PROCESS FOR FORMING HYDROXYETHYL CELLULOSE FIBERS HAVING HIGH WATER ABSORPTION AND HIGH WATER RETENTION PROPERTIES

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ABSTRACT

Fibrous cellulosic products are prepared by extruding cellulose solutions through fiber forming devices. These fiber forming devices comprise porous extrusion media containing a controlled number and distribution of discrete flow paths. These flow paths constitute at least about 10 percent of the total extrusion surface area. The cellulosic fibers prepared by these processes exhibit unique and highly desirable physical and structural characteristics for use in forming new and improved nonwoven fabrics and disposables.

1 Claim, 2 Drawing Figures
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PROCESS FOR FORMING HYDROXYETHYL CELLULOSE FIBERS HAVING HIGH WATER ABSORPTION AND HIGH WATER RETENTION PROPERTIES

This invention relates to processes for preparing fibrous cellulose products. More particularly, it relates to processes for preparing new and improved cellulose filaments and staple fibers and to the products prepared thereby. It has for an object, the provision of improved processes to effect economies, especially in the extruding operation, and to produce fibers and webs especially suited for use in nonwovens and disposables.

The new low-cost cellulose fibers are prepared from cellulose solutions such as viscose, hydroxyl cellulose, cellulose acetate and the like and mixtures thereof. These fibers possess a combination of physical and structural properties unattained in any hitherto known comparable fibrous cellulose products. More specifically, the fibers of the present invention have a very low degree of crystallinity, small crystal size, a low degree of polymerization, a randomly oriented internal structure, and unique cross-sectional shapes

Additionally, the fibers of the present invention such as those prepared from viscose solutions, hydroxyl cellulose solutions and the like and mixtures thereof, possess further unique characteristics unattained heretofore. These fibers demonstrate an exceptionally high degree of water or moisture absorption and retention, a high degree of dimensional swelling capability and a large percentage of surface area.

Furthermore, the peripheral shapes of the fibers of this invention, which are formed by extrusion through unique fiber forming devices, substantially conform to the cross section of the extrusion paths or orifices of these fiber forming devices. Consequently, the filamentous products of the present invention can easily be produced in a great variety of special shapes. This feature of the present invention which has not been achieved heretofore has considerable significance. For example, in some circumstances, it is highly desirable to produce fibers exhibiting highly crenulated peripheral shapes resembling in cross section, sting-ray bat-like or crab-like shapes. Such crenulation increases the total surface area of the fiber, and thus enhances the moisture absorptive character of the fiber. It is now possible to produce such shaped fibers, without inter-fiber adhesion, at the discretion of the producer by merely selecting the desired shaped orifice in the fiber forming device.

It should be understood that the term “fiber,” as employed in the present discussion, designates both continuous filaments and staple fibers. It is well known that in the production of cellulose fibers, cellulose solutions are converted into continuous filaments and then staple fibers are produced by cutting the continuous filaments into desired lengths.

Because of the unique physical and structural characteristics of the new and improved fibers of the present invention, they are ideally suited for use in the new convenience fabrics which are neither woven nor knitted. For example, nonwoven fabrics prepared from the new viscose and hydroxyl cellulose fibers of the present invention have a high comfort factor because of the outstanding hydrophilic nature of these new fibers as produced by the processes of the present invention. Additionally, the nonwoven fabrics thus prepared possess sufficient body to exhibit an improved fabric-like hand as compared with nonwovens currently produced from cellulose materials.

The uses for such nonwoven fabrics are varied, and the potentials are exceptional. For example, a great demand exists for nonwoven disposable products in the institutional field, in hospitals, nursing centers, and the like. These products may be in the form of sheets, pillowcases, pads, surgical and other gowns, coats and uniforms. In industry, nonwovens and disposables find acceptance as coversalls, uniforms and cleaning wipes. In the home, where convenience is an overriding factor, disposables may be used as diapers, sanitary napkins or inserts, cleaning wipes, novelty items, and the like. Given this large and ever expanding market for nonwovens, fiber producers have expended considerable research time and capital in the search for methods of producing nonwovens and disposables of improved acceptability to the user.

However, the numerous attempts to provide such processes to produce commercially acceptable fibrous products have not been successful, heretofore. This is evidenced by the fact, that, although the largest proportion of fibers used today to construct nonwoven and disposable fabrics is rayon, the fibers in use are regular textile grades of staple fiber and the processes for preparing these fibers are not generally well adapted for large-scale high speed fiber production. Accordingly, a heretofore unmet need existed for improved nonwoven products.

