydroxide. The yarn was spun as described in Example I except that a speed of 40 m./m. was maintained and the yarn was not stretched.

The following properties of sheet were measured:

BLM, dry	2868
BLM, wet	240
Ream weight	44.0
Dry elongation, percent	6.56

This same procedure was modified only to the extent of stretching the fiber 25% of its original length in the second bath. The properties of the sheet were as follows:

BLM, dry	2256
BLM, wet	146
Ream weight	39.6
Dry elongation, percent	5.81

EXAMPLE III

A procedure similar to Example II was carried out except that 10% of starch (Amazo 100—Amer. Maize Prod. Co.) based on the weight of cellulose was incorporated in the viscose during its preparation. The yarn traveled at 40 m./m. and was stretched 1%.

The following properties of the sheet were measured:

BLM, dry	3189
BLM, wet	155
Ream weight	45.4
Dry elongation, percent	7.58

This same procedure was again employed except that the spin bath consisted of 18% sulfuric acid, 0.1% zinc sulfate and 14.6% sodium sulfate. The fiber was not stretched.

The following properties of the sheet were measured:

BLM, dry	4366
BLM, wet	259
Ream weight	34.0
Dry elongation, percent	6.01

EXAMPLE IV

A viscose of 9 wt. percent cellulose having a D.P. of about 400, 6.2 wt. percent sodium hydroxide and 30.5% carbon disulfide based on the weight of the cellulose was prepared and aged to a common salt test index of 6.7. This viscose was pumped to the spinning machine and enough water injected therein to produce a spinning solution of 4.4% cellulose and 3.0% sodium hydroxide and a ball fall of 2 at 14° C. The viscose was spun by extruding it through a 6500 hole spinneret into an aqueous bath containing 14.8 wt. percent sulfuric acid and 20.5 wt. percent sodium sulfate. The fiber traveled 15 inches through the spin bath at a speed of 40 meters per minute and then through a cool water bath where it received no stretch. The dry filament denier was 1.5.

The wet gel fiber was made into a paper-like sheet as described in Example I.

The physical properties of the sheet were as follows:

BLM, dry	2327
BLM, wet	262
Ream weight	40

The above procedure was carried out a second time except that water was injected into the original viscose to provide a spinning solution of 6.3 wt. percent cellulose and 4.3 wt. percent sodium hydroxide.

The physical properties of the sheet were as follows:

BLM, dry	1893
BLM, wet	¹ 150
Ream weight	40

¹ Interpolated.

The above procedure was carried out a third time except that water was injected into the original viscose to provide a spinning solution of 7.2 wt. percent cellulose and 5.0 wt. percent sodium hydroxide. This again shows that increase of the cellulose content of the viscose is undesirable as the strength of the non-woven is substantially decreased.

The physical properties of the sheet were as follows:

BLM, dry	1695
BLM, wet	80
Ream weight	40

While the paper-like non-woven sheets produced in accordance with this invention do not have as high strength as those produced in the expensive, high viscosity viscose systems as shown in U.S. Pat. 3,320,117 or the derivatized viscose systems as shown in U.S. Pat. 3,525,735, they are sufficiently strong for use as covers in such products as disposable diapers and sanitary napkins. They are inexpensively produced and have strength about equal to those produced from hollow cellulose filaments such as shown in U.S. Pat. 3,156,605.

The filaments of this invention can be blended with regular rayon to make non-woven webs, however, as the percent of regular rayon in the blend increases, the strength of the web decreases.

I claim:

1. A method of preparing a non-woven from self-bonding regenerated cellulose filaments which comprises ex-

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truding filaments of viscose comprising from about 1 to about 6.5% cellulose, from about 0.6 to about 5% sodium hydroxide, said percent based on the weight of the viscose, and from about 27 to 40% carbon disulfide, based on the weight of the cellulose, into an aqueous bath containing from 9 to about 25% by weight of sulfuric acid, from 0 to about 0.15% by weight zinc sulfate and from about 0.3 to about 23% by weight of sodium sulfate to coagulate and regenerate said filaments, stretching said filaments from 0 to 30% of their spun length, cutting the filaments into staple fiber while in the wet gel state and forming a non-woven web from the wet-gel filaments.

2. The method of claim 1 wherein the viscose contains from 2 to 4% cellulose having a degree of polymerization of at least 400.

3. The method of claim 1 wherein the said viscose is mixed with from 1 to 100% starch, based on the weight of the viscose.

4. The method of claim 1 wherein said viscose is mixed with from 1 to 100% sodium silicate, based on the weight of the viscose.

5. The method of claim 1 wherein said viscose is mixed with from 1 to 100% of a polyamide-epichlorohydrin resin wet strength agent, based on the weight of the viscose.

6. The method of claim 1 wherein a pressure of at least 100 up to about 800 p.s.i. is applied to the non-woven web during its formation.

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S. LEON BASHORE, Primary Examiner

P. CHIN, Assistant Examiner

U.S. Cl. X.R.

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