PROCESS EMPLOYING AQUEOUS MEDIA IN THE TREATMENT OF FIBROUS MATERIALS

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ABSTRACT

A method of treating fibrous materials to render them more amenable to subsequent textile and papermaking processes employing aqueous media which comprises: treating fibrous materials with a softening and swelling agent whereby the individual fibers become soft, limp and swollen; exposing said fibrous materials to textile or papermaking processes employing aqueous media wherein the individual fibers show less tendency toward undesirable fiber agglomeration, exhibit greater tendency toward fiber individualization, and demonstrate enhanced response and receptivity to fiber processing or manipulation because of their soft, limp and swollen nature; and forming a nonwoven fabric of the individual fibers. Also included are methods combining (1) the treating of the fibrous materials with the softening and swelling agent and (2) the textile processes into a single operation.

14 Claims, No Drawings
PROCESS EMPLOYING AQUEOUS MEDIA IN THE TREATMENT OF FIBROUS MATERIALS

This patent application is a continuation-in-part of co-pending, commonly assigned patent application Ser. No. 4,405, filed Jan. 20, 1970, now abandoned.

Many people have been engaged for many years in the manufacture of nonwoven textile fabrics which can be made without resorting to the spinning and twisting of fibers into yarns and strands, and the subsequent weaving, knitting or other fabrication of such yarns and strands into fabrics or cloth.

Such nonwoven fabrics have usually been manufactured by laying down one or more fibrous layers or webs of textile length fibers by textile carding techniques which generally align the majority of the fiber segments lengthwise of the fibrous web being laid down. During the subsequent processing of the fibrous web into a nonwoven fabric, one of the key steps in the operation is the treatment of the fibrous web with resin binders, usually in the form of aqueous emulsions or dispersions, to bond the individual fibers into a stronger and more coherent nonwoven fabric.

Such methods have been generally successful but it has been noted that some of the fibers used, particularly some synthetic fibers, have not been as strongly bonded to each other by the aqueous resin binder systems as is desired.

In some cases, it has been proposed that the carded fibrous webs be given a pretreatment under the influence of aqueous media prior to the bonding process whereby the individual fibers of the fibrous webs are rearranged, reorganized, or otherwise manipulated to form fibrous webs of improved properties and characteristics. Examples of such proposed aqueous media pretreatments are noted in the following issued patents: U.S. Pat. Nos. 2,862,251 (note FIGS. 7 and 23), 3,025,585 (note FIG. 1), 3,033,721 (note FIG. 11), 3,485,706 (note FIG. 2), and British Pat. No. 1,088,376 (note FIG. 2). As a result of such aqueous media pretreatments, it has been found that the subsequent aqueous resin binder treatment may sometimes be omitted.

However, again, it has been noted in some cases that some fibers, particularly some synthetic fibers, have not been completely amenable to and have not fully responded to the aqueous media rearranging, reorganizing or other fiber manipulating processes as much as would be desired.

It has also been proposed to suspend textile fibers in an air stream in a more or less individualized state, and then to filter the fibers out onto a collection screen to form an air-laid fibrous layer in which the individual fibers are arranged more or less at random. Subsequent to such random formation, the fibrous layer is usually treated with a resin binder, again usually in the form of an aqueous system, to bond the individual fibers into a stronger and more durable fabric.

In such cases, again, although such techniques have been relatively successful, it has always been desired to improve on them to obtain better fabric properties and characteristics, particularly in the area of improvements imparted by the aqueous media bonding process.

Efforts have also been directed to the possibility of laying down a substantially uniform fibrous layer of individualized fibers by papermaking techniques in aqueous media but such efforts have generally been relatively unsuccessful whenever textile length fibers are involved.

In efforts to use textile length fibers in the manufacture of fibrous webs by papermaking techniques in aqueous media, it has been proposed that paper formation aids or dispersing agents be added to the aqueous slurry of fibers at an early stage or at an intermediate stage, such as the furnish in the stuff box, or perhaps at the fan pump of head box, immediately prior to delivery of the aqueous fiber slurry to the papermaking machine proper, whether it be a Fourdrinier of a cylinder machine.

Attempts to use textile length fibers have been mildly successful and, as a result, textile length fibers of from about 1½ to about 3 denier having a length of about one-fourth inch and even ranging up to about three-eighths inch have been used and moderately well-formed fibrous webs have been obtained.

Another related effort in this general area is described in co-pending, commonly-assigned patent application Ser. No. 810,573, filed Mar. 26, 1969, wherein the use of specific high molecular weight dispersants is described for papermaking techniques in aqueous media using fine denier rayon fibers of very long lengths.

It is therefore still desired that techniques be developed whereby fibers of greater textile length be used in the manufacture of acceptable fibrous webs by papermaking techniques in aqueous media. If such paper formation aids or dispersing agents are used in the aqueous media, it is highly desirable that techniques be developed whereby lesser amounts of these relatively expensive dispersant materials are required.

In all these prior art techniques, the use and influence of aqueous media are noted and it is a principal purpose of the present invention to pre-treat fibrous materials in such a way to render them more amenable to, and cause them to respond more fully to, subsequent textile processes in the aqueous media.

It has now been found that such desires and purposes, and other desires and purposes which will become clear hereinafter, can be accomplished by giving the fibrous materials a pretreatment with a swelling agent whereby the individual fibers become soft, limp and swollen, prior to exposing the fibrous materials to the textile processes employing aqueous media. It has been found that the individual fibers, while they are still soft, limp and swollen may show decreased fiber-to-fiber friction, possess less tendency toward undesirable fiber agglomeration, show a greater tendency toward fiber individualization, exhibit an enhanced response to fiber rearrangement and manipulation and, in general, are rendered more amenable and more responsive and receptive to the subsequent aqueous media treatment. As a result, the resulting nonwoven fabric possesses properties and characteristics which are improved over the properties and characteristics of nonwoven fabrics which have not been given pretreatments with swelling agents.

The present inventive concept is particularly applicable to fine denier textile length synthetic or man-made organic polymeric fibers. As used herein, the term "textile length" fibers is intended to include fibers having an average length of at least about one quarter inch and preferably greater, say, on the order of at least about one-half inch or three-fourths inch and up to about 2 inches or more. Such fibers, and especially
those longer than one-half inch, are often referred to in the textile industry as "cardable" fibers, indicating that they have sufficient length as to be used in carding processes on conventional textile carding machines.

Such textile length fibers are to be distinguished from "papermaking" fibers which are extremely short and are usually in the range of from about one-twentieth inch or less up to about one-sixth inch. Wood pulp fibers are, of course, the best known example of papermaking fibers. Such shorter fibers are not of textile length, are not cardable, but are still a valuable to some of the narrower aspects of the present invention.

Also of limited applicability to the principles of the present invention are the natural fibers such as the cellulose fibers including cotton, flax, jute, ramie, etc., and other natural fibers such as wool, silk, etc.

The textile length synthetic organic polymeric fibers may be selected from a large group of commercially known synthetic fibers such as the cellulosics (regenerated cellulose rayon, cellulose acetate, cellulose triacetate, etc.); polyamides (nylon 6/6, nylon 6, nylon 610, nylon 10, nylon 11, etc.); polyethylene terephthalate polyesters ("Dacron," "Kodel," etc.); acrylics ("Acri lan," "Orlon," etc.); modacrylates ("Dyneal," "Verel," etc.); polyolefins (polyethylene, polypropylene, etc.); and other synthetic fibers.

Any one of those fibers may be the only type of fiber present in the fibrous layer or web, or such fibers may be used in blends in any and all proportions.

The denier of these synthetic fibers is dependent upon the demands and requirements of the desired fibrous webs. As such, deniers in the range of from as low as about three-fourths or one denier up to as large as about 15 deniers or more are of value depending upon the needs of the particular circumstances. Within the more commercial aspects of the present invention, a denier range of from about 1 1/2 to about 3 is preferred.

The lengths of these synthetic fibers, as mentioned previously, are as low as about one-fourth inch and as long as about 2 inches, or longer, if desired or required. Such lengths are normally obtained by cutting or stapling synthetic continuous filament tow to the desired lengths.

The swelling agents which are used to render the fibers soft, limp and swollen may be selected from a wide variety of swelling agents known in industry which are capable of use in such concentrations and in such a way that they swell the fibers but do not dissolve, degrade, discolor, or decompose them. For example, swelling agents for rayon include a large number of water soluble chemical compounds: alkali metal hydroxides, such as sodium hydroxide, potassium hydroxide, lithium hydroxide, etc.; cuprammonium hydroxide; liquid ammonia; quaternary ammonium compounds such as tetramethyl ammonium chloride; tetra methyl benzyl amnonium chloride, etc.; strong acids such as phosphoric acid, sulfuric acid, nitric acid, etc.; strongly acidic salts such as zinc chloride; relatively neutral salts such as lithium chloride; etc.

