

[72] Inventors **Franklin James Evans**
Wilmington, Del.;
Charles Shambelan, Chattanooga, Tenn.
[21] Appl. No. **775,767**
[22] Filed **Nov. 14, 1968**
[45] Patented **Feb. 16, 1971**
[73] Assignee **E. I. DuPont de Nemours and Company**
Wilmington, Del.
Continuation-in-part of application Ser. No. 693,757, Oct. 6, 1967, now abandoned, which is a continuation-in-part of application Ser. No. 660,411, Aug. 15, 1967, now abandoned, Continuation-in-part of application Ser. No. 594,024, Nov. 14, 1966, now abandoned, Continuation-in-part of application Ser. No. 486,502, Sept. 10, 1965, now abandoned.

[54] **WATER-DISPERSIBLE NONWOVEN FABRIC**
38 Claims, 8 Drawing Figs.
[52] U.S. Cl. **128/284,**
128/290; 161/153; 264/88
[51] Int. Cl. **A61f 13/16**

[50] Field of Search..... **161/153,**
169; 128/284, 290, 296; 264/88, 120, 154

[56] **References Cited**

UNITED STATES PATENTS			
2,626,214	1/1953	Osborne	128/2904X
2,862,251	12/1958	Kalwaites	128/2904X
3,005,456	10/1961	Graham, Jr.	128/285
3,067,743	12/1962	Merton et al.	128/285
3,070,095	12/1962	Torr	128/284
3,370,590	2/1968	Hokanson et al.	128/284X

Primary Examiner—Charles F. Rosenbaum
Attorney—E. Leight Hunt

ABSTRACT: A soft, water-dispersible, nonwoven fabric of fibers secured in place by random fiber entanglement with at least 10 percent of the fibers chemically modified to have a surface that becomes slippery when wet with water to promote water-dispersibility. Processes for producing the above entangled fabrics which include both fiber modification to the water-sensitive form prior to fiber entanglement and subsequent to fiber entanglement are also disclosed.

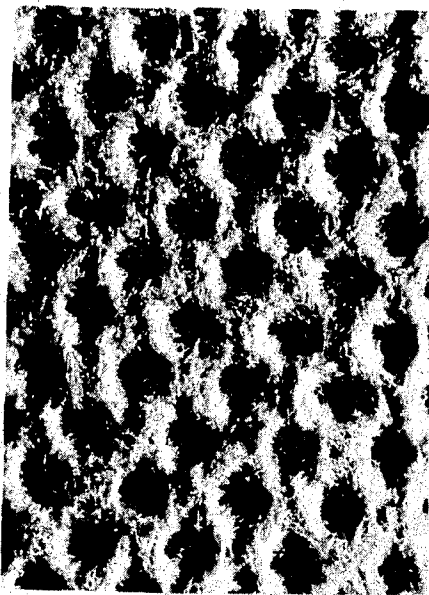


FIG. 1

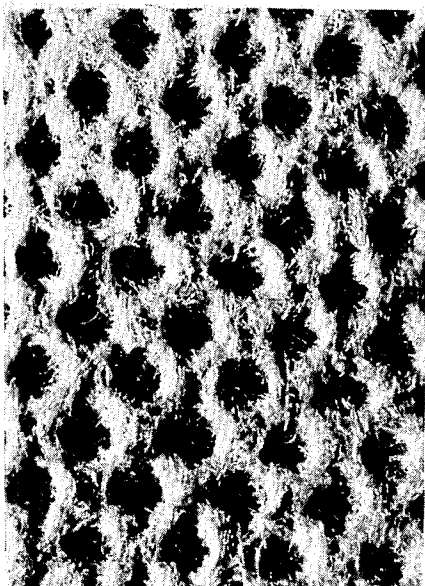


FIG. 2

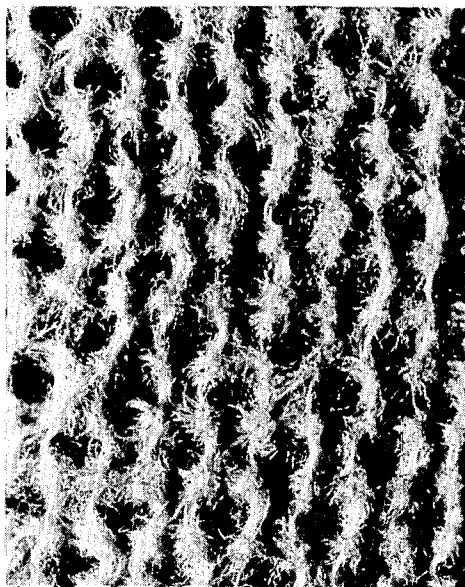


FIG. 3

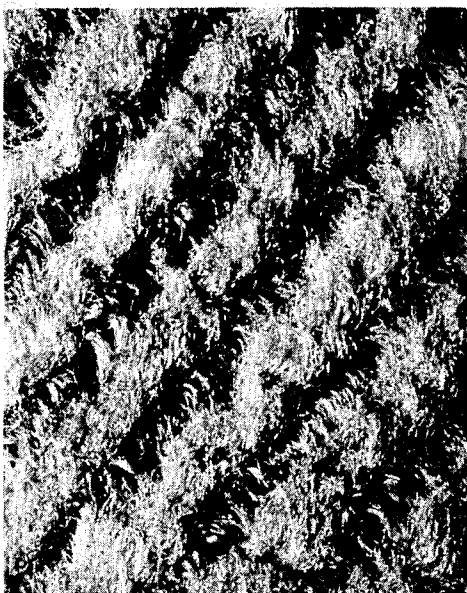
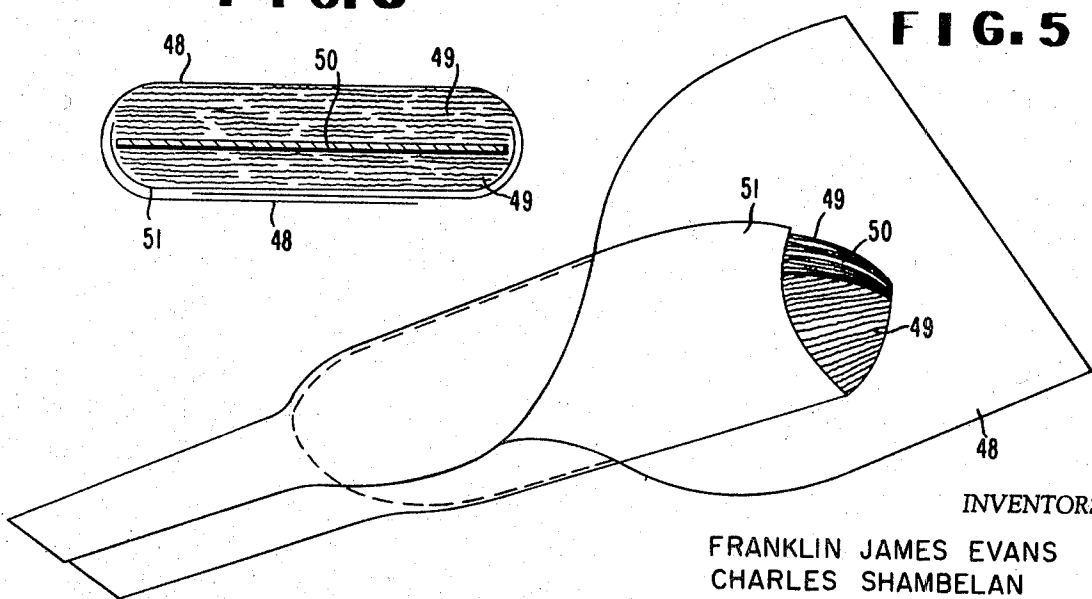
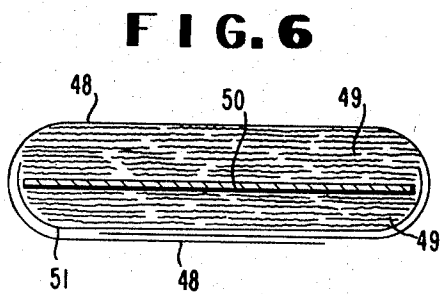
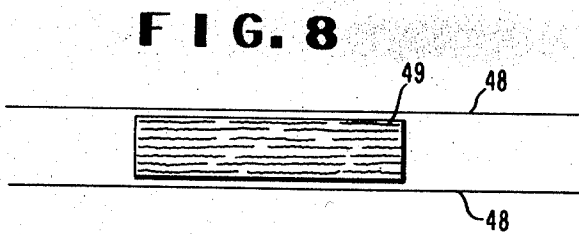
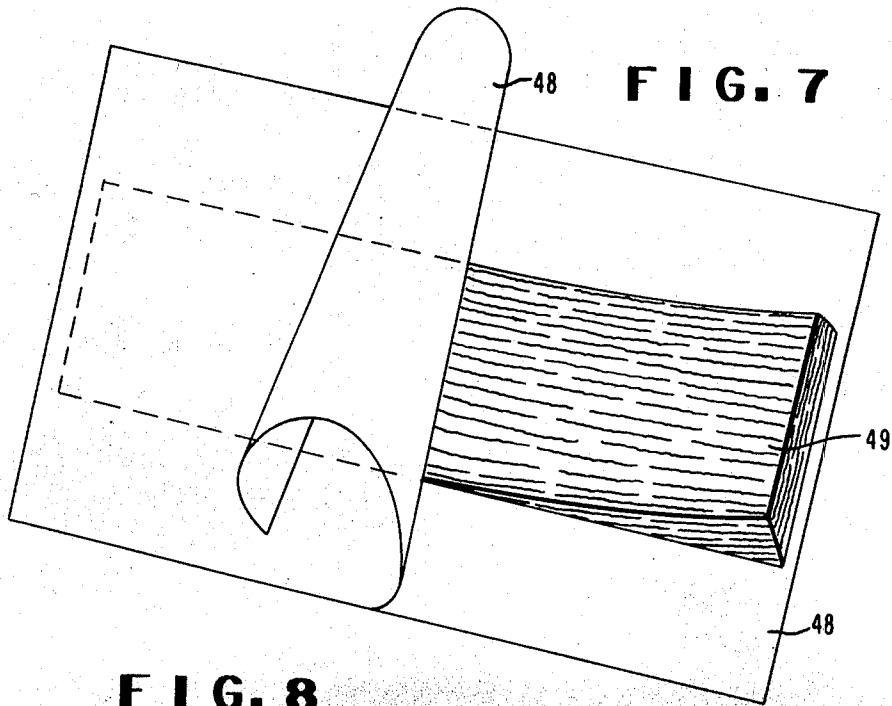


FIG. 4



INVENTORS
FRANKLIN JAMES EVANS
CHARLES SHAMBELAN
BY *E. Leigh Hunt*
ATTORNEY



INVENTORS

FRANKLIN JAMES EVANS
CHARLES SHAMBELAN

BY

E. Leigh Hunt

ATTORNEY

WATER-DISPERSIBLE NONWOVEN FABRIC

CROSS-REFERENCE TO RELATED APPLICATIONS

This is a continuation-in-part of our abandoned applications identified as follows: Ser. No. 693,757, filed Oct. 6, 1967 (which is a continuation-in-part of our application Ser. No. 660,411, filed Aug. 15, 1967 and now abandoned); Ser. No. 594,024, filed Nov. 14, 1966; and Ser. No. 486,502, filed Sept. 10, 1965. Reference is also made herein to a related patent of one of the inventors, Belgium Pat. No. 673,199, Jun. 2, 1966 and to Guerin, U.S. Pat. No. 3,214,819, dated Nov. 2, 1965. All are assigned to the assignee of the present application.

BACKGROUND OF THE INVENTION

The invention relates to soft, nonwoven fabric structures of fibers secured in place by random fiber entanglement to provide a pleasing appearance, drape and hand comparable to woven textile fabrics. The present invention is more particularly concerned with highly absorbent textilelike structures for single-use purposes, which have adequate strength and durability in use and are suitable for disposal in sewage systems after use.

Woven or knitted cotton fabrics have been used for diapers and other purposes where absorbency is desired. These have a relatively low capacity for absorbing fluids. Because of their cost, they are generally reused after an inconvenient and possible unhygienic laundering. The inconvenience of disposal after use is also a problem.

The present invention provides inexpensive, soft, highly absorbent, textilelike structures which are readily disposable in sewage systems. They are well adapted for sanitary fabric use, such as diapers, bandages and panties, where unusually high absorbency for body fluids and ease of disposal after a single use are especially desirable in a structure having a textilelike pattern of pleasing appearance and soft tactile hand. They have adequate strength and surface stability for such uses.

SUMMARY OF THE INVENTION

The fabric of this invention is defined as (I.) A soft, water-dispersible, nonwoven fabric of fibers secured in place by random fiber entanglement

A. comprising:

1. at least 10 percent fibers having a water-sensitive surface, the water-sensitive fibers having a wet coefficient of sliding friction (f_w) of not greater than 1.3 and a length of less than 3 inches, the f_w being measured on a nonwoven fabric consisting essentially of the water-sensitive fibers;

2. the balance of the fibers comprising non-water-sensitive fibers of less than about 0.5 inch length;

B. the fabric being characterized by:

1. a dry coherence value (C_h , dry) of greater than about 0.2,
2. a wet coherence value (C_h , wet) of less than about 0.6, with the C_h (wet) being less than the C_h (dry).

A more readily water-dispersible fabric of this invention (II) are the fabrics of I characterized by a C_h (wet) of less than 0.5 and the use of fibers having a water-sensitive surface which have a f_w of no greater than 1.1.

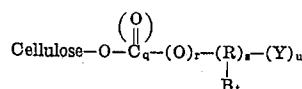
The most highly water-dispersible fabrics of this invention (III) are the fabrics of I characterized by a C_h (wet) of no greater than 0.3, an average wet strip-tensile strength of about 0.0 to 0.1 pounds/inch and the use of fibers having a water-sensitive surface which have a f_w of less than about 1.0.

The preferred fabrics of this invention have a weight of about 0.3 to about 5 oz./yd.² and contain at least 40 percent (and, more preferably, at least 50 percent) of fibers having a water-sensitive surface having ionizable groups (acidic groups as a free acid or partially or completely in the form of a salt of an alkali metal or ammonium ion). The preferred fabrics are characterized as follows:

- a. tensile strength in 1 percent NaCl solution of at least 0.05 lb./in.;

- b. a ratio of tensile strength in 1 percent NaCl to tensile strength in distilled water of at least 1.5;
- c. a bending length of less than about 3.0 cm.; and
- d. an internal bond strength of greater than about 0.08 ft. lbs.

The cellulosic species of the water-sensitive fibers exemplified are embraced by the following description: The water-sensitive cellulosic fibers are chemically modified cellulosic fibers having a degree of substitution within the range of about 0.1 to about 1.5 and are represented by the formula



wherein R is primarily a hydrocarbon group free from aliphatic unsaturation which may contain noninterfering hydrophobic substituents, either oxygen linkages and ester oxygen linkages in the chain; B₁ is a hydrophilic substituent; Y is selected from the group consisting of hydrogen, an acid group having ionizable hydrogen attached to oxygen, and monovalent cation salts of the acid; said formula meeting the following criteria: when Y is hydrogen and t is zero, R has less than 5 carbon atoms; when Y is hydrogen and t is one, R has less than eight carbon atoms; when Y is said acid group and t is zero, R has less than 10 carbon atoms; when Y is said salt of the acid and t is zero, R has less than 19 carbon atoms; q, r, and s are zero or one but when r is one, q and s are one; t is zero or any integer up to the number of replaceable hydrogens in R, and u is one or two.

Since the frictional characteristics of the water-sensitive fibers when wet promote water dispersibility by fiber-to-fiber slippage as is manifest from the low wet coherence value (C_h , wet) of the fabric, a wide variety of fibers are useful in the practice of this invention as only the surface characteristics of the fibers must be established. This is apparent from Examples 15 and 16 which illustrate useful products of this invention wherein the water-sensitive fibers are only modified on or near the surface of the fibers (heterogeneously), thereby forming a water-sensitive skin on the fibers.

Included among the noncellulosic fibers exemplified that can be modified to a water-sensitive form useful in the practice of this invention are polyvinyl alcohol fibers, acrylic fibers, nylon fibers, and casein fibers.

The process for preparing soft, water-dispersible, unitary nonwoven structures of this invention from initial layers of fibrous material, at least 10 percent of said fibrous material being fibers having a water-sensitive surface of less than about 3 inches length and up to 90 percent of said fibrous material comprising non-water-sensitive fibers of less than about 0.5 inch in length, comprises supporting the initial fibrous material on an apertured member; impinging fine, essentially columnar streams of a deswelling liquid for said water-sensitive fibers on the fibrous material at an energy flux of at least 23,000 ft. pounds/in.² sec.; and traversing the supported fibrous material with said streams at a Y value of 3×10^6 to 25×10^6 ft./min. to entangle the fibers into a structure having a weight within the range of about 0.3 to about 5 oz./yd.² with a dry coherence value (C_h , dry) of greater than about 0.2; said Y value being the weight of liquid used in lbs./min.; and said water-sensitive fibers having a wet coefficient of sliding friction of less than about 1.0 as measured on an entangled nonwoven fabric consisting essentially of the modified fibers.