Our invention provides specially tailored cellulose fibers for use in nonwovens and disposables. For example, the new and improved viscose fibers, hydroxyl cellulose fibers and the like and mixtures thereof provided herein possess qualities which make them ideally suited for nonwoven fabrics and disposables. These qualities include high swelling capacity; high water or moisture absorption and retention; high strength, orientation and crystallinity; rapid loss of strength and bonding when contacted with excess water for disposal, (i.e., flushability); ready perceptibility to dimensional change or adjustment (i.e., conformability); and relatively lower cost.

Our invention also contemplates the utilization of these fibers to produce webs, batts and other nonwoven and disposable materials and fabrics. We have found that the fibers of the present invention and notably the hydroxyl cellulose fibers, exhibiting high swelling characteristics, possess self bonding or adhesive properties between the fibers themselves or with other structures when they are pressure printed with water in such design shapes as embossed diamonds, waffles and the like. Thus, webs, batts and the like can be readily prepared from these fibers. If bonding strength greater than that achieved by the self adhesion of the fibers is desired, we have found that this can be easily achieved. For example, the bonding may be strengthened by mildly alkalinizing the water employed in the pressure printing process with an alkali such as ammonia, sodium carbonate, sodium hydroxide and the like.

Wells, batts and the like, thus prepared demonstrate exceptional capacity for absorbing, retaining and holding moisture. Additionally, in the disposal phase, these webs, batts and the like exhibit release action in bonds and loss of fiber strength in the presence of excess water similar to that observed in "flushing". In the course of our experimental investigations, we have discovered processes for producing low-cost cellulose fibers such as viscose and hydroxyl cellulose fibers having properties which are superior for nonwovens and disposables with respect to water absorption, water retention, cross-sectional shape, fiber elongation and tenacity. The inventive processes contemplate economies in the preparation, composition, and treatment of the cellulose solutions. Additionally, the new techniques which we have discovered for forming the new and improved cellulose fibers enables the replacement of the costly precious metal spinnersets which have been used almost universally heretofore in the preparation of cellulose fibers. This elimination of the very expensive spinnersets constitutes a major breakthrough in the production of low-cost nonwoven and disposable fibers because of the saving in cost of the fiber forming device, and also, because of the substantial increase in product output per extrusion area.

Within the concept of the present invention, continuous filaments and staple fibers are produced by extruding specially prepared cellulose solutions through relatively inexpensive fiber forming devices comprising porous media having a controlled number and distribution of discrete flow paths, the flow paths comprising at least about 10 percent of
the total extrusion surface area. For example, these flow paths may comprise parallel metallic or plastic capillaries, paths created by sintering aggregates of controlled sized powders and then covered or woven screens of plastic or suitable metals such as stainless steel and the like.

In one embodiment of the present invention, a rapid and continuous process is provided for producing new and improved viscose fibers possessing a water retention capability of between about 120 percent and about 200 percent. A very strong, fast setting viscose solution, having a high solids concentration, is preferentially from dissolved cellulosic substances in sodium hydroxide solution. Preferably, the pulp is treated with a non-ionic agent such as the block copolymers of polyethylene oxide and polypropylene oxide, for example, those produced by the Wyandotte Chemical Corporation and sold under the name Pliuronic, and especially Pliuronic L-64. The pulp is steeped in a sodium-hydroxide solution, then it is pressed, shredded, and aged, preferably by catalysis, to produce an alkali cellulose containing about 32–38 percent cellulose and about 14–16 percent sodium hydroxide and having an average degree of polymerization preferably less than about 400 and as low as about 150. The alkali cellulose is then xanthated at a temperature of about 25°–40° C. with about 25–30 percent calcium hypochlorite, preferably from the xanthate dissolved in sodium hydroxide solution to form a lean viscose solution. Preferably, this viscose solution contains from about 7–12 percent cellulose and about 4–6 percent sodium hydroxide, with a most preferred composition being about 10 percent cellulose and about 5 percent sodium hydroxide. The solution is then subjected to deseration while ripening to a salt index of about 1.5–7, preferably about 1.0. Thereafter, the viscose solution is screened or given a single filtration and then extruded into continuous filaments through a porous fiber forming device having flow paths therein comprising at least about 10 percent of the available extrusion area and preferably from about 10 to about 35 percent. The extrusion is carried out at about 25°–55° C. into a fast setting bath containing about 8–10 percent sulfurous acid and about 12–20 percent sodium sulfate. No regeneration retarding components, such as zinc salts and the like, are required in the bath since very fast coagulation and regeneration is highly desirable in avoiding filament adhesion. It is generally advantageous to add a surface active agent such as lauryl pyridinium chloride, to maintain the surface tension of the bath below a level of about 50 dynes/cm for rapid penetration of the emerging filaments. The filaments are stretched 10–60 percent, washed, and dried by conventional means. If desired, the viscoses may be cut into staple fibers preferably in the acid state before washing and drying.

