

1

3,101,294 PROCESS FOR FORMING A WEB OF SYNTHETIC FIBERS

Anton Fridrich Fridrichsen, Kennett Square, Pa., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware
No Drawing. Filed Dec. 17, 1959, Ser. No. 860,110
5 Claims. (Cl. 162-146)

This invention relates to non-woven structures and more specifically to a process for treating non-woven structures comprising synthetic staple fibrous material.

Non-woven sheet materials from naturally occurring fibers are well known. They are widely used in the form of paper, wool felts and the like. With the development of synthetic fiber materials, there has been much study given to ways in which synthetic fibers could be formed into non-woven sheets and sheet-like structures to take advantage of the simple processing steps which are employed in the manufacture of non-woven fabrics from naturally occurring fibers while retaining at the same time the advantages of high strength, high abrasion resistance, and indifference to water which characterize many of the synthetic fiber materials.

One of the main difficulties in the utilization of synthetic fibrous materials in non-woven structures has been due to poor self-bonding characteristics of such fibers. Thus in the past production of such structures required the use of thermoplastic resins as bonding materials, and solvent bonding techniques. More recently the use of fibrils for bonding synthetic fiber materials has been taught. Fibrils are a form of synthetic fibrous material described more fully in Belgian Patent 564,026.

The term "fibril" is employed herein to designate a non-rigid, wholly synthetic polymeric particle capable of forming paper-like structures upon a paper-making machine. Thus to be designated a "fibril," a particle must possess (a) an ability to form a waterleaf having a couched wet tenacity of at least about 0.002 gram per denier when a plurality of the said particles is deposited from a liquid suspension upon a screen, which waterleaf, when dried at a temperature below about 50° C., has a dry tenacity at least equal to its couched wet tenacity and (b) an ability, when a plurality of the said particles is deposited concomitantly with staple fibers from a liquid suspension upon a screen, to bond a substantial weight of the said fibers by physical entwinement of the said particles with the said fibers to give a composite waterleaf with a wet tenacity of at least about 0.002 gram per denier. In addition, fibril particles have a Canadian freeness number between 90 and 790 and a high absorptive capacity for water, retaining at least 2.0 grams of water per gram of particle under a compression load of about 39 grams per square centimeter. Tests by which these measurements are made are detailed below.

Any normally solid wholly synthetic polymeric material may be employed in the production of fibrils. By "normally solid" is meant that the material is non-fluid under normal room conditions. By "... an ability to ... bond a substantial weight of ... (staple) fibers ..." is meant that at least 50% by weight of staple based on total staple and fibrils can be bonded from a concomitantly deposited mixture of staple and fibrils.

It is believed that the fibril characteristics recited above are a result of the combination of the morphology and non-rigid properties of the particle. The morphology is such that the particle is non-granular and has at least one dimension of minor magnitude relative to its largest dimension, i.e., the fibril particle is fiber-like or film-like. Usually, in any mass of fibrils, the individual

2

fibril particles are not identical in shape and may include both fiber-like and film-like structures. The non-rigid characteristic of the fibril, which renders it extremely "supple" in liquid suspension and which permits the physical entwinement described above, is presumably due to the presence of the "minor" dimension. Expressing this dimension in terms of denier, as determined in accordance with the fiber coarseness test described in Tappi 41, 175A-7A, No. 6 (June 1958), fibrils have a denier no greater than about 15.

Complete dimensions and ranges of dimensions of such heterogeneous and odd-shaped structures are difficult to express. Even screening classifications are not always completely satisfactory to define limitations upon size since at times the individual particles become entangled with one another or wrap around the wire meshes of the screen and thereby fail to pass through the screen. Such behavior is encountered particularly in the case of fibrils made from soft (i.e., initial modulus below 0.9) polymers. As a general rule however, fibril particles, when classified according to the Clark Classification Test (Tappi 33, 294-8, No. 6 (June 1950)) are retained to the extent of not over 10% on a 10-mesh screen, and retained to the extent of at least 90% on a 200-mesh screen.

Fibril particles are usually frizzled, have a high specific surface area, and as indicated, a high absorptive capacity for water.