Another useful process for preparing the product of this invention is defined in detail on page 28. In this process the fabric is first made of unmodified fibers and then the fibers of the entangled fabric are modified to a water-sensitive form.

The coherence value (C_h) is a measure or indication of the proportion of fibers that break (rather than disentangle or slip apart) when a long and short strip of a fabric is broken. The products of this invention have a wet coherence value (C_h , wet) in water that is less than the dry coherence value (C_h , dry) indicating that more fibers are disentangling in the wet

state than in the dry state. This behavior is in contrast to a product made of unmodified rayon or cotton alone. The wet coherence of entangled rayon or cotton nonwovens such as are used in Examples X—XVIII are greater than the dry coherence. This is presumably due to the greater friction between wet rayon or cotton fibers.

The broad invention (I) includes nonwoven fabrics of entangled fibers that will disentangle into smaller pieces in water under relatively mild agitation (such as used in the breakup time test of Example XXI) within about 100 seconds or under the turbulent conditions of the flow through institution-size sewer mains. The less dispersible products of this class may be soaked in a water tank with some agitation to promote breakup and then disposed into a large sewer main such as would be present in a hospital. The use of high-powered machinery such as paper beaters or grinders is not required to break up such fabrics.

The invention in its narrower scope (III) includes nonwoven fabrics of entangled fibers that are so highly water-dispersible that they can be disposed of in the usual household toilet by dipping a few times and then flushing. A C_h (wet) of no greater than 0.3 characterizes such behavior in soft water. When composed of homogeneously modified water-sensitive fibers, such fabrics preferably have single layer (S.L.) absorbencies (defined later) of at least about 2.0. The preferred products of this class have a C_h (wet) value of less than about 0.15 and are flushable in the form of diaper-size portions (such as 13 in. $\times$ 17 in.) in a household toilet without the need of predipping before flushing. Such preferred products contain at least 40 percent of water-sensitive fibers and have a salt-free fabric weight of about 0.3 to 5 oz./yd.².

The invention in its intermediate scope (II) includes nonwoven fabrics of entangled fibers that have a water-dispersibility greater than class I as measured by a breakup time (B.U.T.) of 40 seconds or less.

The products are termed "fabrics" since they have the appearance, handle, and elongation-at-the-break (at least 90 percent in at least one direction) of textile fabrics. The preferred products have an average elongation in both directions of at least 20 percent. Preferred products have a drape as measured by a bending length of less than 3.0 cm. in both directions. The fabrics are not papers and do not resemble papers at all in their essential properties.

Products having a C_h (dry) of at least 0.2 generally have average dry tensile strengths of about 1 lb./in. or greater with abrasion resistance which is adequate for many uses. The broad class of products includes products that may be satisfactory as absorbent pads, for example, while lacking the strength to be used for such products as diapers. In general, the dry integrity will increase with higher C_h (dry) values.

Another class of preferred products is characterized by a sufficient resistance to disentangling when wet with body fluids such as urine or menstrual fluid that the fabric can survive the stresses accompanying body movements and removal of the used fabric without disintegration. Such products have an average tensile strength in synthetic urine of at least 0.04 lb./in. and more preferably at least about 0.2 lb./in. Such products when comprised of homogeneously modified fibers are also characterized by an S.L. absorption of less than about 13.

It is to be understood that although a fabric may contain strips of non-water-sensitive fibers, the portion of the fabric having water-sensitive fibers is to be considered for determining if the fabric falls within the scope of applicant's invention and claims.

Although the products of the invention as shown in the examples utilize water-sensitive fibers that are uniform along their length, it will be understood that the products can be surface-treated, e.g., cross-linked, to change the wet hand, without substantially changing the dispersibility of the product.

WATER-SENSITIVE FIBERS

Fibers which are slippery in water as determined by a wet coefficient of sliding friction of no greater than about 1.3 (preferably no greater than about 1.0) as measured on an entangled nonwoven fabric consisting essentially of the fibers are generally suitable. The fibers must also be insoluble in synthetic urine.

The fibers may be homogeneous, i.e., have a substantially uniform chemical composition throughout their cross section, or may be heterogeneous, thereby having an adherent coating or skin of a water-sensitive composition. The composition of the skin or the entire fiber (if homogeneous) should be such that a homogeneous fiber of that composition will have a S.L. absorbency of at least 1 and preferably at least 2.

The water-sensitive polymers which form the fibers and the water-sensitive fibers themselves are well known. An easy and well-known route to such products is by the modification of cellulose. Suitable chemical reactions involving derivatives of the hydroxyl group are discussed in "Cellulose" Vol. II by Ott and Spurlin, pages 673 to 1026 (Interscience Publishers, Inc., N.Y., 1954) and water-soluble derivatives given. Also useful is oxycellulose. Further products are cited in "A Survey of Soluble Chemically Modified Cotton Fiber" by R. Reinhardt et al. in Textile Research Journal 27, 59—65 (1957). The chemical modification of cellulose and other polymers is discussed at length in "Chemical Reactions of Polymers" by E. M. Fettes (Interscience Publishers, N.Y., 1964).

The reactions used to convert cellulose to a water-sensitive derivative such as esterification and etherification can be applied to noncellulosic polymers that have reactive hydroxyl groups along the chain. Such polymers are readily obtained by hydrolysing polymers such as polyvinyl acetate (to give polyvinyl alcohol) or copolymers of vinyl acetate with monomers such as ethylene, vinyl chloride and methyl methacrylate.

Another route to water-sensitive fibers is to hydrolyse polymers having pendant side groups of esters, amides and nitriles to carboxyl groups or amides. Polymers and copolymers from acrylonitrile, alkyl acrylates, alkyl methacrylates, acrylamides and vinyl phosphonate are suitable for this purpose. The same type of polymer can also be made by copolymerizing acidic monomers such as acrylic acid, methacrylic acid, maleic anhydride, styrene sulfonic acid, and vinyl sulfonic acid, and the like with another monomer such as acrylonitrile, styrene, vinyl chloride or vinylidene chloride. The combinations of monomers and the appropriate polymerization techniques are well known.

Another route is to start with known water-soluble fibers such as polyvinyl alcohol, polyacrylic acid, polymethacrylic acid, polyacrylamide, N-alkyl derivatives of polyacrylamide such as N-isopropyl- and N,N-dimethyl-, polymethacrylamide, polyethyleneoxide, polyvinyl methyl ether, amylose, sodium alginate, polystyrene sulfonic acid, polyvinyl sulfonic acid, polyallyl fibriclike acid, polyvinyl tetrazole, polyvinyl benzoic acid, polyvinyl pyrrolidone, polyvinyl amine, polyvinyl pyridine, poly cis dimethoxy ethylene and polymethylol acrylamide and cross-link them by ionic bonds (such as calcium ion on an acid polymer), or covalent bonds with difunctional reactants (diamines, glycols, etc.) or by the application of high energy ionizing radiation (e.g. by a 2 MEV Vandegraff electron accelerator). The water solubility of these fibers can also be reduced by increasing the molecular orientation e.g. by further drawing.

Another route to water-sensitive fibers is the irradiation bonding of acidic compounds to fibers as taught in U.S. Pat. No. 2,999,056 to Tanner.

The following condensation polymers are known to be water soluble:

poly(methylene thio dibutyramide)
poly(methylene thio divaleramide),
polymers from ethylene diamine, pentamethylene diamine, 2,2'-(ethylene dioxy) bis [ethylamine] or ethylene glycol with the diacid methyl imino diacetic acid;

polymers from 3,3'-Diamino-N-methyl dipropyl amine and such diacids as adipic, sebacic, dodecanedioic or isophthalic;

polymers from 2,2'-(ethylene dioxy) bis [ethylamine] and diacids such as adipic, 6-hydroxy undecanedioic, 1,4-piperazine diacetic acid, or oxy diacetic acid;

polymers from ethylene diamine or tetramethylene diamine with oxy diacetic acid;

polymers from amino acids as alpha amino propionic, beta amino propionic or N-methyl alpha amino propionic acid.

The above polymers can be cross-linked to reduce the water-solubility and still retain adequate water-sensitivity or the polymers can be made as copolymers with condensation monomers such as hexamethylene diamine and adipic acid to give a water-sensitive polymer directly.

One means of selecting cellulose esters and ethers is by the expression Cellulose-O-A where the compound A-OH is soluble in water at about 20° C. to the extent of at least 3 percent (by weight).

A can be an organic or inorganic molecule or a combination of organic and inorganic as shown below:

	A-OH	Solubility
Cellulose ethers:		
n-Butyl-.....	n-Butyl alcohol.....	7.9
Cyanoethyl-.....	Cyanoethyl alcohol.....	∞
Amino butyl-.....	2-amino butanol-1.....	∞
Carboxy propyl-.....	α-OH butyric acid.....	>20
Cellulose esters:		
Cellulose propionate.....	Propionic acid.....	∞
Cellulose hemi succinate.....	Succinic acid.....	6.8
Cellulose phosphate.....	Phosphoric acid.....	∞
Cellulose sulfate.....	Sulfuric acid.....	∞
Potassium salt of cellulose hemi phthalate.....	Potassium acid phthalate.....	10

Preferably the group A above and substituent Y of the formula on page 4 contain or consist entirely of an ionic group such as an acidic group as a free acid or partially or completely in the form of a salt of an alkali metal or ammonium ion. Such groups include -COOH, -PO(OH)₂, -OP(OH)₂, -OSO₂OH, -SO₂OH, -OB(OH)₂ attached to a carbon atom or groups as -PO(OH)₂, P(OH)₂, and -SO₂OH 2OH which may compose all of group A and are connected to the cellulose through oxygen.

Suitable acid groups for the cellulose ethers and esters include those groups having oxygen connected directly to carbon, sulfur or phosphorus (with molecular weights less than about 100). These acidic groups have an ionization constant for the first hydrogen of at least 1×10^{-5} .

Suitable hydrophilic substituents, i.e., B of the formula on page 4, for the cellulose ether or ester derivatives include sulfamyl, sulfonamide, phosphoramidate, phosphonamide, hydroxyl, carbamyl, amino, lower alkyl substituted amino, lower alkyl substituted amino (low molecular weight groups less than about 80). These groups are those which increase the water solubility of a given hydrocarbon when replacing a hydrogen.

The hydrophobic or noninterfering substituents referred to on page 4 are those groups that have little effect on the water-sensitivity of a cellulose compound such as halogen, nitrile, and nitro, but which may be present to a limited extent due to the synthesis used.

Suitable cellulose ethers include lower alkyl ethers such as methyl-, ethyl-, propyl-. Substituted ethers such as cyano ethyl-, cyano methyl-, hydroxy ethyl-, acry- amido-, amino ethyl-, dimethyl amino ethyl-, ethyl amino ethyl-, dinitro methyl-, poly(ethylene oxide)-. Preferred cellulose ethers containing acid groups include carboxy methyl-, carboxy ethyl-, sulfoxy ethyl-, sulfoxy butyl-, sulfato ethyl-, phospho propyl-, phosphate ethyl, malonic-, carboxy benzyl, sulfoxy benzyl.

Suitable cellulose esters include the lower alkyl esters such as formate, acetate, propionate, and substituted esters such as hydroxy acetate, hydroxy butyrate, α-hydroxy dimethyl propionate, ethyl carbonate, to mention a few.

Preferred cellulose esters containing an acid group include cellulose phosphate, cellulose sulfate, cellulose sulfite, cellulose phosphite, cellulose borate, cellulose hemi oxalate, cellulose hemi malonate, cellulose hemi succinate, cellulose hemi glutarate, cellulose hemi phthalate, cellulose hemi maleate and cellulose hemi adipate.

The above basic esters and ether-forming groups may have a wide variety of other substituents as desired. Such compounds are well known in the art and the water solubility of the theoretical A-OH can be found in handbooks and the chemical literature.

It will be understood that the water-sensitivity of these fibers is dependent upon the nature of the cellulose itself, the manner in which the fibers are formed, the degree of substitution (D.S.) of the cellulose, the distribution of the groups throughout the fiber and the degree of neutralization of any ionic groups.

In general the highest water-sensitivity for a given D.S. for a given compound is observed with fibers made by extruding a solution of the desired derivative. Thus extruded fibers of cyanoethyl cellulose of D.S. 0.15 to 0.80, hydroxyethyl cellulose of D.S. 0.1 to 0.9, sodium carboxymethyl cellulose of D.S. 0.1 to 0.3 and sodium carboxyethyl cellulose of D.S. 0.1 to 0.3 are suitable for making the most highly water-dispersible products.

Suitable fibers for the most highly water-dispersible products made by chemical modification of cellulose fibers include sodium carboxymethyl cellulose of D.S. 0.2 to 0.4, sodium cellulose sulfate of D.S. 0.2 to 0.4 sodium cellulose phosphate of D.S. 0.2 to 0.4 and sodium cellulose hemisuccinate of D.S. 0.2 to 0.4. In general a higher D.S. is required for this type of fiber to obtain a given degree of water-sensitivity than with the extruded fibers. The D.S. ranges for the above sodium salts are for completely neutralized derivatives. Partially neutralized or partially cross-linked (e.g., by polyvalent cations) derivatives of higher D.S. can also provide the desired water-sensitivity.

The bulk of the water-sensitive fibers present will preferably be textile fibers of about 0.5 to 15 denier (preferably less than 5 denier) with a length from about 0.25 to 2 inches. Smaller fibers made by chemical modification of paper making fibers, cotton linters and the like, may be present in amounts as large as 90 percent.

NON-WATER-SENSITIVE FIBERS

These fibers are the usual textile fibers of commerce (e.g. rayon, cotton, nylon, acrylic or polyester) and are characterized by a SL absorption (in fabric form) of less than about 0.9. The fibers should be no longer than about 0.5 inch. Fibers of 1 to 5 denier are most satisfactory but smaller fibers such as paper-making fibers and cotton linters can be used. Preferably the fibers are biodegradable since the products are to be dispersed in sewage systems. Cellulosic fibers such as cotton or viscose rayon are especially useful.

DESCRIPTION OF THE DRAWINGS

In the accompanying illustrations of the invention, FIG. 1 is a photomicrograph at 10X magnification of a representative portion of the fabric prepared as disclosed in Example I, showing the top or upstream face of the fabric by direct illumination,

FIG. 2 is a photomicrograph at 10X magnification corresponding to FIG. 1, but showing the bottom face of the fabric which is next to the screen support during production,

FIG. 3 is a photomicrograph at 10X magnification of a representative portion of the fabric prepared as disclosed in Example VIII, showing the top face of the fabric by direct illumination, and

FIG. 4 is a photomicrograph at 10X magnification corresponding to FIG. 3, but showing the bottom face of the fabric.

The dispersibility of the products are a function of the length, and the degree of water-sensitivity of the fibers, the composition and the extent of entanglement. Thus, at about 100, 40 and 10 percent water-sensitive fibers content the length of the water-sensitive fibers should not exceed 3 in., about 1.5 in. and 0.75 in. respectively when using a moderate degree of entanglement and fibers with a moderate degree of water-sensitivity.

FIGS. 5 and 6 illustrate a sanitary napkin comprising an absorbent core covered with the flushable fabric of the invention as the outer wrapper 48 which has end extensions forming attachment tabs in the usual manner. Within the cover there is the absorbent core formed of a composite of absorbent creped tissue 50 known as a cellulose wadding and loosely felted fibrated wood pulp 49 commonly known as fluff. The thickness and composition of each of these absorbent layers may vary considerably and may be interchanged in position or have one or the other type omitted depending on how much total absorbent capacity and softness is desired in the core. All components of the core must be water dispersible. Between the core and the bottom and sides of the napkin wrapper is creped tissue 51 which has been treated with a hydrophobic material to act as a menstrual fluid barrier.

FIGS. 7 and 8 illustrate a diaper comprising an absorbent core covered with a wrapper 48 consisting of the flushable fabric of the invention. The core consists preferably of wood fluff 49 but can be any fiber blend or composite which is water dispersible.