In another embodiment of the invention, new and improved low-cost fibers having an exceptionally high water retention (between about 200 percent and about 400 percent) are prepared from hydroxyalkyl cellulose solutions, particularly hydroxyethyl cellulose solutions. The process comprises forming an alkali cellulose crumb containing about 32–38 percent cellulose and about 14–16 percent sodium hydroxide by steeping dissolving grade wood pulp in an 18–19 percent sodium hydroxide solution at ambient temperatures. Then, the alkali cellulose is pressed and shredded. This alkali cellulose crumb is etherified to a uniform hydroxyalkyl content of from about 0.1–0.3 molar substitution, and preferably to a molar substitution of about 0.15 which is equivalent to substitution of about 0.0.4 percent of ethylene oxide (by weight) added to the cellulose. The etherified cellulose is then aged by oxidative means, preferably with catalysts such as cobalt, to a degree of polymerization preferably less than about 400 and as low as about 150. This aged hydroxyalkyl cellulose crumb is dissolved in a sufficient amount of dilute sodium hydroxide solution to form a solution having a viscosity of about 20–200 poises at 20° C. This solution has a cellulose content of about 6–12 percent and a sodium hydroxide content of about 4–8 percent, and preferably about 10 percent cellulose and about 5–6 percent sodium hydroxide. After this solution is filtered or screened to remove any lumps therein and deaerated, it is extruded into continuous filaments through a porous fiber forming device having flow paths comprising at least about 10 percent of the available extrusion area and preferably from about 10 to about 35 percent. The extrusion is conducted at about 20°–50° C. into an acid bath containing about 10–15 percent sulfurous acid and about 12–20 percent sodium sulfate. The filaments are stretched about 10–60 percent, washed, and dried by conventional means. If desired, the hydroxyalkyl cellulose filaments can be cut into staple fibers, preferably in the acid state before washing and drying.

A further embodiment of this invention comprises a process for the preparation of low-cost cellulose acetate fibers having physical properties that make them attractive as components of the furnish used in preparing nonwoven fabrics and disposables. The cellulose acetate fibers are thermoplastic which makes them particularly useful as bonding fibers for such fabrics. Additionally, the acetate fibers are quite attractive in appearance and tend to improve the"hand"of the fabrics. We have found that, under proper conditions, hydrolyzed or partially hydrolyzed cellulose acetate reaction solutions can be converted into fibers by extrusion through a fiber forming device of the present invention. The process comprises forming filaments of cellulose acetate in sodium hydroxide solution to form a single stage filaments of cellulose acetate by dissolving grade wood pulp by treatment with acetic acid and esterification with an acetic acid-acetic anhydride mixture in the presence of an acetylation catalyst such as sulfuric acid. The triacetate therein is hydrolyzed, at a temperature of from about 40° to about 80° C., to an acetyl content of about 30–44 percent, or if desired, to much lower acetyl to increase hydrophilic properties. If desired, the residual acetylation catalyst is then neutralized with sodium or potassium acetate. The solution containing about 10–15 percent cellulose acetate and about 78–83 percent acetic acid is single stage filtered or screened and then extruded through a porous fiber forming device of the present invention into a coagulating bath containing about 5 to about 40 percent sodium or potassium acetate and about 5–15 percent acetic acid at a temperature of from about 5° to about 30° C. The filaments are stretched about 10–60 percent, washed, and dried by conventional means. If desired, the cellulose acetate filaments can be cut into staple fibers, preferably in the acid state before washing and drying.

In other embodiments of the present invention, various cellulosic solutions are mixed or blended to provide cellulosic fibers having unique physical properties. For example, it has been discovered that the moisture absorption and retention of extruded viscose fibers is controllably increased by the blending of from about 5 to about 30 percent (by volume) of a compatible alkyl hydroxyethyl cellulose solution and up to about 5 percent carbon disulfide with a lean, aged viscoso solution of the present invention having a salt index of about 1.5–7.0. This blend is then immediately extruded into an acid bath at a temperature of about 20°–50° C. through a fiber forming device of the present invention. The fibers formed are cut into staple, if desired, washed and dried by conventional methods.