Preferred fibrils are those the waterleaves of which when dried for a period of 12 hours at a temperature below the stick temperature of the polymer from which they are made (i.e., the minimum temperature at which a sample of the polymer leaves a wet molten trail as it is stroked with a moderate pressure across the smooth surface of a heated block) have a tenacity of at least 0.005 per denier.

By the use of fibrils it is possible to prepare non-woven fabrics from synthetic fiber staple material and fibril binding agents which have a high as-formed strength. However, for development of a maximum strength and many other properties it has been desirable, as the already-mentioned Belgian patent indicates, to calender or press the composite sheet sufficiently to fuse or coalesce the fibrils and thereby increase the bonding action which they exert upon the staple fibers. In general, for the strongest sheets it has been found useful to form such a sheet, then dry it, as by vacuum or pressure means, and then to subject the composite sheet to a high temperature bonding treatment combined with pressure. In such treatment, the temperatures involved are usually in the range of 190° C. to 220° C. or higher. Such temperatures fuse the fibrils and under proper conditions only minor damage is done to the staple fibers.

It is an object of this invention to provide a simplified and improved procedure for bonding of non-woven fibril-bonded fibrous sheets from synthetic fiber materials and in particular to provide a means of bonding such sheets employing substantially lower temperatures whereby there is no damage to the staple fiber material and whereby there is accordingly obtained a stronger and more uniform sheet. Other objects will become apparent from the description which follows.

These objects are achieved in a process which consists of warm-pressing a wet composite sheet of synthetic fiber staple and fibrils at a temperature at least about 20° C. below the melting point of the lowest melting polymeric material in the sheet, and below about 160° C., and preferably at a temperature between 105° C. and 140° C., the sheet containing, at the start of the warm pressing treatment, at least about 10% by weight of water. As a result of such processing there is obtained a non-woven sheet consisting essentially of fibrous materials; that is,

the calendering process, employing a temperature below 160° C., does not fuse the binder material completely. In particular, while temperatures in the range of 140° C. to 160° C. are useful, it is particularly desirable to use temperatures below 140° C., since in this range the above-indicated processing conditions result in spot-fusion of the fiber materials throughout the structure without destructive coalescence of the fibrous form of the material. There is thus provided a uniform bonded, fibrous sheet of high-melting synthetic polymer material in which the bonds are provided by fibrils heated sufficiently to coalesce them without completely destroying their fibrous form.

The surprising feature of the present invention is that by the use of a modest amount of water it is possible to obtain a uniformly and completely bonded material while employing mild temperatures below the melting point of both the staple fiber material and the fibrils comprising the sheet.

The following examples illustrate the principles and practice involved in the present invention without indicating any limitations thereto.

Example 1

A solution is prepared by adding 15 parts of 20/80 6-6/6 nylon to 85 parts of N,N-dimethylformamide and heating to 130° C. Sufficient zinc stearate is added so that it represents 20% of the total weight of solids. Fibrils are prepared from solution by adding it to a 70/30 water/N,N-dimethylformamide mixture at 27° C. in a 5-qt. Waring Blendor operating at approximately 7,000 r.p.m. The fine, uniform fibrils obtained are filtered and washed free of solvent.

A water slurry of nylon staple fiber, 1/4" in length, 1.5 denier per filament is prepared by adding one part of the staple fiber to 10,000 parts of water. This slurry is agitated gently and to this is added sufficient fibrils to provide a slurry containing 30% of the fibrils prepared as described above. The slurry is agitated for several minutes and then is poured into a 10" by 20" sheet mold onto a screen and permitted to drain. The unpressed paper obtained therefrom is dried at 105° C. to a water content of less than 5% in an oven. It is then calendered at 210° C. This treatment causes discoloration and subsequent degradation of paper properties. However, when the experiment is repeated using the same components but employing the process conditions of the present invention, much improvement in appearance of the sheet is noted. Specifically, the hand sheet after formation, containing 100% water by weight, is passed through the nip of a set of calendar rolls at 115° C. with a nip pressure of 140 lbs./in. The speed of the sheet through the nip is approximately 30 ft./min. With this treatment the sheet is obtained in a dry form with an appearance much better than the sheet obtained in the oven drying technique described above and equal to or better than sheets processed by calendering at 190° C. with 1500 lbs./in. or higher nip pressure. In particular, no discoloration was observed. Further, the warm wet calendering treatment gave a paper which did not adhere to either of the rolls of the calendar.