Swelling agents for polyamides include solutions of phenol, diethyl formamide, dimethyl formamide, concentrated formic acid, xylenol, trifluoroethanol, zinc chloride, chloral hydrate, γ-butrolactone, etc.

Swelling agents for polyolefins include petroleum ether solutions of tetrahydrophthalene (tetralin), decahydropenthalene (decalin), carbon tetrachloride, toluene, xylene, etc.

Swelling agents for polyesters include hot phenolic compounds, chloral hydrate, n-methyl pyrrolidone, etc.

Swelling agents for acrylics include aqueous solutions of dimethyl formamide, diethyl formamide, dimethyl sulfoxide, etc.

Swelling agents for modacrylics include aqueous solutions of dimethyl formamide, diethyl formamide, diethyl sulfoxide, etc.

The concentration of the swelling agent, the duration of time and the temperature of the pretreatment may be varied relatively widely but it is essential that the conditions be sufficient to induce a soft, limp and swollen state in the fibers. Once such a soft, limp and swollen state is reached, additional pretreatment does not add materially to the efficacy of the subsequent fiber dispersion and web formation steps or other aqueous media processing and, in fact, may be undesirable inasmuch as the possibility of fiber discoloration, dissolution, degradation, or decomposition may arise. Also, it is possible that the swelling action could be too drastic in which case the fibers could become so swollen as to result in gelation at their surfaces whereby they could adhere to each other.

The concentration of the swelling agent and the other factors of the pretreatment must be such that merely a softening and swelling of the synthetic fibers is obtained and that no material solubilization, degradation, discoloration, or decomposition of the synthetic fibers take place. For example, in the case of rayon, concentrations of aqueous sodium hydroxide of from about 0.5 percent to about 8 percent are satisfactory, with a preferred range of from about 2.5 percent to about 7 percent.

In the case of nylon, phenol is the preferred swelling agent and concentrations of phenol in water in the range of from about 1 percent to about 5 percent are utilizable, with preferred ranges extending from about 2 percent to about 5 percent.

The temperatures of the swelling pretreatments are preferably room temperature for most synthetic fibers but may range in special cases up to elevated temperatures of 100°C. or more, where such temperatures may be desired or required.

The duration of the time of the swelling pretreatment will be varied from as short a period as a few seconds for some synthetic fibers up to about 3 or 5 minutes for other fibers. Continued pretreatment for longer periods, for example, up to about 100 or more minutes does not produce materially improved results. As a matter of fact, such extended periods may be undesirable inasmuch as the possibility of fiber dissolution, discoloration, degradation, or decomposition may arise.

Once the fibrous materials have been treated with the swelling agent and the individual fibers have become soft, limp and swollen, they retain such properties as long as they remain wet or damp. While they are in such a state, they respond quite readily and are very receptive to processing in aqueous media. Such a soft, limp and swollen physical condition naturally leads to a low fiber modulus and may also have a profound effect on the interaction between fiber surface and aqueous environment, which is not the case for fibers which have not been pretreated by the method of our invention.

The soft, limp and swollen nature of the fibers also permits a closer, more intimate and more frequent contacting relationship between the individual fibers of a
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web inasmuch as the fibers are not as resilient and springy and do not have as great a tendency to spring apart into less dense fibrous configuration in which the individual fibers have less intimate and fewer contacting relationships with each other. The existence of a greater number of contacts between the individual fibers is, of course, conducive to better bonding and adhering by aqueous resin binder dispersions.

Our invention is usually practiced by:

a. pretreating the fibers in a swelling agent;

b. removing the swelling agent by washing, dilution, or neutralization; and

c. subjecting the fibers to the appropriate papermaking or textile process in an aqueous medium.

It is not essential that the swelling agent physically be removed from the fibrous materials or be inactivated in all cases prior to the subsequent processing in aqueous media. If such processing takes place in a large excess of an aqueous medium, whereby the concentration of the swelling agent is diluted and falls off to extremely low or negligible values, then the swelling agent is permitted to remain in the fibrous materials in such diluted form. However, if the subsequent processing takes place in relatively small amounts of water, so that the concentration of the swelling agent would remain at a relatively high or objectionable value, then it must be removed by either a dilution or washing technique, or neutralized, or rendered inactive or inoperative as a swelling agent prior to the subsequent processing operation.

Unexpectedly, the effect of the swelling agent is not reversed by dilution, washing, neutralization, or the like, and the soft, limp and swollen nature of the fibrous materials remains substantially greater than fibers which have only been immersed in water, so long as the pretreated fibrous materials are not dried, but remain wet or damp. As a result, the fibrous materials remain amenable to and responsive or receptive to the subsequent aqueous media processing after the swelling agent has been removed. However, the fibers may not be allowed to dry or else they will lose their soft, limp and swollen nature and will return to their original physical characteristics and thus will no longer be responsive or receptive to the subsequent processing in aqueous media.

To illustrate the lack of necessity for the continued presence of the swelling agent, it can be pointed out, for example, that rayon fibers which have been treated with caustic may be neutralized and introduced into a paper sheet mold on the acid side, that is, a pH of less than 7 and the desirable properties and characteristics of the soft, limp and swollen fibers still remain.

It is also to be realized that, regardless of whether the swelling agent is diluted, washed out, or neutralized, such processing may leave small amounts of chemical agents remaining in the fibrous materials. Although these impurities usually do not interfere with the subsequent processing treatment, it is normally desired to remove them before the final finishing treatment and before the resulting products are prepared for sale or use. A final washing or rinsing may be in order prior to such sale or use.

The principles of the present inventive concept are applicable to a very wide range of processing in aqueous media. For example, some fibrous webs containing certain synthetic organic polymeric filaments or fibers are very difficult to bond particularly by aqueous emulsions, dispersions or latexes or resin binders. However, if such synthetic fibers are given a pretreatment with a swelling agent and become soft, limp and swollen, they respond very readily and can be bonded with aqueous binder resins to yield bonded nonwoven fabrics having significantly improved properties and characteristics, particularly tensile strengths, over the properties and characteristics of nonwoven fabrics not given such a swelling pretreatment.

In a similar way, it is frequently desired to make fibrous webs by papermaking techniques in aqueous media, particularly when using textile length fibers. A pretreatment with a swelling agent whereby the textile length fibers are rendered soft, limp and swollen produces improved responsiveness and receptivity to papermaking techniques. Specifically, the use of the swelling pretreatments of our invention permits the use of appreciably longer fine denier textile fibers in the denier range of from about 1½ to about 3 and having lengths of about one-half inch or even five-eighths inch, without using any paper formation aids or dispersing agents in the aqueous media.

Additionally, it can be shown that the use of the swelling pretreatments of the present invention provides for the dispersion of fine denier textile length rayon fibers of about three-fourths inch or more in aqueous media by the addition of much lower amounts of such paper formation aids or dispersing agents. Specifically, in many cases, it can be shown that the amounts of paper formation aids or dispersing agents required for the papermaking techniques can be reduced to as little as only one-twentieth of the previous requirements.

Also, it can be shown that the amount of beating and working of the textile length fibers required to insure a uniform dispersion can be drastically reduced. In some cases, it can be shown that the number of strokes required to insure a uniform dispersion of fibers in a sheet mold can be reduced from 50 strokes to as few as 5 strokes.

Additionally, it is noted that the individual soft, limp and swollen fibers show a greater tendency toward fiber individualization and a lesser tendency toward agglomeration and clumping into tangles and bundles of matted fibers in the papermaking process. And, if bundles were originally present, such as following a cutting or stapling operation, there is an increased tendency for such bundles to break up into individual fibers during the papermaking process in aqueous media whereby a more uniform fibrous web is obtained. This easier breakdown of the bundles into individual fibers is particularly noted in the case of rayon, nylon, polyester, acrylic and polyolefin fibers.

Still another field wherein vastly improved response and receptiveness to aqueous media processing is noted in the rearranging, reorganizing or manipulating of individual filaments subsequent to their original formation as a fibrous web. The soft, limp and swollen fibers are found to be more amenable to such manipulation.

The reason for such improved results is not fully or completely understood but it is believed to be related to the soft, limp and swollen condition of the individual fibers whereby they become more responsive and more receptive to the particular textile treatment taking place in the aqueous media. The greater response or receptivity is thus believed to be due to the changed physical condition of the individual fibers rather than...
to any chemical change. The soft, limp and swollen fibers have a low modulus and yield more readily to applied forces and also appear to possess some form of surface lubricity or slipperiness whereby they do not cling or stick to each other.

Of particular interest in the area of rearranging, reorganizing, or manipulating individual fibers subsequent to their original formation as a fibrous web are the processes and apparatus described and illustrated in the previously mentioned U. S. Pat. Nos. 3,025,585, 3,485,706, and the British Pat. No. 1,088,376.