As previously described, it is now desirable within the purview of this invention to provide fibers having high swelling capability. However, in some applications, it may be useful to reduce or control such swelling characteristics. This control can be achieved easily by means of cross-linking the fibers, for example, by incorporating a small amount of formaldehyde or an amino resin and the like in either the cellulose solution or the acid bath.

In a further embodiment of our invention, fibrous continuous filament webs are formed directly. This can be achieved by extruding sodium hydroxide solution through special arrangements of the fiber forming devices of the present invention. For example, a single long, narrow rectangular fiber forming device or a multiplicity of such devices, properly positioned, may be employed to form webs of filaments in a continuous sequential operation comprising extrusion, washing, bonding,
and drying to yield a finished roll goods product suitable for conversion into nonwoven disposables such as sheets, wipes, pads, garments and the like.

Randomizing or texturizing of fiber arrangement and orientation within these filament webs can also be achieved if desired. For example, we have found that employing three separate, parallel fiber forming devices and oscillating the central one of the devices back and forth out of phase with the other two outside devices provides a three layered web with wave-like overlaps. Such webs can be led through an acid bath on a coagulating device virtually without stretch, washed and dried in a manner which preserves web continuity. Prior to drying, the web can be pressed through patterned squeeze rolls embossed with such designs as a diamond overlap pattern or a wave like pattern to enhance bonding capability and produce webs that require no additional adhesive, no special purification, or bleaching. If special fabric like hand is desired, use of a softening agent may be desirable as a flush through treat either prior to or subsequent to the embossing pattern pressing and prior to drying.

Additionally, the filament webs prepared in accordance with the present invention, in a partially regenerated state, may be combined with a web of flocked fibers such as wood, polymeric synthetic fibers, acetate fibers, rubber, glass or fibers from various different types of spun rayons. The combined webs can be passed through squeeze rolls and the inherent binding power of the filaments of the present invention, after complete regeneration, holds the nonwoven structure together to produce a nonwoven fabric. If desired, various after-treatments may be employed subsequently, such as desulfurizing, bleaching, washing, softening, finishing, cross-linking and the like.

In previously proposed processes for preparing cellulosic fibers, it has been customary to employ precious metal spinerettes wherein open area for fiber formation normally constituted less than 5 percent of the available extrusion area. In the process of the present invention, these costly spinerettes are replaced by fiber forming devices of the group consisting of porous metal and plastic materials such as sintered aggregates, finely woven metal and plastic screens, porous metal structures containing parallel capillaries and the like. These porous extrusion media have a substantially greater open area than the heretofore conventional spinerettes. Consequently, the presently proposed processes employing these devices enable a substantial increase in fiber extruded per square inch of extrusion area as well as a more rapid extrusion rate.

The fiber forming devices employed in the processes of the present invention may be prepared from corrosion-resistant metals such as platinum-gold, platinum-rhodium, nickel, tantalum and the like, if desired. However, the use of these metals is optional with the devices of this invention. If fact, as normally practiced, the fiber forming devices of the present invention are prepared from a variety of different plastics, relatively inexpensive metals such as stainless steel, and other materials. In a preferred embodiment, the fiber forming device comprises a finely woven stainless steel dutch double twill screen coated with a protective, non-stick coating compound such as a fluorinated polyethylene, a polyvinyl chloride or a silicone or the like.

The extrusion area of these fiber forming devices will vary in a wide range, for example, from about one-fourth inch to several inches in diameter. The devices may contain from very few to several thousand holes or orifices per extrusion area, constituting flow paths through the devices; although, it is preferred to employ from about 5,000 to 100,000 holes per extrusion area. These holes or orifices may be circular, square, rectangular, oblong, random or irregularly shaped and normally range in size from about 1 mil (equivalent diameter) to about 5 mils (equivalent diameter). However, other shapes and sizes may be employed if desired to provide special effects in the extruded fibers. Nonetheless, we have found that for best results, the extrusion flow paths through these devices should comprise at least about 10 percent of available extrusion area, and preferably between about 10 percent and about 35 percent.