FIGS. 1 to 4 illustrate products of this invention of fibers secured in place by random fiber entanglement in a repeating pattern of ordered fiber groups arranged in parallel rows interconnected by fibers extending between adjacent rows to form a unitary structure. A wide variety of such patterns can be prepared by using different patterning supports, as disclosed in the applications referenced previously. The product of FIGS. 1 and 2 is prepared on a plain weave, 24 x 24 mesh/inch woven wire screen. The resulting product has ordered fiber groups arranged in regular parallel rows extending in two principal fabric directions and interconnected in a network by fibers randomly entangled with each other to defined an ordered geometric pattern of apertures. Fiber groups which form ridgelike protrusions on the face of the fabric are seen in FIG. 2. In this embodiment the ordered fiber groups follow paths located between adjacent parallel wires of the screen.

The product of FIGS. 3 and 4 is prepared on a grid of 40 mil diameter parallel rods spaced 12 to the inch. The repeating pattern of the product is characterized by groups of entangled fibers forming a regular pattern of ridgelike protrusions separated by grooves lying along parallel straight lines, the ridge-forming groups being interconnected by arrays of generally parallel fibers bridging under the ridge-separating grooves and entangled in adjacent groups. As disclosed in our previously referenced application, a somewhat similar product is prepared on oblong screens having five to 12 heavy wires per inch in one direction and about 3 to 5 times as many finer wires per inch in the other screen direction. The product has a repeating pattern of fiber bands of substantially greater width than thickness which extend continuously along substantially straight parallel axes spaced regularly in one fabric direction, the bands being interconnected by generally parallel fibers which extend laterally between adjacent bands and are held in place by fiber entanglement in the bands. The band-interconnecting fibers may be grouped in bundles spaced regularly along the edges of the bands and defining rows of apertures therebetween. Useful products can also be made which have no repeating pattern of ordered fiber groups or apertures and can resemble felts, for example. Such products can be made by using fine mesh screens such as 80 or more mesh per inch as a support for the fibers while entangling.

Process Considerations When Using Water-Sensitive Fibers As Starting Material

Products of the present invention can be produced by treating webs containing water-sensitive fibers with essentially columnar (i.e. having a total divergence of not greater than 5°) streams of a liquid exerting an energy flux of at least 23,000 foot-poundsals per square inch-second (9100 Joules/cm.² min.) at the web.

The energy flux of a stream (EF) can be calculated by means of the formula:

$$EF = 77 PG/A \text{ ft.-poundsals/in.}^2 \text{ sec.} \\ (= 98 PG/A \text{ Joules/cm.}^2 \text{ minute})$$

where P is the pressure at which the liquid is jetted in pounds per square inch gage (p.s.i.) [kilograms per square centimeter (kg/cm.²); G is the average volume flow for one jet stream in cubic feet (liters) per minute and A is the cross-sectional area of the streams in square inches (centimeters), at a location just prior to impact with the fibrous webs. This area can be determined from photographs of the stream with the webs removed, or by means of micrometer probes. Suitable streams can be obtained by propelling a suitable liquid at high pressures through small-diameter orifices under conditions such that the emerging streams remain essentially columnar at least until they strike the fibrous material. The intensity of the streams is sufficient to entangle the fibers firmly in place. The treatment is stopped short of the maximum obtainable entanglement to provide a product having a dry coherence value (C_h , dry) high enough for in-use integrity and a low wet coherence value (C_h , wet) so that the fibers will readily disperse in a sewage system. The low wet C_h results to a large extent from the combination of the low Y value and the slipperiness of the water-sensitive fibers when wet. This involves a critical control of the extent of treatment, taking into account the rate of application of energy by the streams, the rate at which the fibrous layer is traversed and the weight of the layer. For control purposes, it has been found sufficient to use a value, hereinafter designated the Y value, which is equal to the weight of liquid used in lbs./min., times the liquid velocity in ft./min., divided by the weight of the fibrous material treated in lbs./min. When the fibrous material is treated a plurality of times, the Y value is the sum of the values calculated for each individual treatment. The total treatment conditions should be adjusted to provide a Y value of 3×10^6 to 24×10^6 ft./min. whenever low-swelling fibers are present in the fabric. The weight of liquid used and the weight of fibrous material treated are measured for a convenient time interval. The liquid velocity is then calculated from the lb. liquid/min. by dividing by the density of the liquid in lbs./ft.³ and by the total orifice area in ft.².

A deswelling liquid is used in the streams to avoid swelling the water-sensitive fibers during treatment which would result in damage. A variety of aqueous salt solutions have been found suitable including sodium sulfate (17—20 percent), ammonium sulfate (20—30 percent), and sodium citrate (30 percent). Nonaqueous liquids can also be used.

Preparation of Cyanoethyl Cellulose Fibers

Regenerated cyanoethyl cellulose fibers having about 0.4 to 0.8 degree of substitution are especially preferred as the water-sensitive fibers. A suitable method for preparing them from wood pulp cellulose is as follows:

Conventional wood pulp sheets are steeped in 18 percent caustic soda solution at 27.5° C. for 45 minutes. The sheets of alkali cellulose are pressed to a 3/1 press weight ratio, shredded and stored at 0° C. They are allowed to warm to room temperature a predetermined time before the next treatment.

22 pounds of alkali cellulose that has been held at 0° C. (typically 15.8 percent NaOH, 31.2 percent cellulose with a D.P. of 300—400) is added to a 12-gallon baratte with water at 40° C. circulating through the baratte jacket. The baratte is rotated at 5 r.p.m. A mixture of 2.35 pounds (1066 grams) of

carbon disulfide and 2.75 pounds (1250 grams) of acrylonitrile is added to the alkali cellulose over a period of 20 minutes. When the temperature of the alkali cellulose reaches 19° C., the 40° jacket water is replaced with 20° C. water. When the temperature of the reaction mixture drops 1° C. from its highest value, the reaction is considered complete. The xanthate crumb is transferred to an agitated dissolving tank containing 111.4 pounds (51.8 kilo) of a 3.72 percent solution of NaOH in soft water and mixed for 60 minutes. The resulting viscose solution of cyanoethyl cellulose is transferred to plastic-lined boxes and stored at 0° C. for 16 to 24 hours before using.

The above viscose solution is then filtered through a filter press, deaerated, heated to about 20° C. and pumped through candle filters to a spinneret having 2200 orifices of 0.003-inch (0.076 mm.) diameter, and is extruded into a 48-inch (1.22 m.) long coagulating bath (typically 9 percent H₂SO₄, 9 percent ZnSO₄ and 17 percent Na₂SO₄ at 60–65° C.). The filament bundle is forwarded over a feed roll and a skew idler roll where regeneration of the yarn is completed. The regenerated yarn is forwarded to another feed roll idler-roll set where it is sprayed with a solution (35° C.) containing 17 percent Na₂SO₄ and 3 percent Na₂HPO₄ to neutralize the acid in the yarn and keep it in a deswollen condition. The neutral yarn is passed through squeeze rolls to remove excess solution and then wound up on packages at 60 yards (Ca 55 m.) per minute.

A sample of the dry yarn contains 30.5 percent of salts and 19 percent soluble (at 10° C.) material. The yarn has the typical following properties calculated on a water-insoluble-fiber weight:

Denier per filament (d.p.f.)	2
Tenacity, dry grams per denier (g.p.d.)	1.7
Elongation at the break, dry percent	9.7
Tenacity, wet g.p.d. (25° C.)	0.03
Water absorbency, g./g. at 25° C.	14.4
Urine absorbency, g./g. at 25° C.	11.0
Percent nitrogen	3.1
Degree of cyanoethyl substitution	0.4
Percent COOH	0.5
Degree of carboxyethyl substitution	0.018

Preparation of Initial Layer of Fibrous Material

The initial layer may be a web or batt of fibers assembled by known methods such as carding, random laydown, air deposition or by papermaking processes. The fibers may be disposed in random relationship with one another or in any degree of alignment. A plurality of layers of the same or different composition or orientation can be used. When prepared by assembling dry fibers, the water-sensitive fibers can be up to about 3 inches in length in the final assembly. Preferably the initial layer is formed on a papermaking machine, such as a Fourdrinier machine, from a slurry of water-sensitive fibers of 1 to 15 denier and 0.25 to 2.0-inch staple length. Conventional textile fibers of 1 to 5 denier and 0.125 to 0.5-inch staple length can also be included in the slurry. Slurries of staple fibers over 0.25 in. in length may be conveniently processed on a drum-type paper machine such as the "Rotoformer" manufactured by the Sandy Hill Corp., 29 Allen Street, Hudson Falls, N.Y. Conventional short wood pulp fibers may be substituted for part or all of the textile fibers in the slurry.

Preparation of the initial layer on a papermaking machine starts directly with formation of the slurry, since beating usually is not required. The fibers are added to water and well agitated to give a uniform slurry in the stock chest of the machine. A consistency of 0.1 to 0.40 percent fibers is suitable unless the fibers contain less than 30 percent of water-sensitive fibers. Slurries containing a high proportion of nonwater-sensitive fibers, e.g., 90 percent of conventional textile fibers, are prepared at consistencies of about 0.03 to 0.09 percent fiber. The slurry is fed to the headbox of the papermaking

machine and is diluted with about an equal volume of water en route to the headbox. A double headbox supplied with different compositions of stock slurry can be used to prepare a laminated initial layer. From the headbox the slurry is deposited on the wire of the papermaking machine to form a fiber layer. The machine wire may be several feet wide and of conventional type, e.g., 7.5-mil diameter wire in a 70 × 52 mesh/inch construction. The fibers drain as they are conveyed on the machine wire, the drainage being assisted by the usual suction boxes. The partially drained layer of fibers is sprayed with a 17 to 20 percent aqueous sodium sulfate solution, or other deswelling liquid, to cause deswelling of the highly water-absorbent fibers in the layer. For example, the layer may be sprayed with banks of nozzles at 2, 3 and 4.75 feet from the breast roll of the machine, using about 1.3 gallons per minute of the deswelling liquid. The deswollen layer passes from a vacuum couch roll to a pressing section where water and excess salt are removed. The layer may be dried on rolls heated to 120° C. and stored for subsequent treatment. Alternatively, the damp layer can be passed directly to the fluid treatment with high energy flux streams to form the products of this invention.

Apparatus suitable for use in the continuous treatment of fibrous layers to entangle the fibers into the structures of the present invention is described in the examples.

The pattern produced by the hydraulic entangling is dependent upon the design of the apertured patterning member. The term "apertured member" includes any screen, perforated or grooved plate or the like on which the fiber assembly is supported during the hydraulic entangling and which by reason of its apertures and/or surface contours influences the movement of fibers into a pattern in response to the fluid streams. The apertured member may have a planar or nonplanar surface or a combination of the two types.

Plain weave screens of from 3 to 80 mesh (wires per inch or per 2.54 cm.) having wire diameters of from 0.005 to 0.040 inch (0.127 to 1.02 mm.), and having from about 10 percent to 98 percent open area are suitable apertured members and in general afford the type of pattern shown in FIG. 3.

Twill weave screens and Dutch twill of the above general construction may also be used.

An assembly of a grid of parallel rods contiguous with a second grid and so oriented that the rods of each grid are not parallel to the rods of the other grid can be used as an apertured member. Structures of the type shown in FIGS. 3 and 4 can be made using such supports.

Process Using non-water-sensitive Fibers As The Starting Material

Products of this invention can be made by preparing an assembly of non-water-sensitive fibers, entangling the fibers to give a nonwoven fabric and then chemically modifying some or all of the fibers in the fabric to a water-sensitive form. Suitable reactions on cellulose include: chloroacetic acid and alkali to produce carboxymethyl cellulose; alkali and ethylene oxide to produce hydroxyethyl cellulose; alkali and acrylamide or acrylonitrile to produce cyanoethyl cellulose; phosphoric acid and urea to produce cellulose acid phosphate; succinamic acid to produce cellulose hemi succinate; urea and sulfuric acid (or sulfamic acid) to produce cellulose sulfate; and the like. Such reactions work well with rayon; with cotton it may be necessary to degrade the cotton, mercerize it or use stronger reaction conditions.

In addition to the preparation of a cellulose derivative in the nonwoven form it will be obvious to one skilled in the art that many other reactions are suitable for preparing water-sensitive fibers from noncellulosic fibers such as the hydrolysis of polyvinyl acetate fibers to polyvinyl alcohol, the hydrolysis of cross-linked polyvinyl alcohol fibers, hydrolysis of acrylic fibers, the use of the above-mentioned reactions for rayon on noncellulosic fibers having hydroxyl groups such as copolymers containing vinyl alcohol units, and the like.

It will be understood that the nonwoven fabrics to be treated can contain fibers that will not be chemically modified by the reaction used.

The assembly of non-water-sensitive fibers is conveniently made into a nonwoven using hydraulic entangling as previously discussed. The critical limits of entangling (Y values) for the assemblies containing water-sensitive fibers do not apply in the case of assemblies of 100 percent inert fibers and a greater entangling treatment can be applied.

The chemically-modified nonwoven fabric may be treated with deswelling salt solutions and dried in the manner already described.

Products can also be made by entangling a web of fibers in the acid form with water and then converting the entangled fibers to a more water-sensitive salt form.

Water-dispersible nonwoven fabrics of this invention can be made by (1) assembling a layer comprising cellulosic fibers of less than about 5 inches in length, (2) treating the fibrous layer with essentially columnar streams of a liquid to entangle the fibers into a coherent nonwoven fabric, and (3) chemically modifying the nonwoven fabric with water-sensitive groups to reduce its wet coefficient of sliding friction, the conditions of the process being selected so that the natural logarithm of the breakup time in seconds (A) (Test defined in Example XXI) in the following equation is less than 4.6.

$$A = 1.378 + 0.0180 R - 1.60 L + 0.731 f_w \\ + 0.00292 RL - 0.0000480 R^2 \\ + 0.476 L^2 + 0.847 f_w^2$$

where R is the yield resistance time (YRT - defined in Example XXVI) of the cellulosic nonwoven fabric before chemical modification, L is the weight average length of the fibers in inches, and f_w is the wet coefficient of sliding friction of the chemically modified nonwoven.

A higher degree of water dispersibility can be achieved by using conditions to give a value of less than 3.7 for A . More preferably, the value of A should be less than 3.4 to yield products of higher water-dispersibility which can be flushed in household toilets.

R is a measure of the entanglement of the cellulosic nonwoven fabric. The value of R for a given fiber layer increases as the duration of the liquid entangling treatment is increased (i.e., by slower speeds or by repeated treatments) for a given set of conditions. The value of R increases as the pressure on the liquid streams is increased for a given orifice. The value of R increases as the diameter of the liquid orifice is increased at a given pressure. R is dependent upon the average fiber length (L). In general, R increases with L when all other conditions remain constant.

Preferably R should have a value of at least 10 to provide integrity and should preferably be less than about 400. The fiber length (L) should preferably be less than 2 inches.

The coefficient of sliding friction (f_w) is a measure of the chemical modification of the nonwoven. In general for a given cellulosic nonwoven, the f_w decreases as the extent of the modification with the water-sensitive groups increases up to a certain level. This modification is controlled by the usual variables of a chemical reaction—concentration, time, and temperature.

The products of this invention are capable of many modifications. They can be tinted, printed with designs, and made in a wide variety of patterns.

In addition to the use of simple nonwoven fabrics of this invention for sanitary purposes various combinations are possible. The nonwoven fabric may be used as a cover for a very high absorbency pad for special applications.

Characterization Procedures

Papers made by deposition of fibers on a moving screen such as a Fourdrinier machine usually show different proper-

ties in the continuous direction (i.e., direction of travel on the paper machine) termed "machine direction" (MD) and in the direction transverse to travel termed "cross direction" (CD). These terms are also applied in a like manner to nonwoven fabrics made by passing a long web of fibers under a hydraulic entangling assembly in one direction (MD). Products made from hand paper sheets usually have very similar properties in any two directions at right angles and the direction is not designated.

Tensile Strength and Elongation

Tensile strengths and elongations are measured on an Instron testing machine using a 2-inch length between the jaws and elongating at 50 percent per minute. Results for Examples 1 to 9 are determined on 0.5 inch wide strips while the remainder are determined on 1.0 inch wide strips. In some cases the breaking force determined in the coherence value test is used to calculate the tensile strength; such values are marked with "c." All tensiles are normalized to pounds/inch of width. All three methods give approximately the same results.

Samples are soaked for 5 minutes in distilled water at room temperature and then clamped in the tester and broken in air to determine wet tensile strength. These values are referred to as the wet strip-tensile strength. The same method is used to determine tensile strength after soaking in synthetic urine or other liquids.

Coherence Value (C_h)

The coherence value (C_h) is a measure of the degree to which the fibers within the fabric break as opposed to slipping apart whenever a long strip of the fabric is pulled apart.

The test, in essence, comprises measuring the tensile strength of a long strip of the fabric and dividing this value by the tensile strength of the strip as determined at substantially zero length (Instron clamping jaws together). It is readily apparent that during the zero length strip-tensile measurement fiber slippage cannot occur and hence the maximum tensile strength for the strip is obtained. Therefore the maximum (C_h) is 1 with lower C_h values indicating fiber slippage. A C_h (wet) of no greater than about 0.6 is necessary to achieve the desired dispersibility of the products of this invention while a C_h (dry) of at least 0.2 is required to yield adequate in-use strength.