These patents, along with U. S. Pat. Nos. 2,862,251, 3,033,721 and 3,081,515 disclose nonwoven fabrics having a patterned configuration wherein the fibers are positioned in bundles of fiber segments. The bundles of fiber segments define areas of lesser fiber density or holes in the resultant nonwoven fabric. The fiber bundles may be flat ribbons or they may have an oval or circular cross section or other cross-sectional configurations. In all instances, the fiber segments which make up a bundle, are in increased parallelism and in closer proximity to each other as compared to the fibers in the original starting web. The fiber bundles define areas of lesser fiber density or holes and the bundles are interconnected at junctures. The fiber portions in these junctures are positioned in a random configuration and may be highly entangled, depending on the specific conditions used in the rearranging process.

These patents disclose fluid techniques for rearranging, reorganizing, or manipulating individual fibers of fibrous webs by subjecting them, while being carried on a permeable backing or supporting member, to the action of fine columnar jets, streams, or sprays of a fluid such as water under pressures ranging from atmospheric pressure to 5,000 pounds per square inch gauge. As particularly described in the above-mentioned British patent, when high entanglement of fibers is desired in the final product, it is preferred commercially that columnar streams of liquid be used at pressures of at least 200 pounds per square inch gauge and preferably at least 500 pounds per square inch gauge. Such pressures are rather high and are generally undesirable economically for practical commercial use. Utilization of the principles of the present inventive concept, however, permit the successful commercial use of water pressures as low as about 30 pounds per square inch gauge, which is, of course, more economically advantageous. Lower pressures, even as low as atmospheric pressure, are utilisable but the resulting products are not as commercially satisfactory.

Water pressures up to about 1,000 pounds per square inch gauge or even 5,000 pounds per square inch gauge are, of course, of use in the present invention but such is undesirable, particularly for practical and economical operating reasons. Within the more practical commercial aspects of the present invention, a range of from about 50 pounds per square inch gauge to about 190 pounds per square inch gauge is preferable.

Greater pressures are, of course, of use for specific needs and requirements. For example, greater pressures are of value when very high production line speeds are desired or necessary and the fibrous materials are being fed through the treating apparatus at very high rates of speed. Processing with low pressures occasionally yields unsatisfactory results when production line speeds are too great.

Jets or streams of water having a substantially columnar shape are preferred in some of the applications of the principles of the present inventive concept inasmuch as they are easier to control, prevent undesirable overlapping of fluid rearranging effect, and minimize fluid and air disturbance and turbulence in the fibrous materials being reorganized and rearranged.

Jets, sprays, or streams having a conical or fan-shape, however, are of use, particularly where some degree of overlapping of fluid and air disturbance and turbulence can be tolerated or possibly desired under the circumstances and where the desired design or pattern in the rearranged fabric permits such use.

It is not essential that the jets be substantially parallel or that they be directed at right angles to the plane of the fibrous materials being rearranged. The jets may be at various angles to each other instead of being parallel and they may be at angles, such as 30° or 45° to the plane of the fibrous web. One particular configuration comprises a row of nozzles facing forwardly at an angle of 45° to the direction of travel of the fibrous materials with the succeeding adjacent row facing rearwardly at an angle of 135° to the direction of travel of the fibrous materials. Such an arrangement provides for an increase in the amount of turbulence and swirling of the rearranging fluid which is highly desirable in some applications of the present invention.

One desirable result of the permissible use of lesser water pressures by the present inventive concept is the corresponding lowering of the total energy or momentum required in achieving the same desired results which can be obtained by other techniques only through the expenditure of far greater energy or momentum.

Another desirable result of the permissible use of lesser water pressures of the present inventive concept is the making available of a larger range of nozzle or orifice sizes and shapes for use with a larger range of water pressures for achieving a greater variety of designs and patterns in the resulting product. In accordance with the present inventive concept, orifices are usually circular and possess sizes of from about 0.002 inch to about 0.069 inch which are of use, with a preferred commercial range of from about 0.003 inch to about 0.020 inch.

Additional designs and patterns are, of course, made possible by the use of a great variety of permeable backing or supporting members for carrying the fibrous web. Preferably, such supporting members are of the type of stainless steel, plastic, or similar screen-like member illustrated in FIG. 5 of U. S. Pat. No. 3,025,585, having a mesh size per inch ranging from 3 × 3 to as high as 80 × 80, and commercially preferably from about 10 × 10 to about 30 × 30. Unequal sizes having different numbers of warp wires and filling wires are, of course, possible to create different designs and patterns. Other supporting members containing apertures, openings, orifices, or the like, of circular, square, or other shape, are of possible use and several of these are described and illustrated in the cited patents. The total open area created by such openings of these supporting members will vary widely and can be as low as about 20 percent and as high as about 80 percent. Within the more commercial aspects of the present invention, however, a range of from about 40 percent to about 60 percent open area is found more desirable.
The openings, orifices, apertures, or the like may be uniformly arranged as is normally present in a woven wire screen, or they may be arranged in a square, staggered or clustered arrangement, as desired or required. The openings, orifices, apertures, or the like may be formed in plate members and may merely comprise holes drilled to the desired size, or they may be tapped openings into which are fitted devices capable of forming jets and other columnar streams of fluid.

The greater variety and availability of the above factors provides for a very significant increase in the production line speeds with which the products of the present invention are made. A single pass through the rearranging and reorganizing apparatus of the present invention at relatively high speed is usually all that is required. Additional passes through the rearranging apparatus are possible, however. Production line speeds of up to about 80 yards per minute are obtainable by the use of the principles of the present inventive concept.

Although it is commercially preferred that the fibrous materials be arranged pass under the rearranging jets of water merely once, whereby greater productivity and higher through-put is achieved, several passes under the jets is, of course, utilized where such is desired or required for special or additional rearranging effects. Another variation is the passage of the fibrous materials under the water jets a second or additional times in reverse fashion or with the axis of long direction of the fibrous materials at various directions to the direction of movement during the preceding rearranging process.

The physical properties of the resulting fabrics are also remarkably improved over the physical properties obtained by techniques not utilizing the advantages of the present invention. For example, tensile strengths and normalized tenacities in the long and cross direction are increased up to 38 times the long and cross tenacities of untreated control samples. The modulus in pounds per inch in the long and cross directions is increased up to 31 times the modulus in the long and cross directions of untreated control samples. The toughness in pounds per inch in the long and cross directions is increased up to 112 times the toughness in the long and cross direction of untreated control samples.

The physical properties of the resulting fabrics are particularly improved in those cases where fibers are used which possess low wet modulus. Such fibers possess very limp properties and characteristics in water and are very responsive to the application of the principles of the present invention. Viscose rayon fibers are an outstanding example of such fibers and it is to be noted that the wet modulus of such viscose fibers is extremely low and that they are considered to be one of the limpest forms of fibers in water.

Thus far, the invention has been described primarily with reference to two basic forms: (1) a pretreatment with a softening and swelling agent followed by a subsequent textile process in a large volume of water as is encountered in conventional or modified papermaking processes; and (2) a pretreatment with a softening and swelling agent followed by a subsequent textile process involving jets, streams or sprays of a fluid such as water. A third form is also applicable and involves the combining of (1) the softening and swelling treatment and (2) the textile process into a single simultaneous operation.

Such a combined operation may be obtained by using a softening and swelling agent as the fluid which issues in the form of jets, streams, or sprays during the textile process, whereby the need for the prior pretreatment with a softening or swelling agent is obviated. Care must be exercised in such a process that the apparatus does not comprise aluminum, brass, or other materials which are apt to be affected chemically by the softening and swelling agent and that resistant metals and materials such as selected plastics and stainless steel be used.

The invention will be described in greater detail and greater specificity in the following examples which describe particular embodiments of the present inventive concept for illustrative purposes. Such particularity of detail, however, is not to be construed as limiting of the broader aspects of the present invention.

EXAMPLE I

Bright, no finish, 1.5 denier rayon filaments are cut to a length of one-half inch. These fibers are cut in the wet state and are used in a damp state. Their moisture content is 38 percent, based on the total weight of the fibers and water. There are a considerable number of fiber clumps and bundles in the cut rayon fibrous materials. A 25.6 gm. sample of these fibers is added to 100 milliliters of 5 percent (by weight) sodium hydroxide solution. The fibers are allowed to stand in contact with the caustic solution for 5 minutes at room temperature. At the end of this time, the fibers are softer, limper, and are swollen, and show a strong tendency toward separation of bundles of fibers into individual fibers. The fiber-caustic solution is then dumped into a 23 x 23 inch sheet mold filled to a height of 16½ inches with water (0.008 percent consistency). The sheet mold’s contents are subjected to a standard number of 50 up and down strokes with a paddle consisting of a flat plate attached at right angles to a metal rod. Such stirring is more than ample. A valve at the bottom of the sheet mold is then opened, draining away the water and leaving the rayon fibers in a flat mat on the sheet mold collecting screen. The fibrous web is removed from the screen, sprayed with a 2 percent polyvinyl alcohol solution, and dried on a hot plate. The resulting web, having a weight of about 400 grams per square yard, is very uniform in appearance with virtually no evidence of tangling and clumping of fibers. It is essentially free of bundles of fibers, since virtually all of the bundles are broken down into individual fibers. It possesses excellent isotropicity and has equal properties and characteristics in all directions. Fibrous webs made by these techniques are commercially acceptable and are well suited for further processing into nonwoven fabrics.