In certain instances, depending upon the particular shape of the holes or orifices in the fiber forming devices and the number and spacing of these holes or orifices per unit area, the necessary access of acid within the large masses of fibers being extruded through the devices is not achieved. We have found that in such cases, suitable access channels for the acid can be provided at the downstream surface of the extrusion devices. For example, channels may be formed by blocking off patterned hole arrangements on the surface of the porous extrusion devices or by hole patterns or perforations in the means employed to support the screens. In a preferred embodiment, the flow paths in the porous structure itself can be closed, for example, by using a heat sealable plastic cut out defining acid path access channels. In a particularly preferred embodiment, the patterned design or shape is pressed directly into a metal screen coated with a fluorinated polyethylene such as polytetrafluoroethylene and heat sealed in place to form an integral and serviceable low-cost extrusion structure.

In operating the processes of this invention, we have found that a minimum of filtration is required, and no filtration other than a perfunctory preliminary screening of the solutions prior to extrusion is needed. Thus, the instant processes obviate the need for extensive filtration which has been required heretofore. Also, the instant processes accommodate the use of less expensive dissolving pulps and permit the use of "leaner" cellulose solutions having higher solids concentration than have been possible in previous processes.

In fact, the combination of fast setting, lean solutions and fast setting, non-retarding acid baths promote the extrusion of high density of filaments through a given extrusion area without excessive amounts of stuck filaments, such mass extruded filaments being ideal for nonwovens.

Accordingly, the instant processes provide increased productivity yield per extrusion area of highly water absorbent and water retentive cellulosic fibers, such as the above described viscose fibers and hydroxyethyl cellulose fibers, at minimal cost. These fibers and filaments are admirably suited for formation of non-woven and disposable fabrics with sufficient strength, body, and softness to have an improved hand while retaining high receptivity for moisture pickup. Additionally, these processes provide other cellulosic fibers such as cellulose acetate which are also well suited for use in non-woven fabrics as low-cost binding fibers resulting from their thermoplastic nature.

Where reference is made herein to “water retention” values, it is to be understood that this term specifies the moisture remaining in and on a fiber after a re-wet fiber specimen is centrifuged for 10 minutes at an acceleration of 1,000 times normal gravitational acceleration. The water retention is reported as a percentage of the weight of the oven dry fiber. Thus, we have found that under such centrifuging treatment at 1,000 G, the nonwoven viscose fiber of the present invention has a water retention of at least about 120 percent (preferably ranging up to about 200 percent) and the hydroxyethyl cellulose fiber of this invention has a water retention of at least about 20 percent (preferably ranging up to about 600 percent). As a basis of comparison, water retention of cotton under comparable treatment is about 40 percent. As water retention of high performance rayon is about 70 percent and regular textile rayon is about 90 percent. It should be noted that never-dried fibers so produced hold substantially more water than can be reabsorbed after drying has somewhat collapsed the structure.

As described, the processes of the present invention are adaptable to either batch type or continuous fiber forming operations, although continuous processes are preferred. Furthermore, the instant processes can be employed to produce fibers and filaments having a wide variety of deniers
(e.g., from about 0.5 to about 15.0) depending upon such factors as the rate of extrusion of the fibers, the degree of ripeness of the cellulose solutions, the bath activities and the percentage stretching employed. However, in a preferred embodiment, the density of the fibers should be in a range of from about 1.5 to about 3.0.

The following examples are set forth for purposes of illustration only and are not intended to be construed as being limiting in any respect.

EXAMPLE I

This example illustrates a process of the present invention for preparing new and improved low-cost viscose fibers suitable for use in nonwovens and disposables. A viscose solution was prepared with a composition of 8 percent cellulose, 5 percent sodium hydroxide, and using 30 percent carbon disulfide in xanthation, in the absence of viscose additives or modifiers. A dissolving grade wood pulp cellulose was mercerized in 18.6 percent sodium hydroxide at a temperature of 26°C for 60 minutes, following which excess caustic was removed to provide an alkali cellulose: cellulose ratio of about 2.7. The pressed alkali cellulose was mechanically disintegrated at a temperature of 26°C and depolymerized (aged) in a controlled fashion to provide a fiber degree of polymerization of 350. Xanthation was carried out at 26°C. Dissolution of the cellulose xanthate in caustic was effected at a temperature ranging from 5°C to 20°C for a period of 60 minutes. The solution thus obtained did not have to be filtered through any of the filter media conventionally used in viscose-making and was screened through a stainless steel screen of 100×950 mesh to remove foreign particles or cellulose aggregates. The viscose was ripened at reduced pressure to remove air and to obtain a sodium chloride salt index of 6 for extruding at an extruding viscosity of 80 b.s.