Example 2

In another paper preparation following the technique of Example 1, fibrils prepared from a copolyamide of 20% poly(hexamethylene adipamide) and 80% polycapraamide are employed in the preparation of a nylon paper using staple fiber of 66 nylon, 3 denier per filament, 1/4' long. The slurry is prepared with 90% staple fibers and 10% of the fibril binder. Water is removed to a 50% water content from the sheet formed. The calendar is operated at a temperature of 125° C. under a pressure of 140 pounds per inch and at a speed of 30 feet per minute. The sheet formed has a high tensile strength and no discoloration or degradation of the polymer components.

Example 3

A synthetic fiber paper of polyamide fibrous materials is prepared containing 80% of 1/4", 3 denier per filament, staple fibers of poly(hexamethylene adipamide) and 20% of the copolyamide paper binder described in Example 1. From this mixture a 1.9 oz./sq. yd. paper is obtained. This paper is found to have a moisture content of about 80% after wet pressing. Portions of the paper are then calendered in three different treatments at increasing temperatures from 110° C. to 125° C. A constant nip pressure of 200 lbs./in. is employed, and the speed is 10 ft./min. At the three different temperatures, there is no noticeable difference in paper properties. In each case the paper is still moist after calendering. The average tensile strength as determined on an Instron tester is 9.0 lbs./in./oz./sq. yd. in the machine direction and 10.1 lbs./in./oz./sq. yd. in the cross direction. Microscopic examination shows that the fibers are visually unharmed and that while the binders are fused, their fibrous identity is not destroyed. Pressing of the dried sheet in a flattened press at 190° C. and 625 lbs./sq. in. gives a sheet with a slightly increased tensile strength, but the fibrils are completely fused in the process and lose their fibrous identity.

In a comparative control the couched wet sheet is dried at 105° C. to a moisture content below 5% and thereafter calendered at 115° C. at 200 lbs./in. and 10 ft./min. The average tensile strength in both directions is very low and estimated to be below about 2 lbs./in./oz./sq. yd. Microscopic examination discloses there is no fusion of the binder whatever.

Example 4

30 grams of a solution containing 10% by weight solids of ethylene terephthalate/ethylene isophthalate copolymer (80/20) in trifluoroacetic acid is added in an even stream to a mixture of 350 ml. of glycerol and 50 ml. of water in a Waring Blendor at approximately 14,000 r.p.m. The fibrils obtained are filtered and washed with water until free of organic liquid.

In a similar experiment, a synthetic fiber paper is obtained from staple fibers consisting of "Dacron" polyester fiber, 3 denier per filament, 1/4" length and fibrils prepared as described above. The aqueous slurry of fibrous materials contains 78% of the staple fibers and 22% of the fibrils. From this slurry there is prepared a paper with a weight of 2.1 oz./sq. yd. This paper as formed is found to contain 60% by weight of water after couching and wet pressing. The sheet is calendered at 140° C., under a pressure of 250 lbs./in. and at a speed of 10 ft./min., giving a sheet with a tensile strength of 8.4 lbs./in./oz./sq. yd. in the machine direction and 10.2 lbs./in./oz./sq. yd. in the cross direction. As before, there is only partial fusion of the fibril materials resulting in a sheet with substantially a completely fibrous content.

The nature of the fibre employed in the sheet products of the present invention is not critical. It may be a natural fibre such as wood pulp, cotton, flax, wool or the like or a fibre prepared from a synthetic or man-made fiber may be used provided it is non-fusible at a temperature below about 160° C. Preferably the fibers comprising the sheet are prepared from polymers having a melting point above about 200° C. and a moisture regain of above about 1%. In a similar manner the chemical identity of the fibril is not critical in the process of the present invention. The fibrils may be prepared from any linear synthetic polymer. Preferably they are obtained from a polymer which is water insoluble, has a melting point above about 180° C. and a second order transition temperature above about 0° C. Because of the commercial availability of the polymers and their wide range of versatility, fibrils produced from poly(hexamethylene adipamide), polycapraamide and copolymers thereof and polyesters from copolymers of polyethylene terephthalate and polyethylene isophthalate are particularly desirable.