Samples of salt-free fabrics are cut into 1.6 inch (4.1 cm.) wide strips. The strips are then cut to about 2-inch and 1-inch (5 and 2.5 cm.) adjacent samples. The longer sample is broken using 1.5-inch (3.8 cm.) gauge (distance between the rubber-coated jaws of the Instron Tester). The shorter sample is broken using zero gauge. The coherence value (C_h) is the breaking strength of the longer sample divided by the breaking strength of the shorter sample. The average results from at least 3 pairs of such breaks in each direction of the fabric is reported as C_h in the examples.

For C_h wet values, a bath of distilled water is raised a round the sample which is clamped in the Instron machine and the sample soaked for at least 1 minute before breaking it in the bath. All samples are elongated at a rate of 0.5 inch (12.6 mm) per minute.

Salt-free fabrics for use in coherence value determinations are prepared by extracting with a solution made from 60 volumes of methanol and 40 volumes of water. About 100 g. of the fabric, divided into 4 portions is placed in 1500 ml. of solution, then gently worked to insure saturation, soaked 15 minutes, and then gently squeezed and pressed between blotters. This is repeated using fresh solution until the solution is clear (normally 3 washes). Three additional treatments are then given followed by a final treatment in 100 percent methanol. The blotted samples are opened and dried in air. Twisting or pulling of the fabric during the treatments are avoided. The final fabrics are soft and conformable.

Single Layer Absorbency

The single layer (designated SL) absorbency method has been devised to measure fiber absorbency in the fabric state and to minimize interstitial water. A 3 in. $\times$ 3 in. (7.6 $\times$ 7.6 cm) sample in a 100 mesh screen basket is immersed for 1 minute in 150 ml. of a 0.1 percent aqueous NaCl solution and the basket then dipped in and out of the solution 5 times and drained for 15 seconds. The procedure is repeated in a fresh salt solution and the sample soaked for 5 minutes before draining the basket for 30 seconds. The wet sample is dropped from the basket onto a dry blotter and immediately dropped from the first blotter to a second dry blotter and covered with another dry blotter. The 2 blotters (5 $\times$ 5 in.) and the enclosed sample are covered with a 5 in. $\times$ 5 in. aluminum plate and a weight added on top to give a total weight for the weight and plate of 3 kg. The sample is pressed at the 3 kg. pressure for 5 minutes and weighed immediately. The sample is dried for 2 hours in a hot air oven at 125° C., cooled in a dessicator and weighed immediately. The SL absorption is obtained by dividing (pressed wet weight minus dry weight) by the dry (final) weight to yield grams of solution/grams of dry fabric.

If the SL absorbency is less than 1.0 g./g., the determination should be repeated using blotters that have been slightly dampened (about 0.2 g/g of dry blotter) with a fine spray of the salt solution. Such a procedure avoids a false reading caused by some of the solution from the fibers (as well as interstitial) being absorbed by the dry blotters.

Conversely, if the blotters appear to be fully soaked the procedure should be repeated with the addition of a paper towel on the outside face of each blotter as all of the interstitial solution is not removed otherwise.

Water-Sensitive Fiber Frictional Characteristics

The coefficient of sliding friction, (f) (or kinetic friction) is the ratio of the force (F) needed to pull one surface over another to the force (N) normal to the surfaces in contact.

A piece of fabric of about 2 in. $\times$ 6 in. (5.1 $\times$ 15.3 cm.) is placed on the horizontal bottom of a metal tank with the longer length in the direction of sliding and one end held in place with a heavy metal block. A second portion of fabric of 1 in. $\times$ 3 in. (2.5 $\times$ 7.6 cm.) is wrapped around a sled so that the bottom, front and back faces of the sled have 1 thickness of cloth and the top face has 1 to 2 thicknesses. A 5 gram brass weight is placed on top of the fabric. The sled consists of a 1 $\times$ 1 $\times$ 0.063 in. (25 $\times$ 25 $\times$ 1.6 mm.) piece of poly(methyl methacrylate) plastic with an outer layer of crocus cloth (abrasive face out) held to the plastic with double-faced adhesive tape. The sled has a filament attached to each side near the forward edge forming a yoke. A 0.1 percent aqueous solution of NaCl is slowly added to the tank to a depth of about 8 mm., the sled, fabric and weight placed on top of the lower fabric, the yoke attached to a filament leading under a pulley in the tank and upward to the crosshead of an Instron Tester and the tester started so as to pull to sled at a rate of 2 in. (5.0 cm.) per minute. The sliding is continued for about 90 seconds. The charted force from 30 seconds to the end is averaged and reported as F after suitable correction for the force necessary to overcome the friction of the pulley. Force N is the sum of the sled weight (1.9 g.), the fabric (normally about 0.15 g.) and the 5.0 g. weight, minus the buoyancy of the solution. The solution completely covers the sled and about 50 percent of the weight. (Total effective force, N , 5.8 gms).

The above conditions should be followed since f varies with N . The results of this test are reported as the wet coefficient of sliding friction (f_w).

The test is made on an entangled nonwoven fabric consisting of 100 percent of the water-sensitive fibers and having a C_d (dry) of at least 0.2. Fabric pattern or structure variation has been found to have little effect on the sliding friction with essentially planar fabrics.

Products of this invention have been made of fibers which have f_w values (as measured on an entangled nonwoven fabric containing 100 percent of the fibers) of from 0.3 to 1.3.

The F_w values of rayon increase as the amount of entanglement of the nonwoven fabric is increased and increase as the average fiber length is decreased at a constant entanglement condition. Thus, at a near minimum entanglement condition f_w varies from 1.30 for 1.38 inch average fiber length to 1.44 for a 0.38 inch average fiber length. At a near maximum entangling condition, the f_w ranges from 1.77 to 1.90 for the same blends respectively. When rayon nonwoven fabrics are modified to a water-sensitive fiber form, the f_w is no more than about 80 percent of the starting rayon f_w values for the lesser dispersible products and can be as low as 40 percent or lower of the starting values for the highly dispersible products.

Bending Length

Bending length is 0.5 the length of a strip of sample that bends under its own weight to a 45° angle. It is determined on 1 $\times$ 6 inch sample of a Drape-Flex Stiffness Tester (made by Fabric Development Tests, Brooklyn 32, N.Y.). The procedure utilized for the bending length determination is as described in American Standards of Testing Materials (ASTM) test number D 1388-55T. Unless otherwise stated the CD value is given as it is usually lower than the MD value.

Fabric Weight

Fabric weights are expressed in ounces/square yards, hereafter designated as oz./yd.², and are based on the weight of the fibers present minus water-soluble impurities.

Liquid Absorption

The liquid absorbency of a sample is determined by soaking a small sample in an excess of the liquid at 25° C. unless otherwise designated (1 g. in 3000 g. of liquid). The sample is removed from the liquid and spread out to cover a 5 $\times$ 5 cm. area on bleached sulfite blotter paper. The sample is placed between layers of blotter paper and loaded with a 3 kilogram weight to give a pressure of 120 g./cm.². Pressure is applied for 5 minutes after which the sample is removed and weighed, giving the wet weight. Then the sample is dried to constant weight using a Noble and Woods sheet dryer at 100° C. Absorbency equals the water absorbed (wet weight minus dry weight) divided by the dry weight. In the case of absorbency determinations in body fluids, the dry weight must be corrected for solids dissolved in the liquid absorbed.

In examples 1—8, absorbencies of urine are measured in a salt solution of essentially the same composition as human urine [16 g. NaCl, 35 g. urea, 2 g. MgSO₄, and 3 g. Ca(H₂PO₄)₂H₂O, per liter of solution in distilled water.] In examples 9—18, the "synthetic urine" comprises a salt solution containing 10 g. NaCl, 24 g. urea, 0.6 MgSO₄, and 0.7 g. calcium acetate monohydrate per liter of solution in distilled water. All water absorbencies are measured in distilled water unless otherwise stated. Except for examples 1—8, "synthetic urine" is of the composition as used in examples 9—18.

Dispersibility

The dispersibility is determined in a 250 ml. filter flask having an added side arm at the bottom of the conical wall and containing a magnetically rotated bar. The bar is 3.8 cm. long by 8 mm. in diameter, weights 11.73 grams and is rotated at 500 revolutions per minute. A 3 $\times$ 3-inch sample is folded in half and inserted under the surface of the water (at the top side arm). Tap water at about 25° C. is added through the bottom tube at a rate of 0.70 liters/minute for period of 2 minutes. The effluent liquid from the upper side arm is filtered and the residue dried to constant weight at 100° C. to give the weight of fibers dispersed. The contents of the filter flask are filtered after the test and dried to yield the weight of undispersed fibers. The percent dispersibility is equal to 100 times the weight of fibers dispersed divided by the total weight of fibers recovered. Conventional toilet tissues have a dispersibility of 7 percent.

Flushability

The flushability of a sample is determined by dropping a 10 × 26-inch sample that has been folded to 10 × 13 in. into the bowl of a household toilet (Model F2122 made by the American Radiator and Standard Sanitary Corporation of New York, N.Y.) and flushing. The discharge from the toilet is passed through a length of horizontal flange pipe 2.33 ft. (71 cm.) long and of 10.8 cm. inside diameter containing an artificial obstruction. The obstruction is constructed of standard flattened expanded metal with 0.5 in. (1.27 cm.) wide perforations, formed into a cylinder of 1 ft. (30.5 cm.) long and about 10.8 cm. in diameter and provided with 41 inside projections randomly distributed and made by making parallel pairs of cuts about 0.25 to 0.75 in. (0.63 to 1.9 cm.) long and about 0.3 in. (0.75 cm.) apart, and bedding the cut sections to stand perpendicular to the walls of the cylinder. One flushing gives a flow of about 20 liters of water in 7 to 8 seconds. The toilet is flushed 3 times for each sample. The percentage of the sample that passes the hooks in the glass pipe is estimated and recorded after each flush. A sample is termed "flushable" if at least 60 percent passes by the hooks after 2 flushes. At least 85 percent of a sample of the preferred products passes by the hooks after 2 flushes.

It has been observed that samples of examples 1—9 termed "Flushable" by the above test have a dispersibility of at least about 20 percent (i.e., in 2 minutes), or require less than 20 minutes to completely disperse in the small-scale dispersion test. Preferred products have a dispersibility of at least 40 percent.

For products of the type prepared in Examples I—V, the following relationship between breakup time (B.U.T.) and percent dispersibility is observed:

Percent Dispersibility	B.U.T.
>35	<30
>25	<40
>15	<100

Wickability

The wickability of a sample is determined by fastening the ends of a 2 × 5.5-inch strip to a perforated metal plate with rubber bands, resting the end of the plate under about 0.5 inches of "synthetic urine" at about 25° C. at an angle of 25° to the level of the water and noting the time in minutes for the liquid to wick the sample for a distance of 5 inches from the top of the liquid. Preferred products have a wickability in "synthetic urine" of less than 5 minutes. All product in the examples containing low-swelling fibers and made from a water dispersion have wickabilities of less than 30 minutes.

Loose Fiber Test

The loose fiber test is a measurement of the fabric integrity and is conducted by rolling a wet sample of the nonwoven fabric (9.8 × 12 cm.) located on a glass plate once with a dry rubber roller having a surface area equal to the sample. The fibers adhering to the roller are collected, washed with acetone, dried and weighed. Before testing, the sample is thoroughly wet with an excess of distilled water, covered with 2 paper towels and blotted by rolling lightly once with the above roller.

Nonwoven structures with no entanglement of the fibers lose about 0.12 gram by this test (or 14 percent of the original 0.86-gram sample). The preferred products of this invention show zero loss by this test. Useful structures may show a loss as high as 8 percent. Fabrics having a loss of less than about 10 percent are deemed to be unitary.

The invention will be better understood from the following specific illustrations of products and processes for making them. The examples are not intended to be limitative.

Examples

In the examples all solutions are aqueous unless otherwise stated. All screens are designed by mesh [(per inch or per 2.54 cm.)] and are understood to be the same in both directions, i.e. square mesh unless otherwise stated. The amount of liquid on a fabric is designated by pick up which is the grams of liquid on the fabric per gram of original dry fabric.

EXAMPLE I

Cyanoethyl cellulose fibers of 0.4 degree of cyanoethyl substitution, 2 denier per fiber, and 0.5-inch staple length are prepared as previously described. A mixture of 80 parts of these fibers (based on the dry weight of the water-insoluble fiber) and 20 parts of viscose rayon 0.25-inch staple of 1.5 dpf is added to water at 30° C. in a stock chest and well agitated to give a uniform slurry containing 0.24 percent fibers. This slurry is diluted to a consistency of 0.12 percent and fed to a headbox on a Fourdrinier machine as described previously. The machine wire is 31 inches wide made of 7.5-mil diameter wire in a 70 × 52 mesh/inch construction. The partially-drained fiber layer containing swollen cyanoethyl cellulose is sprayed with a 20 percent water solution of Na₂SO₄ at 40° C. at 2, 3, and 4.75 feet from the breast roll. The nozzles supply about 1.3 gallons/minute. The salt solution deswells the fibers. The wet paper is passed from the couching roll to a pressing section where it is pressed and is then dried on rolls heated to 120° C. The dry layer (A) has a fabric weight (water-insoluble fiber) of 1.54 oz./yd.².

A second layer (B) with a fabric weight of 0.80 oz./yd.² is made from a mixture containing 10 percent of the above cyanoethyl cellulose staple and 90 percent of the rayon staple by the above procedure with omission of the salt solution spray. The stock chest consistency of 0.06 percent is reduced to 0.02 percent just prior to entering the headbox.

An assembly of layer (A) covered with layer (B) is continuously fed to a belt of a 24 × 24 mesh/inch screen woven of 20-mil diameter wire moving at 6 feet per minute.

The assembly (on the screen) is hydraulically entangled by streams of a 20 percent water solution of Na₂SO₄ at 50° C. from a single row of 5-mil diameter orifices spaced about 25 mils (0.63 mm.) center-to-center in a 12-mil (.30 mm.) thick plate mounted on a manifold. The orifices are spaced about 1 inch from the fibers and a pressure of 300 p.s.i. is maintained on the salt solution in the manifold. Both ends of the horizontal manifold are oscillated by eccentrics on a 0.5-inch diameter eccentricity at a rate of 9 to 10 revolutions/second. At the pressure used, the 27-inch long row of orifices delivers 165 pounds of salt solution per minute through the 1080 orifices onto the 27-inch wide layer of fibers. The total weight of the double layer of fibers passing under the fluid streams in 1 minute is 0.220 pounds. The Y value is thus 11 × 10⁶ feet/minute.

If the salt solution is replaced with water alone, the papers are washed away and/or driven partially through the screen.

The unitary nonwoven fabric produced is pressed between blotting papers at 1450 p.s.i. Portions of the pressed products (about 50—60 percent solids) of about 10 × 26 inch are dried and softened by tumbling in a home laundry dryer at about 45° C. with 4 baseballs of 9.7 cm. in diameter.

Drying without softening yields a web that is stiff, boardlike, has poor drape and no aesthetic appeal.

The dry, softened product is a unitary nonwoven fabric with the 2 layers intimately connected. It has the appearance and hand of a soft woven fabric. FIG. 1 is a photograph at 10X magnification of the face of the fabric that faced the liquid streams and FIG. 2 shows the fabric face which was next to the screen.

The softened, nonwoven fabric has a fabric weight (as is) of 2.6 oz./yd.², contains 12.5 percent of Na₂SO₄, 41.1 percent cyanoethyl cellulose and 46.4 percent of rayon and has a density of 0.138 g./cm.³. Properties corrected to a salt-free basis of the product are given below.

Property	Non-softened		Softened	
	MD	CD	MD	CD
Tensile, dry				
(lb./in./oz./yd. ²).....	1.98	0.65	1.18	0.76
lb./in.	4.43	1.37	2.57	1.64
Elongation, dry (%).....	5	38	28	27
Tensile, wet				
(lb./in./oz./yd. ²).....	0.0168	0.0125	0.0136	0.0106
lb./in.	0.039	0.028	0.030	0.027
Elongation, wet (%).....	31	36	26	26
S.L. Absorbency.....			4.1	
Bending length (cm.).....	3.8	2.8	2.0	1.4
Water absorbency at				
25° C. (g./g.).....	7.8		7.8	
Urine absorbency at				
25° C. (g./g.).....	7.2		7.4	
Wickability (monutes).....	3		3.5	
Dispersibility, (%).....	48		54	
Flushability, 1st flush.....	50		100	
2nd flush.....	100			
Coherence value—				
dry/wet.....			0.46/0.08	
f _{1(wet)} (cyanoethyl fibers).....			0.48	

For use as a diaper the nonwoven fabric is folded to give a double thickness with the rayon-rich faces on the outside. The diaper is pinned about the baby in the usual manner. The diaper will absorb the total urine output of an average 6-months old baby for a period of 4 hours (i.e., 75 grams of urine). Even under this extreme condition the diaper retains its integrity, does not tear at the pins, leaves no loose fibers on the skin and is nonirritating The soiled diaper is dropped into a toilet and flushed away. This product is completely biodegradable so that no problems with sewage disposal are caused.