The viscose solution thus prepared was extruded into a primary spin bath containing 8% H₂SO₄ and about 14 percent sodium sulfate. No other bath additive or modifier was required. This bath was maintained at a temperature of 50°C and length of tow exposure in the bath, adjusted to yield maximum stretchability of the fiber, was about 20 inches. Stretch was 50 percent.

The viscose was extruded into the primary bath from a container held under slight pressure, through a porous, sintered metal disc which provided a finite number of discrete flow paths of random diameter. The sintered metal material was "Poral", a sintered stainless steel material manufactured by Ugis Carbones, Grenoble, France. The flow paths through this sintered metal extruding material comprised about 35 percent of the total extrusion surface area. The size of the randomly shaped flow paths varied from about 20 to about 40 microns.

The resulting viscose fibers were cut into staple, purified, finished and dried by conventional methods. The fibers were analyzed and found to have the following physical properties: average denier (per filament) of 3, a conditioned tenacity of 2.5 g/d, a wet tenacity of 1.5 g/d, a conditioned elongation of 15 percent, a wet elongation of 18 percent, and a wet modulus (tenacity measured at 5 percent extension) of 0.2 g/d. This fiber had a water retention value of 120 percent.

EXAMPLE II

This is another example illustrating a process of the present invention for preparing new and improved viscose fibers suitable for use in nonwovens and disposables. A viscose solution was prepared with a composition of 11 percent cellulose, 5 percent sodium hydroxide, using 27 percent carbon disulfide in xanthation, in the absence of viscose additives or modifiers. A dissolving grade wood pulp cellulose was mercerized in 18.6 percent sodium hydroxide at a temperature of 26°C for 60 minutes, following which excess caustic was removed to provide an alkali cellulose: cellulose ratio of about 2.7. The pressed alkali cellulose was mechanically disintegrated at a temperature of 26°C and depolymerized (aged) in a controlled fashion to provide a fiber degree of polymerization of 250. Xanthation was carried out at 26°C.

Dissolution of the cellulose xanthate in caustic was effected at a temperature ranging from 5°C to 20°C for a period of 120 minutes. The solution thus obtained did not have to be filtered through any of the filter media conventionally used in viscose-making and was screened through a stainless steel screen of 100×950 mesh to remove foreign particles or cellulose aggregates. The viscose was ripened at reduced pressure to remove air and to obtain a sodium chloride salt index of 4 for extruding at an extruding viscosity of 80 b.s.

The viscose so prepared was extruded into a primary bath containing 12% H₂SO₄ and about 14 percent sodium sulfate. No other bath additive or modifier was required. This bath was maintained at a temperature of 25°C and length of tow exposure in the bath, adjusted to yield substantial stretchability of the fiber, was about 20 inches. Stretch was 20 percent.

The viscose fibers were extruded into the primary bath from a container held under slight pressure, through an uncoated double cotton twist stainless steel screen of 100×950 mesh. The flow paths or open area for extrusion in this screen constituted about 10 percent of the total extrusion surface area. The size of the individual holes was nominal 0.1 mil and the holes were oblong shaped.

The resulting viscose fibers were cut into staple, purified, finished and dried by conventional methods. The fibers were analyzed and found to have the following physical properties: a denier (per fiber) of 2, a conditioned tenacity of 1.5 g/d, a wet tenacity of 0.5 g/d, a conditioned elongation of 18 percent, a wet elongation of 20 percent, and a wet modulus (tenacity measured at 5 percent extension) of 0.1 g/d. This fiber had a water retention value of 150 percent.

The cross-sectional configuration of fibers prepared according to the procedures of the present example are illustrated in FIG. 1. These fibers exhibiting acute angular configuration offer a large ratio of surface to weight and are particularly useful in bonded nonwoven web formation with respect to bonding, absorbency and water retention.