EXAMPLE II

The aqueous media papermaking procedures of Example I are followed substantially as set forth therein with the exception that the concentration of the swelling agent is lowered from 5 to 3 percent (by weight) sodium hydroxide solution. The results are generally comparable. The individual rayon fibers become softer, limper and are swollen. Fibrous webs made by these techniques are commercially acceptable and are suitable for further processing into nonwoven fabrics.
EXAMPLE III
The aqueous media papermaking procedures of Example I are followed substantially as set forth therein with the exception that the concentration of the swelling agent is lowered from 5 to 1 1/2 percent (by weight) sodium hydroxide solution. The results show substantial improvement over processes not using sodium hydroxide, although not to the same degree as obtained in Example I. The individual rayon fibers become softer, limper and are swollen. Fibrous webs made by these techniques are marginally acceptable and are suitable in some cases for processing into nonwoven fabrics.

EXAMPLE IV
The aqueous papermaking procedures of Example I are followed substantially as set forth therein with the exception that the swelling agent is 4 percent (by weight) potassium hydroxide. The results are generally comparable. The individual rayon fibers become softer, limper and are swollen. Fibrous webs made by these techniques are commercially acceptable and are suitable for processing into nonwoven fabrics.

EXAMPLE V
The aqueous media papermaking procedures of Example I are repeated except that the rayon fibers are soaked for 5 minutes in plain water instead of 5 percent sodium hydroxide solution. The individual fibers do not become as soft, limp or swollen as in Example I. The resulting web has poor uniformity, and tangling and clumping of fibers is very evident. In addition, many rayon fiber bundles are still intact. Fibrous webs made by these techniques are not commercially acceptable and are not suitable for further processing.

EXAMPLE VI
The aqueous media papermaking procedures of Example I are repeated except that the rayon fibers are treated for 5 minutes with 50 milliliters of 52 percent solution of zinc chloride in water. The individual fibers become softer, limper and are swollen. The resulting web is uniform in appearance with little or no tangling and clumping in evidence. The results are comparable to those obtained in Example I with respect to both uniformity and lack of fiber bundles. Fibrous webs made by these techniques are commercially acceptable and are suitable for further processing into nonwoven fabrics.

EXAMPLE VII
The aqueous media papermaking procedures of Example I are repeated except that the rayon fibers are treated for 5 minutes with 100 milliliters of 43.6 percent lithium chloride solution in water. The individual fibers become softer, limper and are swollen. The resulting web is substantially more uniform and untangled than the web of Example V which is merely pretreated with plain water. The degree of bundle break-up is not quite as good as that of Example I and VI, however. Fibrous webs made by these techniques are commercially acceptable and are suitable for further processing into nonwoven fabrics.

EXAMPLE VIII
The procedures set forth in Example I are followed substantially as set forth therein with the following exceptions.

1. Instead of dumping the pretreated rayon fiber-caustic solution directly into the sheet mold, a washing process is employed first to remove substantially all traces of the sodium hydroxide. Care is taken, however, that the rayon fibers are not permitted to dry out at any time before they are dumped into the water in the sheet mold. This change creates no adverse effects and the results are generally comparable to those obtained in Example I.

2. Instead of dumping the pretreated rayon fiber-caustic solution directly into the sheet mold, a neutralization step is used first wherein a sufficient amount of acetic acid is added to the sheet mold which will neutralize the sodium hydroxide. This is done before the fibers are dumped into the sheet mold. Care is taken, however, that the fibers are not permitted to dry out at any time before they are dumped into the sheet mold. This change creates no adverse effects and the results are comparable to those obtained in Example I.

3. Instead of adding the rayon fibers to the 100 milli-liter solution of 5 percent (by weight) sodium hydroxide solution and swelling them therein, the sodium hydroxide solution is added to the large amount of water in the sheet mold and stirred, and then the rayon fibers are subsequently added to the water in the sheet mold. This change affects the results adversely. The fibers do not become soft, limp or swollen to any appreciable or sufficient amount and the results are not satisfactory. There are tangles and clumps of fibers in the resulting web, which is not uniform and is not commercially acceptable.

EXAMPLE IX
The aqueous media papermaking procedures of Example I are followed substantially as set forth therein with the exception that the consistency in the sheet mold is reduced from 0.008 to 0.004 percent by merely using a 12.8 gram sample of cut rayon fibers in the sodium hydroxide swelling treatment and by keeping the water content in the sheet mold the same. The resulting web has a grain weight of only about 200 grams per square yard and is also essentially free of bundles of fibers. It possesses excellent isotropicity and has equal properties and characteristics in all directions.

EXAMPLE X
The procedures of Example I are followed substantially as set forth therein with the exception that three-fourths inch long, 1.5 denier, bright, no finish rayon fibers are used, and in addition to a swelling pretreatment with sodium hydroxide, a 10 ppm Polyoxy Coagulant Polyethylene oxide solution is used in the sheet mold. Instead of using the standard 50 up and down stroke stirring, only 5 up and down strokes are required. Furthermore, essentially equally good results are obtained when the concentration of Polyoxy is reduced as low as 0.6 ppm.

The procedures are repeated without using the swelling pretreatment with sodium hydroxide. The full number of 50 up and down strokes are required and, at that, it is barely sufficient to create a uniform fiber dispersion, as compared to Example I wherein the standard 50 up and down strokes were far more than sufficient. If this procedure is repeated, i.e., without the swelling pretreatment, and the Polyoxy Coagulant concentration...
is reduced substantially, for example, to 0.6 ppm, it is not possible to obtain sheets of three-fourths inch long, 1.5 denier fibers free of tangles and uniform in fiber deposition, acceptable to commercial standards.

EXAMPLE XI

Semi-dull, 3-denier polyamide nylon 66 tow is cut to a staple length of 1.2 inches. There are a considerable number of fiber clumps and bundles, resulting from the tow cutting process. To 9.7 grams of these fibers is added 100 ml. of 4 percent aqueous phenol solution. After a treatment of 5 minutes, during which time the fibers become soft, limp and swollen, the fibers and phenol solution are dumped into the sheet mold described in Example I. The sheet mold is filled to a height of 16% inches with a 10 ppm solution of Polyox Coagulant polyethylene oxide. The purpose of the Polyox Coagulant is to act as a dispersant and to reduce the tendency of the fibers to tangle with one another. After 200 vigorous strokes with the paddle described in Example I, the water is drained off leaving a web which, after drying, is reasonably uniform in appearance and contains few, if any, of the original bundles resulting from the tow cutting process. Fibrous webs made by these techniques are commercially satisfactory and are well suited for further processing into nonwoven fabrics.

EXAMPLE XII

The aqueous media papermaking procedures of Example XI are followed substantially as set forth therein with the exception that the concentration of the swelling agent is lowered from 4 to 2.4% phenol solution. The results are generally comparable. The individual nylon fibers become soft, limp and swollen. Fibrous webs made by these techniques are commercially acceptable and are suitable for further processing into nonwoven fabrics.

EXAMPLE XIII

The aqueous media papermaking procedures of Example XI are followed substantially as set forth therein with the exception that the concentration of the swelling agent is lowered from 4 to 1.4% phenol solution. The results are generally comparable. The individual nylon fibers become soft, limp and swollen and have less fiber-to-fiber surface friction. Fibrous webs made by these techniques are commercially acceptable and are suitable for processing into nonwoven fabrics.

EXAMPLE XIV

The aqueous media papermaking procedures of Example XI are repeated except that the nylon fibers are soaked for 5 minutes in plain water instead of in 4 percent phenol solution. The individual fibers do not become as soft, limp and swollen. The resulting nylon web gives the appearance of being a very lightweight sheet owing to the fact that many of the fibers are still bound up in the original bundles which resulted from two cutting. Literally hundreds of these bundles are easily identifiable. Fibrous webs made by these techniques are commercially unacceptable and are unsuitable for further processing.

EXAMPLE XV

The aqueous media papermaking procedures of Example XI are repeated except that the nylon fibers are treated for 5 minutes with 50 gm. of 40 percent aqueous zinc chloride solution. The individual nylon fibers become soft, limp and swollen. As in Example XI, the resulting web contains very few bundles of the type which are formed during tow cutting. Fibrous webs made by these techniques are commercially acceptable and are well suited for further processing into nonwoven fabrics.

EXAMPLE XVI

The aqueous media papermaking procedures of Example XI are followed substantially as set forth therein and the nylon fibrous web made in accordance with such aqueous papermaking procedures is impregnation-bonded with an aqueous dispersion of polyvinyl acetate (15 percent resin binder solids). The impregnation takes place on the wet or damp fibrous nylon web while the individual fibers are still soft, limp and swollen. The resulting bonded fibrous web is improved in wet and dry tensile strengths, both in the long and cross directions, as compared to similarly bonded fibrous webs which are not given a pretreatment with 4 percent phenol swelling agent.