5

The amount of pressure employed in the calendering operation will vary with the thickness of the waterleaf and the temperature employed in the warm pressing operation. In general the use of higher pressures permits the operation to be performed at lower temperatures as will be apparent to those skilled in the art. The processing of a sheet of high basis weight will in general require the use of a higher pressure and adjustment of calendering speed to permit thorough and even distribution of heat. Pressures as low as 5 lbs. per inch on the calender roll may be used for appropriate fibrids, particularly where the basis weight of the sheet is low. The use of pressures as high as 600 lbs. per inch is not unusual. Where static pressures are used a range of from about 10 lbs./sq. in. to about 1200 lbs./sq. in. in the pressing operation is recommended.

It is readily apparent from the foregoing that the present invention provides a simplified and economical process for the preparation of strong papers from synthetic fiber materials. It is already known that fibrids offer superior bonding properties and that their use permits the preparation of unfused and unbonded papers of adequate strength. However, for critical uses, it has heretofore been considered necessary even with fibrid binders, to heat synthetic fiber papers at a temperature sufficiently high to completely melt or fuse the fibrid binder particles. Now, however, by the practice of the present invention it is possible to prepare a synthetic fiber paper containing predominantly fibrous materials even after bonding. In the preferred range of temperatures, the process herein described provides papers which are equivalent in strength to completely fused papers but which still contain the fibrid-binding agent in a fibrous form. Moreover, the bonding calendering treatment required for the production of such materials makes much less stringent demands upon the machinery involved. Temperatures in the range preferred for the present invention, that is, 105° C. to 140° C., are much easier to obtain and much easier to control than the higher temperatures heretofore considered necessary. In addition, because of the low temperatures involved, there is no chance that the full strength of the staple fiber materials employed in the sheet can be impaired in any way. Thus, by the practice of the present invention superior sheets are obtained in a process which offers substantial advantages and improvements over those hitherto considered necessary.

6

Many equivalent modifications will be apparent to those skilled in the art from a reading of the above without a departure from the inventive concept.

What is claimed is:

1. In the process for forming a non-woven, fusion bonded, composite sheet from synthetic polymer staple fiber and fibrid binding agent, said polymer being selected from the group consisting of poly(hexamethylene adipamide), polycapraamide, polyethylene terephthalate, copolymers containing hexamethylene adipamide and caproamide units, and copolymers containing ethylene terephthalate and ethylene isophthalate units, by concomitantly depositing an aqueous slurry of a mixture of said staple fiber and at least 10% by weight of fibrid binding agent to form a sheet, drying, and thereafter heat calendering the sheet to increase the bonding action of the fibrid binding agent upon the staple fibers; the improvement for providing a stronger and more uniform sheet without destructive coalescence of the fibrous character of the fibrid binding agent and staple fibers wherein said calendering is conducted (a) upon said sheet while containing an initial amount of at least 10% by weight of water and (b) at a temperature at least 20° C. below the melting point of the lowest melting polymeric component in the sheet, and between about 105° C. and about 140° C., and at a nip pressure of at least 5 pounds per inch.

2. The process of claim 1 wherein the synthetic polymer forming the staple fiber is poly(hexamethylene adipamide).

3. The process of claim 1 wherein the synthetic polymer forming the staple fiber is polyethylene terephthalate.

4. The process of claim 1 wherein the synthetic polymer forming the fibrid binding agent is a copolymer containing hexamethylene adipamide and caproamide units.

5. The process of claim 1 wherein the synthetic polymer forming the staple fiber is a copolymer containing ethylene terephthalate and ethylene isophthalate units.

References Cited in the file of this patent

UNITED STATES PATENTS

2,999,788 Morgan ----- Sept. 12, 1961

FOREIGN PATENTS

572,962 Great Britain ----- Oct. 31, 1945