The nonwoven fabric may be used as a water-flushable sanitary garment.

EXAMPLE II

This example shows the effects of variables in the hydraulic entangling step.

Two single layers of fibers are made from cyanoethyl cellulose fibers and viscose rayon staple of Example I by the method of Example I. One layer contains 70 percent of the cyanoethyl cellulose fibers and has a nominal fabric weight of 1.18 oz./yd.². The other layer contains 10 percent of the cyanoethyl cellulose fibers and has a nominal fabric weight of 0.77.

A series of nonwoven fabrics are made by hydraulically entangling a top layer of the aryon-rich fibers to a layer of the cyanoethyl cellulose-rich fibers under various conditions. Each of the products has an average composition of 46.5 percent cyanoethyl cellulose fibers.

Items (a) to (h) are prepared with an orifice assembly having 540 orifices of 5-mil diameter evenly spaced in a single 27-

inch row. Items (i) to (k) are prepared with an orifice assembly having 1080 of the orifices of 5-mil diameter in a single 27-inch row. The same patterning screen and general conditions of Example I are used for both series, but pressures from 200 to 500 p.s.i. are used to give different total flow rates (*W*) and the rate of traverse (*V_s*) is varied. The entangling conditions and properties of the products are given in Table I. Tensile properties have been corrected by the average value of 17.2 percent Na₂SO₄ content to a salt-free fiber basis. The products have S.L. absorbencies within the range of about 3.1 to 3.4.

Items (a) to (f), (i), and (j) are products of the invention with dry strip-tensile strengths sufficient for use demands (such as pinning), wet strip-tensile strengths low enough to give ready flushability but high enough in body fluids to be useful, a water and urine absorbency of 7 grams/gram at 25° C., bending lengths of 1.1 to 1.8 cm. and a soft handle. These products have densities of from 0.113 to 0.159 g./cm.³ based on thicknesses measured with an Ames gauge.

Items (g), (h), and (k) show the effects of excessive hydraulic entangling on wet tensile properties and flushability. The effect of the liquid flow rate used at the same or similar duration of treatment is shown for items (d) vs. (g), and (c) vs. (h).

Items (a)—(f) and (i) are the most highly water-dispersible fabrics and correspond to fabric definition III in the summary of this invention. Item (j) falls within the intermediate dispersible definition II and items (h) and (k) are not water-dispersible. Item (g) falls within the water-dispersible fabrics of class

EXAMPLE III

The fibers of Example I are used to make a composite sheet having two layers of fibers as described below, using a double-layer headbox and the method of Example I.

Portions of the layer are dried with softening to give item (a).

Fiber layer	CNEC, percent	Rayon, percent	Fabric weight, oz./yd. ²
Bottom.....	10	90	0.9
Top.....	100	0	1.3
Average.....	64	36	2.2

Portions of the sheet are treated with fluid streams of 20 percent aqueous Na₂SO₄ at various pressures and speeds by the method of Example I but with an orifice assembly having 280 orifices of 5-mil diameter evenly spaced in a single row 14 inches long. The aperture patterning member is a 20 × 20 mesh screen of 0.025-inch diameter wire. The rayon-rich face

TABLE I

Item	Hydraulic entangling conditions			Coherence value		Tensile, dry		Tensile wet, average ¹		Elongation dry, percent CD/MD	Flushability, percent passed by 2d flush	Dispersibility, percent
	W, lb./min.	V, ft./min.	Y, ft./min. ×10 ³	C _b , dry	C _b , wet	Lb./in./oz./yd. ² CD	Lb./in. MD/CD	Lb./in./oz./yd. ²	Lb./in.			
a.....	79	5	7.6	0.46	0.06	0.85	0.68/1.65	0.014	0.027	32/16	100	50
b.....	79	4	9.3	0.39	0.06	0.50	1.5/0.98	0.014	0.027	40/21	100	48
c.....	79	3	12.5	0.20	0.07	0.18	1.4/0.35	0.0165	0.032	30/26	100	46
d.....	101	6	10.2		0.12	0.40	1.5/0.86	0.014	0.030	34/29	100	48
e.....	101	5	12.5	0.27	0.11	0.72	1.3/1.4	0.0155	0.033	28/23		40
f.....	101	4	15.7			0.79	2.1/1.6	0.021	0.041	46/33	100	63
g.....	145	6	20.7			0.67	2.3/1.4	0.057	0.12	44/29	50	24
h.....	145	3	42.0	0.30	0.49	0.67	3.3/1.3	0.105	0.21	37/32	55	8
i.....	137	5	11.1	0.24	0.05	0.50	1.1/1.0	0.018	0.039	33/27	100	50
j.....	166	6	13.6	0.44	0.10	0.55	2.0/1.2	0.042	0.090	36/28	90	32
k.....	195	4	28	0.55	0.44	0.75	2.6/1.5	0.127	0.27	40/37	40	11

¹ MD only.
² Estimated.

of the sheet faces the fluid streams. Pressures range from 50 to 500 p.s.i. The rate of traverse (V_s) is 6 feet per minute for item (c) and 2 feet per minute for item (d). The total flow rate (W) of the fluid streams and the calculated Y values are given in Table II. Properties of the products have an absorbency of 9.2 grams of water per gram of material at 25° C. and S.L. absorbencies of about 4.1.

TABLE II

Item	Entangling conditions		Coherence value, C_h , dry/wet	Tensile, dry		Elongation dry, percent MD/CD	Tensile wet, average		Dispersibility, percent
	W, lb./min.	Y, ft./min. $\times 10^6$		Lb./in./oz./yd. ² , CD	Lb./in./MD/CD		Lb./in.	Lb./in./oz./yd. ²	
a.-----	0	0	-----	1.02	5.3/2.7	2/1	<0.0002	<0.00008	62
b.-----	0	0	-----	0.79	3.2/2.1	1/1	<0.0002	<0.00008	-----
c.-----	53	9.1	0.65/0.08	1.11	3.4/2.6	28/28	0.025	0.010	67
d.-----	76	55	0.56/0.61	0.93	4.8/2.3	30/43	0.23	0.094	8

Both untreated products (items *a* and *b*) have dry strengths sufficient for use, but their usefulness is drastically limited by the low elongations at break. Urine wet strengths are too low for them to be useful as a diaper or sanitary garment. Both products have the appearance and hand of a paper. They lose 14 percent of their dry weight in the loose-fiber test.

Item (*c*) represents a preferred product of this invention. It has the appearance, handle and elongation of a fabric. It does not lose fibers in the loose-fiber test.

Item (*d*) has the appearance and handle of a fabric but the fibers have been so firmly entangled that it is not dispersible.

The importance of using essentially columnar, high impact streams of liquid is shown by comparison experiments with spray nozzles. A 63-mil diameter solid cone spray nozzle ("Sprayco No. 2112," made by Spray Engineering Co. of Burlington, Mass.) delivers 1.25 gallons per minute of water at 50 p.s.i. pressure. It was found that the spray entrains large amounts of air, thereby generating a high degree of air turbulence at the surface of the layer of fibers which leads to nonuniformities in the treated product. In order to protect the fibers from this turbulence, a fine screen was interposed on the surface of the sheet exposed to the streams.

On a hand sheet mold (serving as a suction box) is placed in sequence an apertured patterning plate, a sheet of the above two-layer composite with the rayon-rich face up, and a 400-mesh screen on the top. The assembly is sprayed with a 20 percent water solution of Na_2SO_4 at 50° C. from the 63-mil diameter solid cone spray nozzle located 4 inches above the top screen. The aperture plate has 63-mil diameter holes arranged on 94-mil staggered centers giving 132 holes/inch² and 41 percent open area. The spray is moved in one direction at a rate of about 4 feet per minute, which sprays about half the width of the sheet. The spraying is repeated until the entire sheet has received 8 exposures to the spray in one direction and 8 at 90° to that. Samples are prepared at 50, 75 and 100 p.s.i. and dried. In each case there is no entangling of fibers in the two layers. The rayon fibers on the top face of the product have been washed into clumps in each case. The underlying layer of fibers appears embossed at the 2 lower pressures and patterned at the higher. The rayon fibers on the top face are readily removed by the loose fiber test. All samples have dry elongations of less than 7 percent. Other properties are not measured since the products are so nonuniform.

Similar results are obtained when the composite sheet is treated with the rayon-rich layer of fibers on the bottom in contact with the patterning plate.

The inability of the above spraying procedure to entangle the fibers is even more strikingly demonstrated when the above double-layer composite sheet is replaced with 2 separately-prepared layers of fibers as in Example I. At pressures of 50, 100 and 150 p.s.i. the products are readily separated by hand into the two original layers.

Y values of less than 0.01×10^6 are calculated for the above spray treatments, when the liquid velocity is calculated from the liquid flow rate (W) and the area of the spray as it strikes the paper.

EXAMPLE IV

Preparation of Hydroxyethyl Cellulose (HEC) Fibers

This example illustrates the use of highly water-absorbent fibers of different types of cellulose ethers in preparing the products.

Conventional wood pulp sheets are steeped in 18 percent caustic soda solution at 27.5° C. for 45 minutes. The sheets of alkali cellulose are pressed to a 3/1 press weight ratio, shredded, and stored at 0° C.

One pound of the alkali cellulose is held at 26° C. for 27 hours, is added to a 5-liter baratte and the air is evacuated. Fifty-three grams of ethylene oxide are added over a 90-minute period as it is revolved on its side in a 25° C. water bath. The baratte is flushed with nitrogen and the hydroxyethyl cellulose produced is removed and dissolved in dilute aqueous NaOH at 0° to 10° C. to give a solution containing 6 percent of the cellulose ether and 7 percent caustic. The solution is frozen at -30° C., heated to room temperature and filtered. The filtered solution is extruded through a spinneret containing 504 orifices of 3-mil diameter into a coagulating bath (12 percent H_2SO_4 and 18 percent Na_2SO_4) and the yarn is neutralized. The dry yarn has a dpf of 2.6, a dry tenacity of 0.98 g.p.d., a dry elongation of 9.8 percent (all reduced to a salt-free basis), an absorbency for water and for urine at 25° C. of 12 and 11 g./g., respectively, and a degree of hydroxyethyl substitution of 0.61.

Preparation of Sodium Carboxyethyl Cellulose (CbEC) Fibers

Cyanoethyl cellulose fibers are prepared as for Example I except that the modified viscose solution is aged for 3 days at 25° C., and is then spun and neutralized as previously described. The fibers are washed with 70° C. distilled water, deswollen with acetone and dried at 100° C. The fiber has a degree of substitution of carboxyethyl groups of 0.13 and a degree of substitution of cyanoethyl groups of 0.04. It has an absorbency for distilled water and for urine at 25° C. of 34 and 8 g./g., respectively.

Preparation of Cyanoethyl Cellulose Fibers by Another Route

A solution of cyanoethyl cellulose (CNEC) is prepared according to British Patent No. 633,807 by adding the acrylonitrile to conventional viscose. The solution is spun to fibers and neutralized as described previously. The fibers have the following typical properties calculated on a water-insoluble fiber weight:

Denier-----	2
Water absorbency, g./g. (25° C.)-----	19.1
Urine absorbency, g./g. (25° C.)-----	14.0
Percent nitrogen-----	3.4
Percent carboxyl-----	0.4
Degree of cyanoethyl substitution-----	0.45
Degree of carboxyethyl substitution-----	0.015

Each of the above cellulose ether fibers is cut to 0.5-inch staple length and handsheets are prepared from aqueous slurries of the fibers in an 8 × 8-inch sheet mold. The handsheet and the sheet mold screen are removed from the sheet mold box, the fibers are deswelled by wetting with 18 percent aque-

ous sodium sulfate for about 1 minute, and the sheet is then dried. The following sheets are prepared in this manner, the weights and percentages being based on the water-insoluble fiber content:

Sheet a, 2.0 oz./yd.², 39 percent HEC fibers and 61 percent of 0.25-inch viscose rayon fibers;

Sheet b, 1.43 oz./yd.², 100 percent HEC fibers;

Sheet c, 1.43 oz./yd.², 100 percent CbEC fibers; and

Sheet d, 1.43 oz./yd.², 100 percent CNEC fibers.

Sheet (a) is hydraulically entangled with essentially columnar streams of 20 percent aqueous sodium sulfate solution as in Example I. Sheet (b) is plied with a layer B of Example I, containing 10 percent cyanoethyl cellulose fibers and 90 percent rayon fibers, and the assembly is hydraulically entangled, as above, to form a composite (b-B). Composites (c-B) and (d-B) are similarly prepared. The treatments were varied in the range of Y values from 6.6×10^6 to 20×10^6 to give different structural properties.

The products are dried and softened as in Example I. All have dry-strip-tensile strengths of at least 1.8 lbs./inch and dry elongations of at least 38 percent. Other properties are given in Table III.

EXAMPLE V

Cyanoethyl cellulose (CNEC) fibers of 0.5 inch length containing 43.9 percent of Na_2SO_4 and analyzing 3.5 percent N (salt-free basis) and 0.5 percent COOH (salt-free basis) with a D.S. (cyanoethyl) of 0.465 and an absorbency in distilled water of 19 grams/gram and 0.25 inch length rayon staple are used as fibers.

Handsheets of different compositions and a fabric weight (salt-free) of 1.5 oz./yd.² are made from aqueous slurries of the above fibers. The sheets are deswollen in 20 percent Na_2SO_4 solution, pressed between couch rolls and dried between nylon fabric and blotting paper on a sheet dryer at 100° C.

All sheets are hydraulically entangled by passing the sheet resting on a 24×24 mesh screen at 3 feet a minute under oscillating streams of 20 percent aqueous Na_2SO_4 . Water is used as the entangling fluid for the 100 percent rayon sheets. The streams are produced by a plate containing a single row (14" long) of 0.007 inch diameter venturi shape, orifices spaced 20/inch which is mounted on a manifold. The orifice plate is located about 1 inch above the handsheet.

The entangled sheets are blotted and then dried and softened by tumbling in a home laundry dryer at 60° C. with three baseballs of 9.7 cm. in diameter for 25 minutes.

The physical properties obtained at various Y values (by altering the pressure of the entangling fluid) are given in Table IV. All samples have dry elongations of from 20 to 56 percent.

Items a, b, and c represent unsuccessful attempts to make useful flushable products of 100 percent rayon. Water pressures of 100, 150, and 200 p.s.i. are used. The combination of a dry tensile strength of at least 0.3 lb./in. and a wet tensile strength of less than 0.1 lb./in. is not obtained. At higher Y values needed to increase the dry strength the product is not dispersible due to the higher wet strength obtained.

Items d, f, h, j, k, l, m and n represent products of this invention.

Items e, g, and i have been excessively entangled to give wet tensiles of 0.1 lb./in. or higher and dispersibilities of 15 and less so that they are not dispersible.

It is seen that the hydraulic entangling conditions are extremely critical, particularly at the lowest cyanoethyl cellulose

TABLE III

Item	Average composition	Tensile, wet, lb./in./oz./yd. ²	Lb./in.	Elongation dry, percent	Absorbency		Dispersibility percent
					Water	Urine	
a-----	39% HEC-----	0.006	0.012	38	7.2	8	10
b-B-----	64% HEC-----	0.011	0.048	46	8.4	8.5	74
c-B-----	64% CbEC-----			55	34.3	6.0	40
d-B-----	64% CNEC-----	0.015	0.063	52	11.2	8.2	56

levels. The results in the table and results at other (unreported) Y values can be expressed by the following equation:

$$Y_{\text{maximum}} \leq 11 + 1.4 (\% \text{ cellulose ether})^{1/2}$$

Thus the maximum Y values for 10 and 100 percent of cellulose ethers is about 15 and 25 respectively. This relationship applies only to the preferred blends of cyanoethyl cellulose with rayon. In the absence of low-swelling fibers, much higher Y values may be used as shown in the following Example VI.