EXAMPLE III

This example illustrates another process of the present invention for preparing new and improved viscose fibers. A viscose solution having a composition of 11 percent cellulose, 5 percent sodium hydroxide and xanthated using 28 percent carbon disulfide was prepared in accordance with the method of Example I. The xanthated cellulose was depolymerized to yield a fiber degree of polymerization of 275, the viscose was dissolved at a temperature of 15°C in a mixing cycle lasting 2 hours and screened through a 100×100 mesh stainless steel screen coated with "Teflon", a polytetrafluoroethylene product of the E.I. DuPont DeNemours Co., Wilmington, Del. The solution was then ripened to a sodium chloride index of 3 and extruded at a viscosity of 50 b.s., through a porous extrusion medium consisting of a "Teflon" coated stainless steel 100×100 mesh screen. The size of the square-shaped individual flow paths in the screen was 0.0055 inches wide and 0.0025 inches long and there were 10,000 flow paths for every square inch of extrusion surface. The flow paths through this extrusion medium comprised about 30 percent of the total extrusion surface area. The viscose was extruded into a primary bath containing 11.0% H₂SO₄, 14.0% Na₂SO₄ and 0.01 percent laurel pyridinium chloride, maintained at a temperature of 50°C. The fiber was regenerated in a secondary bath containing 3% H₂SO₄ at 95°C. Stretch between the first godet and the wash reel was of the order of 30 percent. The fiber was washed, cut into a staple length of 3 inches, desulfurized, finished with an oil emulsion and dried in the conventional manner.

The viscose fibers thus prepared were analyzed and found to have the following physical properties: a denier of 3 per
fiber, a conditioned tenacity of 1.5 g/d, a wet tenacity of 0.5 g/d, a conditioned elongation of 15 percent, a wet elongation of 18 percent, a wet modulus of 0.15 g/d, a water absorption value of 140 percent, and a cross-sectional shape exhibiting angularity.

EXAMPLE IV

This example illustrates a process of the present invention for preparing new and improved low-cost hydroxyethyl cellulose fibers suitable for use in nonwovens and disposables.

A solution of hydroxyethyl cellulose (hereinafter termed HEC) was prepared by dissolving 4 percent ethylene oxide substituted HEC in a caustic solution. The solution thus prepared contained 8.5 percent HEC and 5.7 percent sodium hydroxide and had a viscosity of 28 bsf. This solution was passed in a single stage through a cotton gauge 2 oz. filter in a filter press to produce an HEC solution having a degree of polymerization of 260. The solution was then extruded through a "Teflon" coated stainless steel 100×100 mesh screen, described in Example III, into a bath containing 11 percent sulfuric acid and 18 percent sodium sulfate at a temperature of 50°C. Tow exposure in the bath was 30 inches and stretch was 30 percent. The fiber was collected wet, cut into staple lengths, washed in hot water, finished with an oil emulsion and dried.

The HEC fibers thus prepared were analyzed and found to have the following physical properties: a denier (per fiber) of about 5, conditioned tenacity of 1.1 g/d, wet tenacity of 0.4 g/d, conditioned elongation of 25 percent, wet elongation of 28 percent, wet modulus of 0.1 g/d, water retention of 250 percent dried and 350 percent never-dried.

EXAMPLE V

This example illustrates the preparation of new and improved hydroxyethyl cellulose fibers by a process of the present invention.

An HEC solution was prepared by dissolving a 4 percent ethylene oxide substituted HEC crumb (D.P. of 200) in a caustic solution. The solution thus prepared contained 10% HEC and 5.6 percent sodium hydroxide. This solution was filtered, in a single stage, through porous filtration grade paper, aerated and then extruded in a bath composed of 14.0 percent sulfuric acid and 18 percent sodium sulfate at a temperature of 40°C. Extrusion was through a 100×950 mesh twill weave stainless steel screen to produce filaments which set quickly under these conditions without fiber sticking. The filaments were stretched 30 percent and wound up at 50 meters/min. The fiber bundle consisting of approximately 100,000 individual filaments was cut into staple of 1 ¼ inch length, washed, and treated with 0.1 percent Arquad, a lubricating anti-bonding and finishing agent. Then the staple fibers were partially dried, "opened" and dried to 90 percent bone dry condition.

When the staple fibers were re-wet with water and centrifuged at 1,000 G. for a period of 10 minutes, the HEC fibers had a water retention of 200 percent. The fibers had the highly angular string ray-like cross-sectional configuration illustrated in FIG. 2. These fibers exhibiting acute angular configuration offer a large ratio of surface to weight and are particularly useful in bonded nonwoven web formation with respect to bonding, absorbency and water retention. The fibers also had a conditioned strength of about 1 g/d, and a wet strength of 0.4 g/d.

The re-wet and dried fibers possessed substantial self-adhesive and bonding qualities either with or without "opening" the fibers. The dried fibers which were treated with "Arquad" could be opened and carded readily and air laid into a uniform web.