The tensile strength of the bonded nylon fibrous web is improved in both the long and in the cross direction in the dry state as well as in the wet state. In some tests, the improvement ranges as high as 50 percent or more.

EXAMPLE XVII

Aqueous media papermaking procedures as described in Example XI are followed substantially as set forth therein except that the 9.7 grams of nylon fibers is replaced by 4.85 grams of 1.5 denier, 1/4 inch rayon fibers and 4.85 grams of 1.5 denier, 1 inch polyamide nylon 66 fibers.

The rayon fibers are pretreated with 5 percent by weight sodium hydroxide for 3 minutes and the polyamide nylon 66 fibers are separately treated with 4 percent phenol solution for 3 minutes. The two batches of fibers are then dumped, while still wet and while they are still soft, limp and swollen, into the large excess of water in the sheet mold. The fiber dispersion is stirred and the water is drained to produce a 50-50 rayon-nylon fibrous web. The properties and characteristics of such blended fibrous web are excellent and the web is well suited for further processing.

One advantage of this blending purpose is the neutralization of the sodium hydroxide and the phenol in the sheet mold aqueous media, whereby the necessity for subsequent processing is reduced.

EXAMPLE XVIII

The aqueous media papermaking procedures of Example XVII are followed substantially as set forth therein except that the rayon and nylon fibers are treated together in 5 percent sodium hydroxide as the swelling agent. The rayon fibers become softer, limper and are swollen. The nylon fibers are relatively unaffected by the sodium hydroxide and do not become soft, limp and swollen. The batch of mixed fibers is dumped into a sheet mold and a fibrous sheet made in the usual way. The sheet is marginally satisfactory and is not as well formed or as uniform as the sheet made in accordance with Example XVII wherein both types of fibers are pretreated and are soft, limp and swollen.
EXAMPLE XIX

The aqueous media papermaking procedures of Example XVIII are followed substantially as set forth therein except that the rayon and nylon fibers are treated together in 4 percent phenol as the swelling agent. The nylon fibers become soft, limp and swollen, whereas the rayon fibers are not as affected. Sheets made of such a mixture of fibers in a sheet mold are satisfactory but are not as well formed or as uniform as the sheets made in accordance with Example XVII wherein both types of fibers are pretreated and are soft, limp and swollen.

EXAMPLE XX

A carded fibrous web having a weight of about 300 grains per square yard and comprising 100 percent semi-dull polyamide nylon 66 fibers having a denier of 3 and a staple length of 1½ inches is treated with a 4 percent phenol solution until the fibers become soft, limp and swollen. The carded fibrous web is then washed in water to remove the phenol and is forwarded for the subsequent textile treatment while still in the wet or damp state. The fibrous web is then bonded with an aqueous dispersion of polyvinyl chloride (15 percent resin binder solids) by impregnation bonding techniques.

The tensile strength of such nylon fibrous web in both the long direction and the cross direction is improved over the tensile strength of nylon fibrous webs not given a pretreatment with a swelling agent. In some cases, the improvement in wet and dry tensile strength, both in the long and in the cross direction, ranges up to 50 percent.

EXAMPLE XXI

The procedures of Example XX are followed substantially as set forth therein with the exception that the resin binder which is used to bond the carded nylon fibrous web is a polyvinyl acetate aqueous dispersion rather than a polyvinyl chloride dispersion. The wet and dry tensile strengths, both in the long and cross directions, are again increased in some cases up to about 50 percent.

EXAMPLE XXII

A carded fibrous web having a weight of 300 grains per square yard and comprising 75 percent (by weight) viscose rayon fibers having a denier of 1½ and a length of 2 inches and 25 percent (by weight) bleached cotton fibers averaging about one-half to three-fourths of an inch is passed through a tank of swelling agent wherein it is treated with a 5 percent (by weight) sodium hydroxide solution for 20 seconds at room temperature.

The treated carded fibrous web containing soft, limp and swollen fibers is then passed through fiber rearranging apparatus disclosed in Figs. 7-10 of U. S. Pat. No. 2,862,251. In this process, the carded fibrous web is placed on a rotatable supporting drum having apertures therein and is covered by a continuous movable screen belt having openings therein which are smaller than the apertures in the rotatable drum. The pressurized jets or streams of water are directed from inside the drum and pass through the larger apertures in the drum, then through the fibrous web, rearranging the individual fibers therein, and ultimately pass through the smaller openings in the screen belt. The rearrangement of the individual fibers is facilitated to a great extent by the soft, limp and swollen nature of the fibers which more readily respond to and are more amenable to the rearranging techniques. The multiplicity of yarn-like fiber groups which are interconnected at junctions are more clearly defined whereby the holes which are formed are cleaner and possess fewer stray fibers therein. The predetermined pattern is very distinct which is brought about by the fact that the individual fibers were more responsive and more amenable to the rearranging techniques.

This fibrous web is compared to the fibrous web resulting from the techniques of Example I in U. S. Pat. No. 2,862,251 which do not involve a swelling pretreatment, and is deemed to be superior thereto. It is also established that lower pressures are useful and production rates may be increased for the process described in this example due to the greater response and receptivity of the soft, limp and swollen fibers to the rearranging in the aqueous media.

EXAMPLE XXIII

The procedures of Example XXII are followed substantially as set forth therein with the exception that the caustic pretreating step and the rearranging step are combined into a single operation. This is accomplished by omitting the caustic pretreatment step and by using 5 percent sodium hydroxide solution as the rearranging fluid.

The results are generally comparable to the results obtained in Example XXII. It is to be appreciated that the improved results are due, in part, to the fact that the viscose rayon fibers have an extremely low wet modulus and the bleached cotton fibers have a low wet modulus whereby they are very limp in water and thus are exceptionally adaptable for use within the principles of the present invention.

EXAMPLE XXIV

A mixture containing 25 percent (by weight) of papermaking fibers, 2 to 3 millimeters in average length, and 75 percent (by weight) of 9% inch, 1½ denier viscose rayon fibers is treated with 5 percent (by weight) sodium hydroxide solution for 4 minutes at room temperature. This fibrous mixture containing soft, limp and swollen fibers is then water-laid to provide a web having a grain weight of 250 grains per square yard. It is treated as in Example XXII and the improved results are comparable. The resulting rearranged fibrous web is compared to the fibrous web resulting from the process described in Example II of U. S. Pat. No. 2,862,251 which does not involve a swelling pretreatment, and is deemed to be superior thereto.

EXAMPLE XXV

A carded fibrous web weighing about 300 grains per square yard and containing 75 percent (by weight) viscose rayon fibers having a denier of 1½ and approximately 2 inches long and 25 percent (by weight) of bleached cotton fibers averaging about one-half inch to about three-fourths inch in length is passed through a treating trough containing 5 percent (by weight) sodium hydroxide for 10 seconds at room temperature.

The treated carded fibrous web containing soft, limp and swollen fibers is then passed through the double screen belt fiber rearranging apparatus disclosed in Fig. 23 of U. S. Pat. No. 2,862,251.
In this process, the carded fibrous web is placed on an endless supporting flexible belt screen having openings therein and is covered by another endless flexible belt screen having openings therein which are larger than the openings in the supporting belt screen. The pressurized jets or streams of water are directed from within the covering flexible belt and pass through the larger openings therein, then through the fibrous web to rearrange the individual fibers therein, and ultimately to pass through the smaller openings in the supporting flexible belt screen.

The multiplicity of yarn-like fiber groups are interconnected at junctures and are very clearly defined. The openings in the rearranged fibrous web are clean and are distinct. The fibrous web is compared to the fibrous web resulting from the techniques of Example III in U. S. Pat. No. 2,862,251 which does not include a swelling pretreatment and is deemed to be superior thereto. Increased production rates are also obtained where the fibers are given a swelling pretreatment.

EXAMPLE XXV-A

The procedures of Example XXV are followed substantially as set forth therein with the exception that the caustic pretreatment step and the rearranging step are combined into a single operation. This is accomplished by omitting the caustic pretreatment step and by using 5 percent sodium hydroxide solution as the rearranging fluid.

The results are generally comparable to those obtained in Example XXV.

It is to be appreciated that the improved results are due in part to the fact that the viscose rayon fibers have an extremely low wet modulus whereby they are very limp in water and thus are exceptionally responsive to the application of the principles of the present invention.

EXAMPLE XXVII

The procedures of Example XXVI are followed substantially as set forth therein with the exception that the caustic pretreatment step and the rearranging step are combined into a single operation. This is accomplished by omitting the caustic pretreatment step and by using 5 percent sodium hydroxide solution as the rearranging fluid.

The results are generally comparable to the results obtained in Example XXVI.

It is to be appreciated that the improved results are due in part to the fact that the viscose rayon fibers have an extremely low wet modulus whereby they are very limp in water and thus are exceptionally responsive to the application of the principles of the present invention.