TABLE IV

Item	$Y \times 10^{-6}$	Percent CNEC	Tensile strength, lbs./in.		Dispersibility, percent	B.W., oz./yd. ²
			Dry	Wet		
a-----	7.0	0	0.12	0.088	32	0.92
b-----	10.8	0	0.16	0.137	16	0.97
c-----	14.3	0			5	1.07
d-----	12.4	10	0.62	0.061	48	1.60
e-----	19.1	10	1.43	0.13	6	1.60
f-----	12.4	15	0.58	0.065	39	1.49
g-----	19.1	15	1.01	0.158	8	1.14
h-----	12.4	20	0.71	0.090	54	1.60
i-----	19.1	20	1.29	0.113	7	1.38
j-----	12.4	50	1.65	0.052	58	2.18
k-----	19.1	50	1.0	0.079	31	2.18
l-----	12.4	75	2.28	0.045	62	2.64
m-----	19.1	75	0.95	0.056	29	2.64
n-----	12.4	100	1.96	0.062	56	2.74

EXAMPLE VI

Cyanoethyl cellulose fibers of about 2 denier per filament and 0.5 inch length, with a D.S. (cyanoethyl) of 0.45 are used alone, without any rayon staple. Handsheets of 1.5 oz./yd.² are made by the method of Example V and dried at 65° C.

The sheets are hydraulically entangled as in Example V, except the screen is moved at 5 feet per minute. One pass is made at a hydraulic pressure of 100 p.s.i., followed by 5 passes at 500 p.s.i. The aggregate Y value of the treatments is calculated to be 103.5×10^6 ft. per min. The dried product has the strength and appearance of a woven fabric.

Although the dry strength of this fabric is high, the absence of any low-swelling fibers permits complete dissipation in turbulent water. The dispersibility of the fabric is 55.8 percent and the dry elongation is 27 percent.

EXAMPLE VII

Cyanoethyl cellulose fiber similar to that of Example I is dried and cut to 1.5 inch length. It is made into a batt by an air deposition process, using a Rando-Webber machine (made by Curlator Corporation of East Rochester, N.Y.). The batt has a fabric weight of 1.57 oz./yd.² including salts and soluble organic matter, or a fabric weight of 1.0 oz./yd.² on water-insoluble fiber basis.

Two layers of the above batt are plied together, completely wetted with a 20 percent solution of Na_2SO_4 and then hydraulically entangled using the apparatus and conditions of Example I. The sample is dried and softened. The soft, fabriclike product has the following properties:

Tenacity, dry, CD—1.8 lb./in./oz./yd.² 5.05 lb./in.
Elongation, dry, CD/MD—43%/33%.
Tensile, wet, average—0.006 lb./in./oz./yd.² 0.0208 lb./in.
Absorbency, distilled water—37° C. 11.6 g./g.
Dispersibility—73%
Wickability—3 hours, 20 minutes.
Water-soluble materials and salt—40%.

EXAMPLE VIII

Initial fibrous layers are made, using the method and fibers similar to those of Example I. Layer (A) contains 70 percent cyanoethyl cellulose fibers and 30 percent rayon, and has a fabric weight (water-insoluble) of 1.29 oz./yd.². Layer (B) contains 10 percent of the cyanoethyl cellulose fibers and 90 percent of rayon, and has a fabric weight (water-insoluble) of 0.79 oz./yd.². Layer (A) is supported on an apertured patterning plate and covered with layer (B). The assembly is passed at 3 feet/minute under essentially columnar streams of concentrated aqueous sodium sulfate solution from 560 orifices of 5-mil diameter evenly spaced in a single 14-inch row. A flow rate of 56.5 pounds/minute is used, which gives a *Y* value of 11×10^6 ft./min.

The apertured patterning plate is a commercial plastic netting consisting of a grid of 40-mil diameter polyethylene rods. The lower face of the grid contains parallel rods spaced 12 per inch. The upper face of the grid contains a similar array of rods arranged 45° to the axis of the lower rods. The rods are fused together where they contact each other. The open area of the plate is 27.1 percent of the total area.

The soft, fabriclike product is illustrated in FIGS. 3 and 4. It has the following properties:

Tensile strength, dry, MD (water-insoluble fiber basis)—0.63 lb./in./oz./yd.² 1.1 lb./in.
Dry elongation, MD—36%.
Tensile strength, wet, average (water-insoluble fiber basis)—0.029 lb./in./oz./yd.² 0.052 lb./in.
Dispersibility—46%.
Absorption, water at 25° C.—6.6 grams/gram.

Useful products having similar properties with S.L. absorbencies of about 3.6—4 are also made from the above initial fibrous layers in similar manner at *Y* values between 8.6×10^6 and 17×10^6 , using the following apertured patterning supports:

1. A grid of 0.038-inch diameter rods spaced 12 inch and having an open area of 54.4 percent.
2. A grid of 0.020-inch diameter rods spaced 25 per inch and having an open area of 50.0 percent.
3. Twill screen of 0.023-inch diameter wire, 24 × 24 mesh in a 2 × 2 twill weave having 20 percent open area.
4. Twill screen of 0.017-inch diameter wire, 30 × 30 mesh in a 2 × 2 twill weave having 23.9 percent open area.

EXAMPLE IX

Handsheets with a fabric weight of 0.5 oz./yd.² (17 g./m.²) are made from an aqueous dispersion containing 10 parts of cotton staple fibers of 0.25 inch length (0.63 cm.) and 1 part of cyanoethyl cellulose fibers of 0.5 inch length (1.27 cm.) (having a D.S. of 0.47) as a binder.

The above handsheet is placed on top of a batt of 100 percent rayon staple fibers (1.56" length and 1.5 d.p.f.) fabric weight of 1 oz./yd.² (33.9 g./m.²) and the layered structure, supported on a screen (20 × 20 mesh) is hydraulically entangled using a 20 percent aqueous solution of Na₂SO₄ at 60° C. as the jetting fluid with two treatments at 400 p.s.i. The salt is washed out with water and the fabric dried.

The doubled-layered integral fabric is saturated with an aqueous solution containing 20 percent sodium chloroacetate and 3 percent sodium hydroxide, and pressed to a wet press ratio of 2.8. The wet fabrics are placed on a hot metal plate covered with a fabric coated with polyfluoroethylene at 125° C. for 5 minutes. The baked fabrics are acidified by immersing in an aqueous bath containing 5 percent H₂SO₄, then neutralized in a bath containing 3 percent disodium phosphate and 17 percent sodium sulfate adjusted to a pH of 8.5. The fabric is dried at about 80° C.

The fabric has a dispersibility of 40 percent so that it is disposable in home sewage systems. When the fabric is soaked in synthetic urine and blotted, the cotton-covered fabric side is distinctly less swollen and feels dryer than the modified

rayon side. The cotton has been scarcely affected by the treatment.

EXAMPLE X

A nonwoven fabric of 1.2 oz./yd.² (41 g./m.²) weight is made by passing a random web of 0.75 in. (19 mm.) rayon fiber on a 24 mesh screen at 5 yards (4.6 m.) per minute under 3 rows of water streams coming from orifices having an upper cylindrical section of 0.005 inch (0.13 mm.) diameter with a lower frustoconical section as an exit and spaced 40 per inch (per 2.54 cm.) at pressures of 400, 500, and 600 p.s.i. (23, 35, and 42 kg./cm.²) respectively. The orifices are about 12 mm. above the web. The rayon fabric has a *C_h*, dry and *C_h*, wet of 0.35 and 0.71 respectively.

The nonwoven rayon fabric is passed through a tank containing a solution of 50 percent urea and 18 percent H₃PO₄ at room temperature. The fabric is squeezed by a pair of rubber-covered rolls to a pickup of about 2.0 g./g. The wet fabric is continuously passed through a commercial fabric over (Benz) where it is exposed to opposed jets of air at 164° C. for a period of about 91 seconds and then wound up at about 6.3 feet (1.9 meters) per minute.

The baked fabric (cellulose acid phosphate) is then carried on a screen underneath sprays of tap water for washing and excess water is removed by squeeze rolls. The wet fabric is then carried under sprays of a solution containing 17 percent Na₂SO₄, 3 percent Na₂HPO₄ and 1 percent sodium hexametaphosphate adjusted to a pH of 6.0 and excess solution is removed by squeeze rolls.

The wet, modified fabric (sodium cellulose phosphate) is then passed through an oven where it is exposed to air at 34—40° C. issuing from pairs of opposed slots transverse to the direction of fabric so that the fabric flutters between the 2 opposed jets of air. The dry fabric is then softened by passing between a pair of meshing grooved rolls with the grooves running in the direction of travel of the fabric followed by 3 pairs of meshing groove rolls having grooves transverse to the direction of travel of the fabric. All of the rolls are set at 3.6 lb./inch of width (0.645 kg./cm.) pressure. The softening treatment is repeated.

Diaper size portions of the product are flushable. The properties are given in Table V. This is a preferred specie having an average tensile in synthetic urine of at least 0.2 lb./in.

EXAMPLE XI

This example illustrates the use of cellulose sodium hemi succinate in this invention.

The nonwoven rayon fabric of Example X is passed through a tank containing a solution at 60° C. of 30 percent succinamic acid and 0.60 percent sulfamic acid, and squeezed by rolls to a pickup of about 2.0—2.1. The wet fabric is continuously passed through a circulating air oven (205° C.) at a speed to give a residence time of about 95 seconds.

The fabric of cellulose hemi succinate is washed by passing through a tank of soft water, squeezed and then neutralized and bleached by passing through a tank containing a solution at room temperature of 3 percent Na₂HPO₄, 17 percent Na₂SO₄ and about 0.5 percent KHSO₅ adjusted to a pH of about 8.5—9.0. The fabric of cellulose sodium hemi succinate is squeezed and dried in circulating air at room temperature.

Diapers made of the product are tested on babies and found to have excellent integrity in use and the used diapers are completely flushable. Properties of the product are given in Table V.

EXAMPLE XII

This example illustrates the use of fibers of sodium cellulose sulfate in the invention.

A nonwoven fabric of 1.3 oz./yd.² (44 g./m.²) weight is made from a random web of 0.75 inch (19 mm. crimped, rayon fibers using the apparatus and speed of Example X at pressures of 300, 500, 800 and 800 p.s.i. (21, 35, 56 and 56 kg./cm.² respectively) for the 4 rows of orifices.

The rayon fabric is placed in solution at 50° containing 300 g. urea, 100 g. 99 percent H_2SO_4 and 75 g. of water for 2 minutes. It is then pressed to a pickup of 3.2 and the wet fabric heated in an oven at about 169° C. for 3 minutes. The heated fabric is neutralized in a solution containing 17 percent Na_2SO_4 and 3 percent Na_2HPO_4 adjusted to a pH of 8.5, squeezed to a pickup of 1 g./g. and dried. The product is flushable. Properties are given in Table V.

EXAMPLE XIII

This example shows the use of sodium carboxymethyl cellulose fibers in this invention.

A nonwoven fabric of 0.8 oz./yd.² (27 g./m.²) weight is made from 1.56 inch (39 mm.) rayon fibers of 1.5 d.p.f. using the procedure and equipment of Example X except that 2 additional rows of orifices at 200 and 300 p.s.i. (14 and 21 kg./cm.²) pressure are used. The fabric has a C_h , dry and a C_h , wet of 0.74 and 0.96 respectively.

The fabric is chemically modified by passing it continuously through a bath containing 2.1 percent NaOH and 15.8 percent sodium chloracetate squeezing it to a pickup of 1.1 g./g. heating in an oven with air at 140° C. for 18 seconds, acidifying to the carboxymethyl cellulose with a bath of 5 percent H_2SO_4 , washing in water and forming the sodium salt in a bath of 2 percent Na_2HPO_4 and 8.5 percent Na_2SO_4 adjusted to a pH of 8.5. The fabric (A) is dried at 40° C. in a tumble dryer with baseballs of 9.7 cm. diameter.

Sanitary napkins are made wrapping a 19" × 7" (64 × 17.8 cm.) portion of the fabric around a core, overlapping the fabric on the bottom of the napkin and gluing the overlap with small spots of a water soluble adhesive. The core consists of wood fluff wrapped with crepe tissue paper which contains a coating of a poly (fluoro alkyl methacrylate) on the sides and bottom of the paper to serve as a fluid barrier. The napkins are used and then flushed. The vast majority (223 out of 230) of the napkins flush with 1 rush of water. The other 7 require a second flush to clear the bowl.

A second fabric (B) of 1.25 oz./yd.² (42 g./m.²) having a C_h , dry and C_h , wet of 0.66 and 0.78 respectively is made as above except at a speed of 7 yards (6.4 m.) per minute under jets at 300, 500, 600, 700 and 900 p.s.i. (21, 35, 42, 49 and 63 kg./cm.²). The fabric is chemically modified as above except that the reagent solution contains 2.22 percent NaOH and 18.4 percent sodium chloracetate and the final salt-forming solution contains 1 percent Na_2HPO_4 and 4.25 percent Na_2SO_4 adjusted to a pH of 8.5.

Diapers are assembled with a layer of the above fabric, a wood fluff, a crepe tissue wadding, wood fluff and the above fabric in order. A medium size diaper uses 11 × 14" (28 × 35 cm.) fabric and 5 × 12" (12.7 × 30 cm.) cores of wood fluff and crepe tissue. The 2 fabric layers are glued together around the edges of the core with about 3 mm. spots of a water-soluble adhesive spaced about 5 cm. apart.

The diapers are used on babies in wear tests and are found to have good integrity in use and all are successfully flushed in home toilets after use.

Properties of the fabrics are given in Table V.

EXAMPLE XIV

This example shows the preparation of a nonpatterned product of the invention.

A nonwoven fabric of 2.3 oz./yd.² (88 g./m.²) is made from the rayon fiber of Example X using the general method and a single row of orifices of that example with a 150 mesh per inch (per 2.54 cm.) screen (37 percent open area) used as the support to carry the web at 4 yards (3.7 m.) per minute under the oscillating row of orifices for one pass at each of the pressures of 200, 500, 1000, and 1200 p.s.i. (14, 35, 70 and 84 kg./cm.²) respectively. The nonforaminous fabric is smooth and resembles a felt with faint grooves.

A portion of the dry fabric is immersed in a solution of 50 percent urea and 18 percent H_3PO_4 for 2 minutes, drained and

blotted to a pickup of about 1.6—1.7. The wet fabric is heated in an air oven at 160° C. for 4 minutes. The fabric is then washed in an aqueous solution of 5 percent H_2SO_4 and 17 percent Na_2SO_4 , drained, blotted, and converted to the sodium salt of cellulose phosphate in an aqueous solution of 4 percent Na_2HPO_4 and 17 percent Na_2SO_4 adjusted to a pH of 8.4. The fabric is drained, blotted, and air dried.

A medium size diaper is made similar to that in Example XIII. The diaper breaks into 2 portions after dipping it 10 times in a toilet bowl and 85 percent of the diaper passes the hoods after 3 flushes. Properties of the product are given in Table V.

EXAMPLE XV

This example shows the use of a heterogeneous water-sensitive fiber in the invention.

A nonwoven fabric of 0.8 oz./yd.² (27 g./m.²) weight is made from a random web of 0.5" (12.6 mm.) rayon using the apparatus and method of Example X but with 4 rows of orifices at 300, 400, 500 and 500 p.s.i. (21, 28, 35 and 35 kg./cm.²).

The dry fabric is placed in a solution of 70 volumes of water and 30 volumes of N-dimethyl formamide for 1 minute and then squeezed to a pickup of 1 g./g. The wet fabric is then placed in a 0.25N solution of SO_3 in N-dimethyl formamide at room temperature for 1 minute and squeezed to a pickup of 1 g./g. The fabric is then treated in a 5 percent aqueous solution of H_2SO_4 for 1 minute, squeezed and then treated in an aqueous solution containing 17 percent Na_2SO_4 and 3 percent Na_2HPO_4 adjusted to a pH of 8.5 for 1 minute, squeezed and air dried to yield a fiber of cellulose (rayon) with an outer skin of the sodium salt of cellulose sulfate.

A 17 × 13 inch (44 × 33 cm.) portion of the fabric is soaked in synthetic urine, dipped 2 times in water and flushes 100 percent. Although the fabric is quite slippery when wet it has a S.L. absorbency of an inert fiber fabric which shows that the modification of the individual filaments are confined to or near the surface or skin. The fabric has the properties as given in Table V. The average D.S. of the rayon core and the modified skin is 0.008.

EXAMPLE XVI

This example shows the use of a heterogeneous, water-sensitive fiber.

A nonwoven fabric of 1.0 oz./yd.² (34 g./m.²) weight is made from a random web of 0.75 inch (19 mm.) rayon using the apparatus and procedure of Example X with pressures of 200, 500, 700, and 800 p.s.i. (14, 35, 49, 56 kg./cm.²) respectively for the 4 rows of orifices. The product has a C_h , dry and a C_h , wet of 0.74 and 0.78 respectively.