EXAMPLE VI

A dissolving grade sulfite wood pulp was treated with 0.1 percent Pluronic L-64 and converted to a 5 percent ethylene oxide substituted HEC crumb and aged to a DP level of 185. This crumb was dissolved in a caustic solution to yield a solution having a composition of 10% HEC and 5 percent sodium hydroxide. The solution was filtered through a fibrous polyamide nonwoven to remove undispersed material and desalted. The solution was then extruded through a screen of 100×950 mesh having a slot ½x2 inches blocking off a patterned hole arrangement to provide tow areas having approximately 50,000 holes each. The extruded filaments were stretched 25 percent without adhesion of filaments, cut to 1 inch staple, washed and treated with 0.1 percent Arquad, anti-bonding and finishing agent. Then, the staple fibers were "opened" and dried.

These fibers had a water retention of 300 percent as measured by centrifuge treatment at 1,000 G. for 10 minutes. The cross section of the HEC fibers exhibited the unique acute angularity of the fibers of the present invention similar to that in FIG. 2.

When the fibers were re-wet and pressure printed in a rectangular pattern, they bonded readily to themselves. A pad of fibers bonded together in this manner were agitated in excess water and were easily dispersed into a washable state.

EXAMPLE VII

This example illustrates a process of the present invention for preparing new and improved cellulose acetate fibers suitable as a component of the furnish used in preparing nonwovens and disposables.

One part shredded rayon-grade sulfite wood cellulose of 92-94 percent O.D. is reactivated for acetylation by treatment for 2 hours at 25°C. with 0.35 parts of acetic acid. The reactivated cellulose is added to a sigma-blade type mixer containing 2.8 parts of 97 percent acetic anhydride, 6 parts of glacial acetic acid and 0.125 parts of 97 percent sulfuric acid at a temperature of 5° C. The reaction temperature is increased to 48° C. over a 60-minute period and maintained at this temperature until the degree of polymerization of the cellulose acetate is about 250. At this point, the esterification reaction is stopped by the addition of 1.34 parts of a mixture of one part of water and 1.6 parts of glacial acetic acid.

The triacetate is hydrolyzed to 39.3 percent acetyl (2.4DS) at 48° C. At this point, the sulfuric acid in the acid gum is neutralized by the addition of magnesium acetate dissolved in 0.5 parts of 60 percent acetic acid. The precipitated magnesium sulfate is filtered off to give a reaction mixture that is then used directly for fiber extrusion that contains 13.1 percent cellulose acetate, 80.7 percent acetic acid and 6.2 percent water.

Fiber extrusion is accomplished by using air pressure to deliver the acid gum to a gear pump which in turn, forces the acid gum through a 400×400 mesh stainless steel screen. The holder for this screen allows a jetting area 0.125 inches in diameter.

The screen is immersed in a bath composed of 5 percent acetic acid, 15 percent sodium acetate and 80 percent water. Bath temperature is 5° C. Fiber travel distance in the bath is 22 inches and the extrusion rate is 10.0 meters/min, with a 20 percent stretch. Physical tests of the cellulose acetate fibers thus produced showed a denier of 2.5, a conditioned tenacity of 1.0 g/d, with a 30 percent elongation.

We claim:

1. A process for preparing water insoluble, self-bonding hydroxyethyl cellulose fibers having randomly oriented internal structures, water absorption of about 200-600 percent (on wet weight to dry weight basis) and water retention of at least about 200 percent (on wet weight to dry weight basis) comprising forming a fast setting, lean aqueous alkaline solution of hydroxyethyl cellulose having a uniform hydroxyethyl content of from 0.1-0.3 molar substitution and a degree of polymerization of from about 150 to 500, said aqueous alkaline solution containing from about 6-12 percent of said hydroxyethyl cellulose and from about 4-8 percent sodium hydroxide and having a viscosity of from about 20-200 poises
at 20° C., filtering said aqueous alkaline solution, then extruding said solution into a fast setting, non-retarding aqueous acid bath at about 20°–50° C., said acid bath containing about 10–15 percent sulfuric acid and about 12–20 percent sodium sulfate, said aqueous alkaline solution being extruded through a porous extrusion media, said extrusion media having flow paths comprising from about 10–35 percent of the total extrusion surface, and stretching by about 10–60 percent the resulting extruded fibers having peripheral shapes substantially conforming to the cross-sectional shapes of said flow paths of said extrusion media.