EXAMPLE XXVIII

A fibrous web comprising irregularly-arranged fibers and having a weight of 300 grams per square yard and comprising 75 percent (by weight) viscose rayon fibers having a denier of 1½ and a length of 2 inches and 25 percent (by weight) of bleached cotton fibers averaging about one-half to three-fourths inch is passed through a tank of 5 percent (by weight) of sodium hydroxide solution for 15 seconds at room temperature.

The treated fibrous web containing soft, limp and swollen fibers is then passed through fiber rearranging apparatus described in U. S. Pat. No. 3,025,585 (FIG. 1). In this process, the web is placed on a supporting carrying screen having tapered projections and the individual fibers are rearranged to define a predetermined pattern of holes or openings around the tapered projections.

The fiber rearrangement is made much easier by the swelling pretreatment and increased production speeds are made available.

EXAMPLE XXIX

Rayon fibers having a denier of 1.5 denier per filament and a staple length of one-half inch are treated with a 5 percent sodium hydroxide solution for 4 to 5 seconds. Without removing the sodium hydroxide, these fibers are then quickly dumped into a large excess of water contained in a 23 inch by 23 inch sheet mold at room temperature and formed into a sheet. The break-up of bundles into individual rayon fibers is excellent and rapid and the fiber dispersing characteristics in the aqueous medium is excellent, thus showing that the swelling action of the sodium hydroxide is very rapid.

EXAMPLE XXX

The procedures of Example XXVIII are followed substantially as set forth therein with the exception that the caustic pretreating step and the rearranging step are combined into a single operation. Specifically, the fibrous web of irregularly arranged viscose rayon fibers and bleached cotton fibers, instead of being pretreated with the 5 percent sodium hydroxide solution and then subsequently rearranged in the fiber rearranged apparatus described in U. S. Pat. No. 3,025,585 (FIG. 1), are rearranged in such apparatus by substituting 5 percent sodium hydroxide solution for water as the rearranging fluid.

The results are generally comparable to the results obtained in Example XXVIII.

It is to be appreciated that the improved results are due in part to the fact that the viscose rayon fibers have an extremely low wet modulus whereby they are very limp in water and thus are exceptionally responsive to the application of the principles of the present invention.
EXAMPLE XXXI

Twelve fibrous card webs are prepared, each having a weight of 350 grains per square yard and comprising viscose rayon fibers having a denier of 1 1/2 and a length of 2 inches. Six of these webs are pretreated by being passed through water at room temperature and six webs are pretreated by being passed through 5 percent sodium hydroxide solution at room temperature. All 12 webs then are rearranged under the conditions set forth in the following table.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Pre-wet</th>
<th>Jet pressure (p.s.i.g.)</th>
<th>Line speed (y.p.m.)</th>
<th>Tenacity (lbs./in./100 gr. wt.)</th>
<th>Modulus (lbs./lin.)</th>
<th>Toughness (lbs./in.)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Long Dry Wet</td>
<td>Long Dry Wet</td>
<td>Long Dry Wet</td>
</tr>
<tr>
<td>1.</td>
<td>Water</td>
<td>110</td>
<td>5.0</td>
<td>7.6</td>
<td>12.1</td>
<td>0.53</td>
</tr>
<tr>
<td>2.</td>
<td>D.O.</td>
<td>110</td>
<td>12.5</td>
<td>3.4</td>
<td>9.1</td>
<td>0.53</td>
</tr>
<tr>
<td>3.</td>
<td>D.O.</td>
<td>110</td>
<td>22.5</td>
<td>11.5</td>
<td>5.5</td>
<td>0.53</td>
</tr>
<tr>
<td>4.</td>
<td>D.O.</td>
<td>110</td>
<td>30.0</td>
<td>15.0</td>
<td>8.0</td>
<td>0.53</td>
</tr>
<tr>
<td>5.</td>
<td>D.O.</td>
<td>110</td>
<td>45.0</td>
<td>23.0</td>
<td>10.0</td>
<td>0.53</td>
</tr>
<tr>
<td>6.</td>
<td>D.O.</td>
<td>110</td>
<td>50.0</td>
<td>33.0</td>
<td>11.0</td>
<td>0.53</td>
</tr>
<tr>
<td>7.</td>
<td>NaOH</td>
<td>110</td>
<td>30.0</td>
<td>12.5</td>
<td>6.0</td>
<td>0.53</td>
</tr>
<tr>
<td>8.</td>
<td>NaOH</td>
<td>110</td>
<td>45.0</td>
<td>23.0</td>
<td>8.0</td>
<td>0.53</td>
</tr>
<tr>
<td>9.</td>
<td>NaOH</td>
<td>110</td>
<td>50.0</td>
<td>33.0</td>
<td>10.0</td>
<td>0.53</td>
</tr>
<tr>
<td>10.</td>
<td>NaOH</td>
<td>110</td>
<td>75.0</td>
<td>50.0</td>
<td>12.5</td>
<td>0.53</td>
</tr>
<tr>
<td>11.</td>
<td>NaOH</td>
<td>110</td>
<td>100.0</td>
<td>75.0</td>
<td>15.0</td>
<td>0.53</td>
</tr>
<tr>
<td>12.</td>
<td>NaOH</td>
<td>110</td>
<td>125.0</td>
<td>100.0</td>
<td>17.5</td>
<td>0.53</td>
</tr>
</tbody>
</table>

The procedures of Example XXXI are followed substantially as set forth therein with the exception that the 14 x 14 mesh screen is replaced by (a) a 22 x 22 mesh woven polypropylene screen, (b) a 13 x 13 mesh high knee woven polypropylene screen, and (c) a perforated belt having 0.058 inch holes on 0.094 inch centers. Line speed was maintained at 5 yards per minute for all samples.

The results are generally comparable to the results obtained in Example XXXI but it is noted that the 13 x 13 high knee wire and the patterning belts are more effective at developing web strengths than the 22 x 22 screen belt.

EXAMPLE XXXIV

The procedures of Example XXXI are followed substantially as set forth therein with the exception that (1) air-formed isotropic fibrous webs made on a Curlator web former and (2) wet-formed isotropic fibrous made by modified papermaking processes described in U.S. Pat. application Ser. No. 801,573 (and particularly Example I therein) are used as the starting materials.

The results are generally superior to the results obtained in Example XXXI, from which it would appear that the present inventive concept is more applicable to and more effective with isotropic fibrous webs rather than carded fibrous webs.

It is again to be appreciated that the benefits and advantages which are achieved are due in part to the fact that viscose rayon fibers are one of the limpest of all fibers in water, possessing an extremely low wet modulus, whereby they are exceptionally adaptable for use within the principles of the present invention.

EXAMPLE XXXV

The procedures of Example XXXIV are followed substantially as set forth therein with the exception that the caustic pretreating step and the rearranging step are combined into a single operation. This is accomplished by omitting the pretreatment with 5 percent sodium hydroxide solution and replacing the water in the rearranging apparatus by 5 percent sodium hydroxide solution.

The results are generally comparable to the results obtained in Example XXXIV. Again, it is to be appreciated that the improved results are due, in part, to the fact that the regenerated cellulose viscose fibers are perhaps the limpest fibers in water and thus are excep-
tionally responsive to the application of the principles of the present invention.

EXAMPLE XXXVI

The procedures of Example XXXI are followed substantially as set forth therein with the exception that the caustic pretreating step and the rearranging step are combined into a single operation. Specifically, six of the samples (samples 7–12), instead of being pretreated with 5 percent sodium hydroxide solution and then subsequently rearranged by means of needle jets of water, are rearranged by means of needle jets supplied with 5 percent sodium hydroxide solution.

The results are generally comparable to those obtained in Example XXXI.

It is again to be appreciated that the benefits and advantages which are achieved are due in part to the fact that viscose rayon fibers are one of the limitest of all fibers in water, possessing an extremely low wet modulus, whereby they are exceptionally adaptable for use within the principles of the present invention.

EXAMPLE XXXVII

The following experiments are carried out to determine the mechanism whereby the caustic or other swelling pretreatments facilitate and expedite the subsequent rearranging operation and yield improved tenacities, moduli, and toughness values.

In these experiments, the starting fibrous materials are fibrous webs comprising viscose rayon fibers, three-fourths inch in length and 1.5 denier per filament. These webs are not carded, oriented webs but are isotropic fibrous webs made by modified papermaking processes described in U.S. Pat. application Ser. No. 810,573, using 20 parts per million of "Polyox" polyethylene oxide coagulant having an average molecular weight in excess of about 5 million.