The fabric is passed through a tank containing a solution of 50 percent urea and 18 percent H_3PO_4 and squeezed to a pickup of 2.03 g./g. The wet fabric is then passed through a tenter frame oven where it is heated by moving air at 163° C. for a period of 3 minutes. The next day the fabric is washed with water, treated with a solution containing 3 percent Na_2SO_4 and 12 percent Na_2HPO_4 adjusted to a pH of 8.5 to form the sodium salt of cellulose phosphate, dried with 67° C. air and softened as in Example X.

Diapers made similarly to those of Example XIII and used on babies show satisfactory integrity and are 100 percent flushable after 1 dip.

The fabric has the properties given in Table V.

EXAMPLE XVII

The example shows the conversion of a cotton fabric to sodium carboxy methyl cellulose.

A nonwoven fabric of about 0.8 oz./yd.² (27 g./m.²) weight is made from a random web of carded middling cotton of 1.03" (26 mm.) length using a single row of orifices of Example X on 24 × 24 mesh screen and 1 pass at 5 yards (4.6 m.) per minute at each of the pressures of Example XVI with the orifices oscillating.

The cotton fabric is dewaxed by boiling in a detergent solution for 30 minutes and rinsing. A portion of the fabric is soaked in a solution of 27.3 percent sodium chloracetate and 9.1 percent NaOH for 2 minutes, drained and blotted to a pickup of about 3.0. The wet fabric is heated in an air oven at 125° C. for 5 minutes, acidified in a solution of 5 percent H₂SO₄ and 17 percent Na₂SO₄, neutralized in a solution of 4 percent Na₄HPO₄ and 17 percent Na₂SO₄ (at pH of 8.4), blotted and air dried.

A medium size diaper, soaked in synthetic urine breaks up after 1 dip in water and is 95 percent flushable on 1 flush.

The fabric has the properties given in Table V.

EXAMPLE XVIII

This example shows the conversion of a cotton fabric to a sodium cellulose sulfate.

A nonwoven of about 1 oz./yd.² (34 g./m.²) and having a C_h, dry/wet of 0.41/0.63 is made using the cotton fiber, apparatus and procedure of Example XVII except at twice the speed.

The cotton fabric is dewaxed by boiling in a solution of detergent and Na₃PO₄ for 30 minutes and rinsing. A portion of the dewaxed fabric is secured (against shrinkage) by its edges to a metal screen and mercerized in 25 percent NaOH at 18° C. for 3 minutes. The fabric is washed in water to a neutral pH and air dried.

The mercerized fabric is immersed for 2 minutes in a solution containing 40 percent urea and 20 percent sulfamic acid, removed, squeezed gently to obtain a pickup of 5.9, placed on an inert plastic screen and heated in a circulating air oven at 150° C. for 5 minutes. The heated fabric is neutralized to the sodium salt in a solution containing 3 percent Na₂HPO₄ and 17 percent Na₂SO₄ (pH 8.5), drained, blotted and air dried.

Although the dispersibility is only 17 percent, the sample is essentially completely dispersed in 45 seconds of the test indicating flushability of a diaper size portion. Properties of the product are given in Table V.

EXAMPLE XIX

The example illustrates the greater internal bond strength of the fabrics of this invention over the prior art structures.

The internal bond test, originally designed to measure the internal bond strength of papers, is useful as a measure of the vertical entangling component through the thickness of the fabric.

In essence, the test consists of measuring the force necessary to rupture the test specimen when the plane surfaces are mounted between and held by two pieces of adhesive tape.

As expected, a laminar type structure is readily parted (low internal bond) whereas a structure held together by a vertical component requires higher forces since the vertical component requires higher forces since the vertical fibers must be broken.

The Internal Bond Strength of a nonwoven is determined on an Internal Bond Tester (model B - made by Sisalkraft-Scott Co. of Providence, R.I.) by measuring the force required to

shear a sample in its major plane with a weighted pendulum. The sample is fastened to a lower fixed steel plate and to an upper aluminum right angle striking bar (which contacts the pendulum) by means of double-faced sensitive tape (Scotch Brand type 400 made by 3M Co. of St. Paul, Minn.).

The faces of the steel plate, striking bar, nonwoven and tape are 1 inch × 1 inch (2.54 × 2.54 cm.) The tester includes a jig for mounting five samples at a time at a standard pressure. Five samples are tested in both machine and cross direction and the average value reported. The nonwoven should be in a salt-free state as obtained by extraction for the Coherence Value (C_h) determination. Pressures of 200 p.s.i. (14 kg./cm.²) are used, although lower pressures may be required with very thin and porous samples to prevent forcing the upper tape into contact with the lower tape. Meaningful results are not obtained with coarsely apertured products [i.e. 12 or less apertures or mesh per inch (per 2.54 cm.) with a fabric weight of less than about 1 oz./yd.² (34 g./m.²)] due to the adhesion of tape to tape through the thickness of the fabric.

Fabrics from a number of the previous examples are tested as well as papers and a resin-bonded commercially available nonwoven.

The preferred products of this invention have internal bond strengths of at least 0.80 foot-pounds (ft.-lb.) [1.1 kilogram-centimeter (kg.-cm.)] while the more preferred products having the most dry integrity have internal bond strengths of at least 0.15 ft.-lbs. (2.1 kg./cm.).

Typical results are given below:

	Internal bond strength	
	Ft.-lbs.	(Kg.-cm.)
Fabric from Example:		
I, softened	0.15	2.1
II, item c	0.09	1.2
II, item j	0.16	2.2
X	0.12	1.7
XI	0.19	2.6
XII	0.17	2.3
XIII (A)	0.37	5.1
XIII (B)	0.27	3.7
XV	0.24	3.3
XVI	0.14	1.9
XVII	0.22	3.0

typical rayon nonwovens

used for example X—XVIII 0.25 to >0.5 3.4 to >6.9

commercial, resin-bonded,

rayon nonwovens (Handiwipe 0.05—0.07 0.7—1.0

- Binder in one-sixteenth inch bands

on one-fourth inch centers across

width for strength - made

by Colgate-Palmolive.

papers, used as starting 0.01 to 0.05 0.14—0.7

material for examples 1 and 2.

TABLE V

Example	D.S.	SL absorbency	Coherence value		Tensile strength, MD/CD, lb./in. (g./cm.)			Elongation dry MD/CD, percent	Fabric weight, oz./yd. ² (g./m. ²)	f, wet
			C _h , dry	C _h , wet	Dry	Wet	Wet, S.U.			
X	0.26-30	4.6	0.29	0.04	6.1/2.6 (1,100/460)	1.018/.011 (3.2/2.0)	0.77/0.38 (137/68)	12/72	2.3 (78)	0.72
XI	0.30	ca 4.0	0.42	0.04	4.2/3.0 (680/490)	.017/.11 (2.6/18)	.49/.40 (80/65)	27/44	1.5 (52)	0.61
XII	0.28	3.3	0.50	0.27	2.4/2.0 (427/360)	.061/.028 (11/4.8)	-----	32/36	1.5 (52)	-----
XIII(a)	-----	4.3	0.66	0.11	4.1/2.8 (730/500)	.0016/.0015 (.29/.28)	² 0.23 (41.)	28/77	1.2 (41)	-----
XIII(b)	-----	8.5	0.77	0.10	6.7/3.0 (1,200/540)	1.016/.005 (2.9/0.9)	.25/.11 (36/20)	20/77	1.8 (62)	0.31
XIV	0.32	6.4	0.69	0.14	c6.8/9.1 c(1,200/1,600)	c.0010/.0015 (.18/.27)	.45/.39 (80/70)	50/30	3.2 (110)	-----
XV	-----	0.9	0.32	0.06	c2.4/1.0 c(430/180)	c.004/.004 (.17/.17)	² 0.14 (25.)	32/36	1.0 (34)	0.53
XVI	-----	1.7	0.58	0.06	c3.0/1.3 c(540/230)	.005/.003 (.90/.54)	² 0.28 (50.)	15/29	1.74 (59)	0.41
XVII	-----	6.4	0.74	0.09	c2.7/2.0 c(480/360)	c.014/.012 c(2.5/2.1)	-----	41/51	1.0 (34)	<1.0
XVIII	-----	3.7	>0.2	0.14	-----	0.0081/ (.141)	-----	-----	-----	-----

¹ Tap water. ² Average.

EXAMPLE XX

This example is a further illustration of the wide variety of substituents with which the cellulosic fibers may be modified to the water-sensitive form to obtain the product of the present invention. A rayon nonwoven fabric is made water-dispersible by modifying the fibers to sodium cellulose hemiphthalate.

The starting fabric is 100 percent rayon of three-fourths inch fibers having a basis weight of 1.4 oz./yd.². The fibers are hydraulically entangled. The fabric (10 in. × 10 in.) is immersed in a 23 percent aqueous phthalamic acid solution containing 0.46 percent MgCl₂·6H₂O for 2 minutes. It is then removed and couched between polyethylene sheets to a pickup ratio of 4.53/1. The fabric and acid are reacted on a sheet dryer at 150° C. for 7 minutes. The modified fabric is then neutralized in a bath of 3 percent Na₂HPO₄/17 percent Na₂SO₄ (pH 8.5–9). The neutralized fabric is removed, blotted, and then dried in a tumble dryer for 25 minutes. The process is repeated using a fresh reaction solution (pickup ratio 3.39/1).

The dried modified fabric is white, has good aesthetics, and is suitable for flushable diapers and sanitary napkins as the dispersibility is 50 percent with 100 percent dispersibility occurring after 15 sec. The SLWA is 19.7 g./g. and D.S. is 0.36.

EXAMPLE XXI

This example shows the use of fibers of a phosphate of polyvinyl alcohol in water-dispersible nonwovens. A nonwoven fabric of about 1.09 oz./yd.² weight is made from commercial, water-insoluble, crosslinked polyvinyl alcohol fibers of 1.5 dpf and 1.5 inch length that is carded and then cut to 0.75 inch length. The fibers are made into a random web by air laying and hydraulically entangled on a 24-mesh screen at 3 yards a minute under a single row of water streams of Example X at pressures of 100, 200 and 300 p.s.i. for each of the 3 treatments. The fabric has a S.L. absorbency of 0.17 and a *f_{wet}* of 1.47.

The polyvinyl alcohol nonwoven fabric is treated with a 50 percent aqueous solution of urea to leave a pickup of 2 g./g. and then baked at 160° C. for 6 minutes. This fabric is then soaked in an aqueous solution containing 45 percent urea and 16.2 percent H₃PO₄, the pickup adjusted to 3 g./g., and the fabric baked at 160° C. for 5 minutes. The product is wrapped in cheesecloth for subsequent handling to protect the fabric. The product is washed in a 5 percent aqueous solution of sulfuric acid containing 15 percent of Na₂SO₄ and then converted to the salt form in a bath (a) adjusted to a pH of 8.4 containing 17 percent Na₂SO₄ and 4 percent Na₂HPO₄. The product (after salt removal) contains 2.7 percent P.

Properties of the salt-free product are given in Table VI.

The product is water dispersible as judged from its breakup time (B.U.T.) of about 100 seconds. The starting fabric of polyvinyl alcohol fibers has a B.U.T. of greater than 120 seconds.

The B.U.T. is determined by dropping a 3 in. × 3-inch piece of fabric (folded in half twice) into 800 ml. of distilled water (room temperature) contained in a 1-liter beaker. The water is stirred by an egg-shaped (dimensions major axis 2.5 in. with a minor axis of about seven-eighths inch No. 4620—Teflon*-covered, egg-shaped of the Cole and Parmer Co. of Chicago, Ill.) magnetic stirring rod that is driven by a 9-inch diameter stirring base ("Jumbo Magnetic Stirrer," Model 14-511-75VI by Fisher Scientific Co. of King of Prussia, Pa.) to give a stirrer speed of 390 revolutions per minute which gives a vortex in the water of about 1½ inches. B.U.T. is the time from submergence until the fabric separates into 2 or more distinct pieces. Mere fraying of loose fibers from the edges is not considered as a separation.

EXAMPLE XXII

This example shows the preparation of a water-dispersible fabric by the hydrolysis of a nonwoven fabric of acrylic fibers.

A nonwoven fabric of about 1.1 oz./yd.² weight is made from 1.5 dpf, 0.75 in. long fibers of a copolymer of acrylonitrile/sodium styrene sulfonate (96/4 percent) by entangling a random web on a 24-mesh screen at 3 yards/minute under a single row of water streams of Example X at pressures of 100, 200, 200, and 600 p.s.i. for each of the treatments. The acrylic fabric has a S.L. absorbency of 0.03 and a B.U.T. greater than 120 seconds.

The entangled nonwoven fabric is hydrolyzed by refluxing (109.5° C.) in an aqueous solution of 10 percent HCl, 10 percent NaCl for 24.75 hours. To minimize mechanical damage, a double layer of the nonwoven is wrapped in an acrylic woven fabric, placed between 2 Teflon* screens that are held apart and attached to a rotating shaft in the hydrolysis bath. After refluxing, the fabric is cooled, converted to the salt form in the bath (a) of Example XXI and air dried. The samples are extracted with 60 percent aqueous solutions of acetone with a final acetone wash to remove the salt before testing.

Properties of the product are given in Table VI. The fabric has a B.U.T. of 30 seconds.

EXAMPLE XXIII

This example shows the preparation of a water-dispersible fabric from nylon-acrylic acid grafted fibers.

The starting fiber is a 2 dpf 6–6 nylon fiber that has been grafted with acrylic acid using the process of U.S. Pat. No. 2,999,056 to Tanner. The graft is in the form of the calcium salt that has been partially changed to the sodium salt. The staple is carded and cut to five-eighth inch, made into a random web and entangled with the apparatus, method and speed of Example XXI using one treatment at 100 p.s.i. and 3 treatments at 200 p.s.i. A similar fabric converted completely to the sodium salt form has a S.L. absorbency of 0.3 and a B.U.T. greater than 120 seconds.

The entangled nonwoven fabric is soaked in a 25 percent aqueous solution of acrylic acid for 30 minutes, padded to a pickup of 0.8 g./g. and exposed under a resonant transformer at 2 MeV and 1 milliamp for a 4 megarad dosage for further grafting. The washed and dried sample has a weight gain of about 16 percent. The fabric is wrapped in cheesecloth, boiled 0.5 hour in a solution of 3 percent NaOH/10 percent Na₂SO₄, excess liquid removed and reneutralized to pH 8.7 in a bath containing 1.5 percent Na₂SO₄, 6 percent Na₂HPO₄ and 3 percent sodium hexametaphosphate and dried.

Properties of the product are given in Table VI. The product has a B.U.T. of about 33 seconds.

EXAMPLE XXIV

This example shows the preparation of a water-dispersible fabric from casein fibers.

The starting fibers are commercial casein fibers of about 5.3 dpf. The 4 in. staple is carded and the card silver cut to 0.75 in. length. A random web of the cut fiber is entangled using the method, speed, and apparatus of Example XXI with one treatment at each of the following pressures: 100, 200, 200, 700 and 1000 p.s.i. The fabric has a S.L. absorbency of 0.3 and a B.U.T. greater than 120 seconds.

The entangled nonwoven fabric is immersed in a 50 percent aqueous solution of urea for 2 minutes, drained, the pickup adjusted to 1 g./g. and baked for 5 minutes at 160° C. The dry product is wrapped in cheesecloth and neutralized in a bath (a) of Example XXI. Properties of the dried product are given in Table VI. The product has a B.U.T. of 12 seconds.

A water-dispersible fabric (B.U.T. of 20 seconds) is also made by neutralizing the original nonwoven (without urea treatment) with bath (a) of Example XXI.

EXAMPLE XXV

This example shows the preparation of a water-dispersible fabric from cellulose acetate fibers.

The starting fibers are commercial cellulose acetate (D.S. ≈ 2.4) fibers of 1.5 dpf, 0.75 in. length. A random web of the

fibers is entangled using the method, apparatus, and speed of Example XXI for one treatment at each of the following pressures: 100, 200, 200 and 500 p.s.i.

The cellulose acetate nonwoven is treated in an aqueous solution at 45° C. containing 0.3 percent sodium hydroxide and 1 percent sodium acetate, washed free of base and dried to give a product with D.S. acetate of about 1.6.

The hydrolyzed fabric is soaked with an aqueous solution containing 45 percent urea and 16.2 percent H_3PO_4 , the pickup adjusted to 1.2 and then baked 5 minutes at 160° C. The baked fabric is washed in a solution containing 5 percent H_2SO_4 and 15 percent Na_2SO_4 , then converted to the salt form using the solution (a) of Example XXI, extracted free of salt with aqueous methanol and dried.

The properties of the final sodium phosphate cellulose-cellulose acetate are given in Table VI. It has a B.U.T. of about 23 seconds. The initial and hydrolyzed nonwoven have S.L. absorbencies of 0.22 and 0.35 respectively and B.U.T. of greater than 120 seconds.