These webs are treated in the apparatus described in Example XXXI in various ways as set forth in the following table wherein the standard pressure of the water jets is 50 pounds per square inch gauge, the production line speed is 5 yards per minute, and the shape of the jets of water is derived by means of orifice plates having openings of 0.012 inch in diameter. All physical properties set forth in the following table are determined on dry samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Untreated (control)</th>
<th>Pretreated with 5% NaOH, NaOH washed out prior to rearranging</th>
<th>Pretreated with 5% NaOH, NaOH washed out prior to rearranging</th>
<th>Pretreated with 5% NaOH prior to rearranging—wool untreated</th>
<th>Pretreated with 5% NaOH prior to rearranging—wool untreated</th>
<th>Formed in high concentration 100 ppm of Polyox Coagulant</th>
<th>Wet web sprinkled with Polyox Coagulant powder</th>
<th>Pretreated with 1% NaClO</th>
<th>NILGH in jet water (pH 9.6)</th>
<th>Pretreated with 50% ZnCl₂</th>
<th>Pretreated with 50% ZnCl₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>01</td>
<td>01</td>
<td>01</td>
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</tr>
</tbody>
</table>

These results indicate that merely making fibers slippery is not enough to bring about improved fiber rearrangement and improved physical properties in the resulting fabric. This is shown in samples 5 through 8, which provide slippery conditions without providing improved results in fiber rearranging.

These results also indicate that improved fiber rearrangement is obtained in the absence of slippery fibers. This is shown in samples 9 and 10 wherein improved fiber rearrangement is obtained without making the fibers slippery.

These results also indicate that the increased strength is not brought about by autogenic bonding of the individual fibers. This is shown in samples 3 and 4 wherein the residual caustic left in the web in sample 3 does not cause autogenic bonding during drying. The slightly lower strength of sample 4 may be due to the additional handling and processing involved in the neutralization of the caustic with acetic acid, followed by a dewatering by a wringer, rinsing with water, and a second dewatering by a wringer, after the fiber rearrangement and prior to drying.

EXAMPLE XXXVIII

The procedures of Example XXXVIII are followed substantially as set forth therein with the exception that the viscose rayon fibrous web with 5 percent sodium hydroxide solution is omitted and the rearranging fluid used in the rearranging jets is 5 percent sodium hydroxide solution rather than water.

The results are generally comparable to the results obtained in Example XXXVIII.

EXAMPLE XL

The following experiments are carried out to determine the effect of the caustic or other preswelling treatment on fibers of varying lengths. In these experiments, the apparatus of Example XXXI is used, the fibrous webs are wet formed, isotropic webs unless indicated otherwise, the pretreating solution is 5 percent sodium hydroxide, the standard pressure of the water jets is 100 pounds per square inch gauge, the production line speed is 5 yards per minute, the orifice plates are provided with openings 0.012 inch in diameter, and the permeable, lower supporting belt is the high knee type of forming wire screen illustrated in FIG. 5 of U. S. Pat. No. 3,025,585 and has a mesh of 14 × 14.

The results are set forth in the following table, wherein all physical properties are determined on dry samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Untreated (control)</th>
<th>Pretreated with 5% NaOH, NaOH washed out prior to rearranging</th>
<th>Pretreated with 5% NaOH, NaOH washed out prior to rearranging</th>
<th>Pretreated with 5% NaOH prior to rearranging—wool untreated</th>
<th>Pretreated with 5% NaOH prior to rearranging—wool untreated</th>
<th>Formed in high concentration 100 ppm of Polyox Coagulant</th>
<th>Wet web sprinkled with Polyox Coagulant powder</th>
<th>Pretreated with 1% NaClO</th>
<th>NILGH in jet water (pH 9.6)</th>
<th>Pretreated with 50% ZnCl₂</th>
<th>Pretreated with 50% ZnCl₂</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>01</td>
<td>01</td>
<td>01</td>
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</tr>
</tbody>
</table>

*NaOH not washed out prior to rearranging.

These results indicate that merely making fibers slippery is not enough to bring about improved fiber rearrangement and improved physical properties in the resulting fabric. This is shown in samples 5 through 8, which provide slippery conditions without providing improved results in fiber rearranging.
TABLE

Effect on Rearranging of Pre-swelling Rayon Webs With 5% NaOH Solution

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>%” Rayon — Untreated</td>
<td>.12</td>
<td>.08</td>
<td>2.77</td>
<td>.26</td>
</tr>
<tr>
<td>%” Rayon — NaOH treated</td>
<td>.28</td>
<td>.10</td>
<td>4.50</td>
<td>.65</td>
</tr>
<tr>
<td>%” Rayon — Untreated</td>
<td>.22</td>
<td>.14</td>
<td>4.50</td>
<td>1.10</td>
</tr>
<tr>
<td>%” Rayon — NaOH treated</td>
<td>.32</td>
<td>.22</td>
<td>7.63</td>
<td>2.37</td>
</tr>
<tr>
<td>%” Rayon — Untreated</td>
<td>.34</td>
<td>.35</td>
<td>4.79</td>
<td>3.32</td>
</tr>
<tr>
<td>%” Rayon — NaOH treated</td>
<td>.60</td>
<td>.55</td>
<td>8.78</td>
<td>3.42</td>
</tr>
<tr>
<td>%” Rayon — Untreated</td>
<td>.20</td>
<td>.21</td>
<td>3.31</td>
<td>5.26</td>
</tr>
<tr>
<td>%” Rayon — NaOH treated</td>
<td>.64</td>
<td>.51</td>
<td>9.90</td>
<td>5.65</td>
</tr>
<tr>
<td>%” Rayon — Untreated</td>
<td>.41</td>
<td>.31</td>
<td>3.50</td>
<td>4.50</td>
</tr>
<tr>
<td>%” Rayon — NaOH treated</td>
<td>.67</td>
<td>.48</td>
<td>9.14</td>
<td>3.67</td>
</tr>
<tr>
<td>%” Rayon Curlator Web</td>
<td>.09</td>
<td>.07</td>
<td>2.59</td>
<td>2.23</td>
</tr>
<tr>
<td>%” Rayon Curlator Web NaOH treated</td>
<td>.37</td>
<td>.40</td>
<td>5.33</td>
<td>6.98</td>
</tr>
</tbody>
</table>

These results clearly show the superiority of the use of longer fibers and caustic pretreated webs. The low strength of the Curlator-formed fibrous web is a result of their heavy weight which is in the range of about 1,000 grams per square yard.

EXAMPLE XLI

The procedures of Example XL are followed substantially as set forth therein with the exception that the caustic pretreatment is omitted and the openings on the orifice plates emit jets of 5 percent sodium hydroxide solution for rearranging purposes.

The results are generally comparable to the results obtained in Example XL.

EXAMPLE XLII

The following experiments are carried out on fibrous webs made in a papermaking sheet mold. The fibrous webs consist of randomly distributed ¾ inch length, 1.5 denier per filament, precision-cut, bright, no-finish, viscose rayon tow. Prior to the formation in the sheet mold, the fibers are pretreated with 5 percent caustic soda solution to facilitate and expedite breakup of the fiber tow bundles and to improve the uniformity and strength of the resulting fibrous web. A concentration of 10 parts per million of “Polyox” polyethylene oxide coagulant having a molecular weight in excess of 5 million is used in the sheet mold to promote web formation. Acetic acid is subsequently added to the sheet mold water to neutralize the caustic treated fibers.

The wet laid webs are removed from the sheet mold and are air-dried on screens and yield essentially unbonded samples which are suitable for subsequent rearranging process. A grain weight of 400 grams per square yard (dry basis) is used in all experiments. The following rearranging conditions are used: the apparatus used in that described and illustrated in U. S. Pat. No. 3,485,706 (FIG. 2); the water jet pressure is 125 lbs. per square inch gauge; the orifice size is 0.015 inch straight cylindrical holes; the orifice spacing is one 6 inch row of orifices, with the orifices spaced 16 to the inch; the orifice to fibrous web distance is 1½ inches; and the supporting pattern belt is a 23 x 23 mesh woven high knee polypropylene screen.

Samples of the fibrous web derived from the papermaking process in the sheet mold are cut to a size of 5½ x 5½ inches and are placed on the supporting pattern screen and are pretreated by being dipped either in water or in 5 percent caustic soda solution depending upon whether or not a blank control pretreatment or a caustic pretreatment is desired. The samples and the supporting screen are then removed beneath the manifold of the orifices. A vacuum of 5 inches of mercury is used beneath the supporting pattern screen. The rate of speed of movement between the orifice manifold and the samples on the supporting screen is approximately 5 yards per minute.

The samples on the screen are then rotated 90° and a second pass is made with respect to the water jets in which the samples receive a second rearranging treatment. Two passes at 90° to one another are designated as one rearranging treatment. This procedure is then repeated, if necessary, to obtain the desired number of treatments. Samples are then dried and pressed on a photographic print drier at a temperature of about 250° F. The strengths or tenacities of the samples in the dry machine direction are then determined.

The pretreatment with caustic soda solution greatly accelerates and increases the benefits and advantages of the rearranging process. One rearranging treatment of a caustic pretreated web yields strength of tenacity values which are almost as high as the strength or tenacity values which are obtained only by three rearranging treatments of an untreated web.

EXAMPLE XLIII

The procedures of Example XLII are carried out substantially as set forth therein with the exception that the samples which were pretreated with 5 percent soda and then rearranged were treated with 5 percent soda and simultaneously rearranged. This is accomplished by charging the manifold with the 5 percent caustic soda. It is noted that the fibrous webs are driven deeper into the patterning carrying screen when the liquid manifold is charged with the 5 percent caustic soda. Otherwise, the results are generally comparable to those obtained in Example XLII except that in a few cases the strengths or tenacities in the long or machine direction are lower for the combined operation.

EXAMPLE XLIV

The procedures of Example XLII are carried out substantially as set forth therein with the exception that the fibrous web is placed between two patterning screens and rearranged while so positioned. The sandwich of two patterning screens and the interleaved fibrous web is then turned over and subjected to a second rearranging process. The strengths or tenacities in the machine direction are then determined for those samples which are treated with water and with 5 percent caustic soda solution as the rearranging fluid. It is noted that improved strengths or tenacities are obtained when the 5 percent caustic soda solution is used. It is also observed that the sandwich technique reduces the effectiveness of the rearranging process and this is believed to be due to the attenuation of the jet energy by the top screen.

3,736,097
EXAMPLE XLV.

The procedures of Example XLII are carried out substantially as set forth therein with the exception that the orifice to fibrous web distance is changed to one-half inch rather than 1½ inches as used in Example XLII. There is no significant difference in strengths or tenacities due to the change in the distance from the orifice to the sample.

EXAMPLE XLVI.

The procedures of Example XLII are carried out substantially as set forth therein with the exception that the fibrous materials are rearranged on a plate having staggered rows of 0.067 inch diameter circular holes on 0.111 inch centers. The staggered center-to-center distance between adjacent rows is 0.093 inch. This particular plate yields a rearranged fabric which more closely resembles the type of fabric which is obtained by the techniques described and illustrated in U. S. Pat. No. 3,033,721. This patent may be described as possessing compact masses of entangled fibers which are interconnected by ordered fiber groups. It is noted that this particular form of patterning plate develops strengths and tenacities in rearranged fabrics faster than the high knee wire patterning screen used in Example XLII.

EXAMPLE XLVII.

Natural polyamide nylon 66 continuous filament tow (3 denier per filament) is cut with a guillotine-type tow cutter to a staple length of three-eighths inch. The resulting fibers are found to be in clumps and are not loose or individualized. The clumps of fibers are particularly held together at their ends, due most likely to the mechanical pressure crushing of the thermoplastic nylon fibers during the cutting process. The ends of the clumps are studied and found to be adhered aggregates of parallel fiber bundles.

These fiber bundles are placed in water and agitated but do not break apart. They resist breakdown into individual fibers. The fibers are removed and are soaked in γ-butyrolactone at 115° C. for 2 minutes and become soft, limp and swollen. Upon being placed in water again, the fibers show marked increase in their tendency to break down into individual fibers whereby clumps and aggregates are no longer noted in the fiber slurry and excellent uniform fiber sheets are made in a conventional sheet mold.

EXAMPLE XLVIII.

“Kodel” polyethylene terephthalate polyester continuous filament tow (1½ denier per filament) is cut on a guillotine-type tow cutter to a staple length of one-half inch. The resulting fibers are found to be in clumps and are not loose or individualized. The clumps of fibers are particularly held together at their ends, due most likely to the mechanical pressure crushing of the thermoplastic polyester fibers during the cutting process. The ends of the clumps of fibers are studied and are found to be adhered aggregates of parallel fiber bundles.

These fiber bundles are placed in water and agitated but do not break apart. They resist breakdown into individual fibers. However, if they are given a pretreatment in N-methyl pyrrolidone at 110° C. for 2 minutes, they become soft, limp and swollen, and they promptly break apart when placed in water and form excellent uniform fiber slurries of individualized fibers which are easily made into excellent, uniform fiber sheets in a conventional sheet mold.

Although the invention has been described by reference to the preceding specific examples, such is illustrative and not limitative. The broader aspects of the inventive concept are not to be limited thereby, except as defined by the appended claims.

What is claimed is:

1. A method of treating fibrous materials to render them more amenable to subsequent hydraulic nonwoven textile manufacturing processes employing aqueous media which comprises: treating fibrous materials with an effective amount of a swelling agent whereby the individual fibers become soft, limp and swollen but do not discolor, dissolve, degrade, or decompose and do not gel at their surfaces to adhere to each other; rendering said swelling agent inactive and inoperative as a swelling agent and incapable of further swelling said fibrous materials but keeping said fibrous materials wet; exposing said fibrous materials and the soft, limp and swollen fibers therein to hydraulic nonwoven textile manufacturing processes employing fluid rearranging forces in aqueous media wherein the individual fibers show less tendency toward undesirable fiber agglomeration, greater tendency toward fiber individualization, and enhanced response to fiber processing and manipulation by said fluid rearranging forces because of their soft, limp and swollen nature; and forming a nonwoven fabric of the individual fibers.

2. A method as defined in claim 1 wherein the fibrous materials comprise regenerated cellulose viscose rayon fibers and the swelling agent is sodium hydroxide.

3. A method as defined in claim 1 wherein the fibrous materials comprise regenerated cellulose viscose rayon fibers and the swelling agent is zinc chloride.

4. A method as defined in claim 1 wherein the fibrous materials comprise regenerated cellulose viscose rayon fibers and the swelling agent is lithium chloride.

5. A method as defined in claim 1 wherein the fibrous materials comprise polyamide nylon fibers and the swelling agent is phenol.

6. A method of treating fibrous materials to render them more amenable to subsequent hydraulic nonwoven textile manufacturing processes employing aqueous media which comprises: treating fibrous materials with an effective amount of a swelling agent whereby the individual fibers become soft, limp and swollen but do not discolor, dissolve, degrade, or decompose and do not gel at their surfaces to adhere to each other; rendering said swelling agent inactive and inoperative and incapable of further swelling said fibrous materials by diluting the concentration of said swelling agent by introducing said soft, limp and swollen fibers into a large excess of aqueous media wherein the individual fibers show less tendency toward undesirable fiber agglomeration and are readily capable of individual independent relative movement in response to applied fluid rearranging forces in said aqueous media because of their soft, limp and swollen nature and form a uniform dispersion having excellent fiber individualization; applying fluid rearranging forces to said fibers in said aqueous media; and forming a nonwoven fabric of the individual fibers.

7. A method as defined in claim 6 wherein the fibrous...
3,736,097

materials comprise regenerated cellulose viscose rayon fibers.

8. A method as defined in claim 7 wherein the swelling agent is sodium hydroxide.

9. A method of treating fibrous materials to render them more amenable to subsequent hydraulic nonwoven textile manufacturing processes employing aqueous media which comprises: treating fibrous materials with an effective amount of a swelling agent whereby the individual fibers become soft, limp and swollen but do not discolor, dissolve, degrade, or decompose and do not gel at their surfaces to adhere to each other; rendering said swelling agent inactive and inoperative as a swelling agent and incapable of further swelling said fibrous materials but keeping said fibrous materials wet; supporting said fibrous materials and the soft, limp and swollen fibers therein on a member having apertures therein; exposing said fibrous materials and the soft, limp and swollen fibers therein to fluid rearranging forces in the form of streams of aqueous media wherein the individual fibers have an enhanced response to fiber manipulation by said fluid rearranging forces because of their soft, limp and swollen nature, said streams passing in turn through said supporting member, through said fibrous materials, and through said covering member having smaller apertures; and forming a nonwoven fabric of the individual fibers comprising a multiplicity of yarn-like fiber groups interconnected at junctures to define a multiplicity of holes.

12. The method according to claim 11 wherein the fibers are cellulose fibers and the softening and swelling agent is sodium hydroxide.

13. A method of treating fibrous materials to render them more amenable to subsequent hydraulic nonwoven textile manufacturing processes employing aqueous media which comprises: treating fibrous materials with an effective amount of a swelling agent whereby the individual fibers become soft, limp and swollen but do not discolor, dissolve, degrade, or decompose and do not gel at their surfaces to adhere to each other; rendering said swelling agent inactive and inoperative as a swelling agent and incapable of further swelling said fibrous materials but keeping said fibrous materials wet; supporting said fibrous materials and the soft, limp and swollen fibers therein on a member having apertures therein; covering said fibrous materials with a member having apertures therein which are smaller than the apertures in said supporting member; exposing said fibrous materials and the soft, limp and swollen fibers therein to fluid rearranging forces in the form of streams of aqueous media wherein the individual fibers have an enhanced response to fiber manipulation by said fluid rearranging forces because of their soft, limp and swollen nature, said streams passing in turn through said covering member having smaller apertures, through said fibrous materials, and through said supporting member; and forming a nonwoven fabric of the individual fibers comprising fibers in mechanical engagement one with another and arranged flatwise in bundles interconnected at junctures by protuberant pivotal packings of fibers protruding out of the plane of said flatwise bundles to define a predetermined pattern.

14. The method according to claim 13 wherein the fibers are cellulose fibers and the softening and swelling agent is sodium hydroxide.

* * * * *