TABLE VI

Example	Coherence, dry/wet	Tensile strength		Fabric weight	f, wet	SL, absorbency	Tensile, 1% NaCl
		Dry (c)	Wet				
XXI.....	0.42/0.30	1.3	1.03	1.10	0.72	2.1	10.06
XXII.....	0.60/0.16	3.7	1.02	2.8	0.30	1.7	10.03
XXIII.....	0.61/0.13	2.3	1.06	2.1	0.62	1.2	10.10
XXIV.....	0.22/0.07	0.7	<0.01	3.3	0.44	2.5	-----
XXV.....	0.40/0.06	1.6	1.01	2.4	0.45	2.9	10.02

¹ MD only.

EXAMPLE XXVI

This example illustrates the relationship between fiber length, coefficient of wet friction of the fabrics, and the degree of fiber entanglement with the degree of water-dispersibility of the product.

Random webs of 1 oz./yd.² weight made from 1.5 dpf. rayon staple of different lengths are entangled at 6 ypm. using the apparatus and method of Example X with four different treatments.

The different fiber lengths used are identified as follows:

- 1.36 in. average (75 percent 1 9/16 in., 25 percent 0.25 in.)
- 0.75 in. (100 percent 0.75 in.)
- 0.38 in. average (75 percent 0.25 in., 25 percent 0.75 in.)

The entanglement conditions used are identified as follows:

	Jet pressures, p.s.i.		
	Row 1	Row 2	Row 3
I.....	200	300	400
II.....	300	600	700
III.....	400	700	900
IV.....	400	1,000	1,100

Portions of the above rayon nonwoven are sewed into long lengths and converted into sodium cellulose phosphate by a continuous process. The fabric is run through an aqueous bath at 80° F. containing 50 percent urea and 16 percent H_3PO_4 ; squeezed to the desired pickup by rolls; baked for about 15 seconds by passing through an oven at 375° F.; washed with sprays of a 0.5 percent aqueous solution of sulfuric acid; converted to the salt form with sprays of an aqueous solution adjusted to a pH of 6.0 containing 1 percent N_2SO_4 , 2 percent Na_2HPO_4 , and 3 percent sodium hexametaphosphate; and then dried. One roll of fabric is run with 15 p.s.i. pressure on the squeeze rolls while 27 p.s.i. is used on a second run (modification levels A and C respectively).

Properties of the rayon fabrics and the modified fabrics are given in Table VII.

Items 9 and 11 (identical except for modification levels) have S.L. absorbencies of 1.63 and 2.20 respectively and analyze 3.51 and 4.08 percent P respectively.

The rayon fabrics used to make items 12, 13, and 14 (all A modification) when modified at the C level have a f_w of 0.79, 0.90 and 0.93 respectively with a b.u.t. of 10, 11 and 15 seconds respectively.

A portion of item 3 is soaked in 0.5 percent H_3PO_4 to convert the fibers to the free acid form, rinsed in distilled water and air dried. The salt-free product has a B.U.T. of 45 seconds, dry and wet coherence values of 0.53 and 0.35 respectively, and a coefficient of wet friction of 0.73.

Water-dispersible fabrics are made by using a blend of 50 percent polyester fiber of 1.5 dpf and 0.25 inch length and 50 percent 1.5 dpf 0.75 inch length rayon in the above procedure which yields a final product composed of polyester fibers and sodium cellulose phosphate fibers.

One such product has a B.U.T. of 75 seconds and dry and wet coherence values of 0.53 and 0.37 respectively. Other products receiving a higher degree of modification have B.U.T. as low as 8 seconds.

Qualitatively it will be observed that with a given fiber blend

and a given modification level, the B.U.T. increases as the entanglement level of the rayon fabric (as judged by the process conditions or Y.R.T.) increases-- compare items 14 and 5; 13, 11 and 3; 12 and 1. It is also noted that with a given rayon fabric the B.U.T. decreases as the chemical modification (as judged by modification level and f_w) increases compare items 1 and 2; 3 and 4; 5 and 6; 9 and 11.

The yield resistance time (YRT) values given for the rayon fabrics are a measure of the degree of entanglement of the fibers. The YRT is determined as follows:

Yield Resistance Time Test

The test consists of measuring the time required for a plunger rubbing on a fabric supported at its edges to produce a given deflection or yield in the fabric. The test is run on Appearance Retention Tester (Fabric Development Test, Brooklyn, N.Y.). The tester consists of a sliding shaft and head which is free to move up and down in a fixed arm of the frame. The shaft and head weigh 1982 grams but are counter-balanced with a weight of 1400 grams so that the net weight is 582 grams. The chromium-plated, smooth-surfaced head is 2.5 inches in diameter with a curved downward face having a radius of curvature of 3 inches. The vertical position of the head is recorded on a strip chart recorder by means of a transducer attached to the shaft and equipped with a power supply. The other main element of the tester is a hollow cylinder with an inside diameter of 6.687 inches eccentrically positioned beneath the center of the head. The cylinder is revolved at a speed of 90 revolutions per minute in such a manner that the center of the head describes about a 1.5 in. circle on the fabric which is clamped taut to the cylinder.

A square piece (about 9 in. × 9 in.) of the fabric is dipped in water. Excess water is removed by passing the fabric between wringer rolls which have been adjusted to provide a pickup of 2.5 grams of water per gram of dry fabric when four layers of a regular woven Curity cotton diaper is passed between the rolls. The wet fabric is securely clamped over the hollow cylinder. The head is lowered onto the fabric, the chart position noted, and the tester is started. The time in seconds required for the head to make a vertical deflection in the fabric of 12.5 mm. is recorded as the Yield Resistance Time (YRT).

TABLE VII

Item	Fiber length	Entangling conditions	Rayon fabric		Modification level	Sodium cellulose phosphate fabric					B. U. T. (sec.)
			YRT (sec.)	f _w		f _w	C _h , dry/wet	Tensile, lbs./in.		Tensile dry (c), lbs./in.	
								Wet (c)	1% NaCl		
1	a	IV	255	1.77	A	0.73	0.71/.35	.07	.30	3.9	44
2	a	IV	255	1.77	C	1.13	0.66/.49	.20	.59	3.9	87
3	b	IV	207	1.75	A	0.91	0.62/.25	.04	.40	3.2	37
4	b	IV	207	1.75	C	1.23	0.71/.60	.27	.68	4.1	97
5	c	IV	98	1.90	A	0.98	0.62/.44	.03	.19	2.0	33
6	c	IV	98	1.90	C	1.32	0.61/.52	.13	.36	2.1	118
7	b	III	128	1.70	C	1.03	0.68/.58	.21	.58	4.4	55
8	a	II	103	1.41	C	0.96	0.56/.38	.14	.54	3.6	33
9	b	II	78	1.54	C	0.97	0.61/.42	.16	.67	3.2	34
10	c	II	40	1.66	C	1.20	0.67/.44	.06	.30	2.5	33
11	b	II	78	1.54	A	0.66	0.52/.11	.02	.26	13.7	13
12	a	I	50	1.30	A	0.58	0.41/.02	.004	.16	12.6	6
13	b	I	40	1.33	A	0.55	0.36/.03	.004	.12	12.8	8
14	c	I	11	1.44	A	0.78	0.33/.08	.003	.10	11.3	7

¹ Measured on samples containing salt.

EXAMPLE XXVII

This example shows the use of layers of paper fibers and rayon fibers to make a dispersible product.

A random web (0.6 oz./yd.²) of 0.75 in. long rayon fibers (1.5 dpf.) is placed on a 24 × 24 mesh screen, covered with two layers of Kraft paper (0.5 oz./yd.² for each layer), and the assembly passed at 10 ypm. under a single row of water streams of Example X at a pressure of 900 p.s.i. Three such entangling treatments are given.

The entangled nonwoven fabric is treated with an aqueous solution of 37.8 percent urea and 15.5 percent H₃PO₄ to a pickup of 1.7 solution/g. of fabric, placed between aluminum plates and heated in an oven at 198–204° C. for 10–15 seconds. The baked fabric is washed in dilute sulfuric acid, converted to the sodium cellulose phosphate form, and dried. The product which is isotropic has the following properties:

Tensile strength:

Dry	lb./in.	4.0
Wet	do	.04
1% NaCl	do	.18
B.U.T.	seconds	20
S.L. absorbency		3.6

Similar properties are obtained using 38 and 45 percent of wood pulp in the starting layer to be entangled on 150 mesh screens and 150 mesh screens followed by a treatment on 24 mesh screens.

The paper fibers and rayon fibers can be intimately mixed before entanglement if desired.

Use of the above phosphorylation reaction on the Kraft paper alone produces a cellulose phosphate with a D.S. of about 0.3.

We claim:

1. A soft, water-dispersible, nonwoven fabric of fibers secured in place by random fiber entanglement providing a unitary nonwoven fabric having a weight of about 0.3 to about 5.0 oz./yd.² with an elongation in at least one direction of at least 9 percent; said fabric comprising at least 10 percent water-sensitive fibers having a water-sensitive surface and less than about 3 inches in length; the balance of said fibers comprising nonwater-sensitive fibers of less than about 0.5 inch length; said water-sensitive fibers having a wet coefficient of sliding friction f_2 of less than about 1.3 as measured on an entangled nonwoven fabric consisting essentially of the water-sensitive fibers; and said water-dispersible fabric having a dry coherence value (C_h , dry) of greater than about 0.2 and a wet coherence value (C_h , wet) of less than about 0.6 with the C_h (wet) being less than the C_h (dry).

2. A fabric as defined in claim 1 wherein the C_h (wet) is less than about 0.5 and the f_2 is less than about 1.1.

3. A fabric as defined in claim 1 wherein the C_h (wet) is less than about 0.3, the average wet tensile strength of the fabric is within the range of about 0.0 to 0.1 lbs. per inch and the f_2 is less than about 1.0.

4. A fabric as defined in claim 1 wherein at least 40 percent of the fibers are water-sensitive.

5. A fabric as defined in claim 1 wherein the water-sensitive fibers have a single layer absorbency of at least 1 when measured on a nonwoven fabric consisting essentially of the water-sensitive fibers.

6. A fabric as defined in claim 1 wherein the fabric comprises at least 40 percent water-sensitive fibers having ionizable groups and said fabric is further characterized as having a NaCl tensile strength of at least 0.05 lb./in. in 1 percent NaCl aqueous solution with the NaCl tensile strength being at least 1.5 times the tensile strength of the fabric in distilled water, a bending length of less than about 3.0 cm., and an internal bond strength of greater than about 0.08 foot pounds.

7. A fabric as defined in claim 1 which comprises essentially water-sensitive fibers.

8. A nonwoven fabric as defined in claim 1 which comprises ordered fiber groups arranged in regular parallel rows extending in two principal fabric directions and interconnected in a network by fibers randomly entangled with each other to define an ordered geometric pattern of apertures, and including fiber groups forming ridgelike protrusions on at least one face of the fabric.

9. A nonwoven fabric as defined in claim 1 wherein said fibers are entangled in a repeating pattern characterized by groups of entangled fibers forming a regular pattern of ridgelike protrusions separated by grooves lying along parallel straight lines, the ridge-forming groups being interconnected by arrays of generally parallel fibers bridging under the ridge-separating grooves and entangled in adjacent groups.

10. A nonwoven fabric as defined in claim 1 wherein said fibers are entangled in a repeating pattern of fiber bands of substantially greater width than thickness which extend continuously along substantially straight parallel axes spaced regularly in one fabric direction, the bands being interconnected by generally parallel fibers which extend laterally between adjacent bands and are held in place by fiber entanglement in the bands.

11. A nonwoven fabric as defined in claim 1 wherein said fabric is essentially a nonpatterned structure.

12. A nonwoven fabric as defined in claim 1 wherein the fabric comprises 30 percent to 90 percent of said water-sensitive fibers and 70 percent to 10 percent of said nonwater-sensitive fibers and said nonwater-sensitive fibers are more heavily concentrated in a surface layer than in the center of the fabric, the fabric having a surface layer containing at least 80 percent of the nonwater-sensitive fibers and a center containing at least 70 percent of the water-sensitive fibers.

13. A sanitary napkin comprising an absorbent core with a covering of the fabric of claim 1.

14. A diaper comprising an absorbent core sandwiched between two layers of the fabric of claim 1.

15. A fabric as defined in claim 1 wherein the water-sensitive fibers comprise modified cellulosic fibers.

16. A fabric as defined in claim 15 wherein the water-sensitive fibers have ionizable groups and said fabric is further characterized as having a NaCl tensile strength of at least 0.05 lb./in. in 1 percent NaCl aqueous solution with the NaCl tensile strength being at least 1.5 times the tensile strength of the

35

fabric in distilled water, a bending length of less than about 3.0 cm., and an internal bond strength of greater than about 0.08 foot pounds.

17. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of cyanoethyl cellulose having a degree of substitution within the range of about 0.15 to 0.80.

18. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of hydroxyethyl cellulose having a degree of substitution within the range of about 0.1 to 0.9.

19. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of carboxymethyl cellulose having a degree of substitution within the range of about 0.1 to 0.4.

20. A nonwoven fabric as defined in claim 19 wherein at least a portion of the carboxymethyl cellulose is converted to a sodium salt form.

21. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of carboxyethyl cellulose having a degree of substitution within the range of about 0.1 to 0.4.

22. A nonwoven fabric as defined in claim 21 wherein at least a portion of the carboxyethyl cellulose is converted to a sodium salt form.

23. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of cellulose phosphate having a degree of substitution within the range of about 0.10 to 0.4.

24. A fabric as defined in claim 23 wherein at least a portion of the cellulose phosphate is converted to a sodium salt form.

25. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of cellulose hemisuccinate having a degree of substitution within the range of about 0.2 to 0.40.

26. A nonwoven fabric defined in claim 25 wherein at least a portion of the cellulose hemisuccinate is converted to a sodium salt form.

27. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of cellulose sulfate having a degree of substitution within the range of about 0.1 to 0.4.

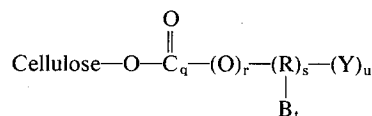
28. A nonwoven fabric as defined in claim 27 wherein at least a portion of the cellulose sulfate is converted to a sodium salt form.

36

29. A fabric as defined in claim 15 wherein at least the surface of the water-sensitive fibers are of cellulose phosphate-cellulose acetate.

30. A fabric as defined in claim 29 wherein at least a portion of the cellulose phosphate is converted to a sodium salt.

31. The fabric as defined in claim 15 wherein said water-sensitive fibers are chemically modified cellulosic fibers having a degree of substitution within the range of about 0.1 to about 1.5 as represented by the formula



wherein R is primarily a hydrocarbon group free from aliphatic unsaturation which may contain noninterfering hydrophobic substituents, either oxygen linkages and ester oxygen linkages in the chain; B is a hydrophilic substituent; Y is selected from the group consisting of hydrogen, an acid group having ionizable hydrogen attached to oxygen, and monovalent cation salts of the acid; said formula meeting the following criteria: when Y is hydrogen and t is zero, R has less than 5 carbon atoms; when Y is hydrogen and t is one, R has less than 8 carbon atoms; when Y is said acid group and t is zero, R has less than 10 carbon atoms; when Y is said salt of the acid and t is zero, R has less than 19 carbon atoms; q , r , and s are zero or one but when r is one, q and s are one; t is zero or any integer up to the number of replaceable hydrogens in R , and u is 1 or 2.

32. A nonwoven fabric as defined in claim 1 wherein said water-sensitive fibers comprise modified polyvinyl alcohol fibers.

33. A fabric as defined in claim 32 wherein said water-sensitive fibers have phosphate groups converted to a salt form.

34. A fabric as defined in claim 1 wherein said water-sensitive fibers comprise modified acrylic fibers.

35. A fabric as defined in claim 34 wherein said acrylic fibers are hydrolyzed and converted to a salt form.

36. A fabric as defined in claim 1 wherein said water-sensitive fibers comprise modified nylon fibers.

37. A fabric as defined in claim 36 wherein the nylon fibers have grafted acrylic acid groups converted to a salt form.

38. A nonwoven fabric as defined in claim 15 wherein at least the surface of said water-sensitive fibers are of cellulose hemiphthalate

50

55

60

65

70

75

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,563,241 Dated February 16, 1971

Inventor(s) FRANKLIN JAMES EVANS and CHARLES SHAMBELAN

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 33, line 62, "f₂" should read -- f_w --
line 68, at the end of the sentence and before the period
add the following -- , and being insoluble in an aqueous
solution of 16 g. NaCl, 35 g. urea, 2 g. MgSO₄ and 3 g.
Ca(H₂PO₄)₂·H₂O per liter of solution --.

Column 36, line 18, "either" should read
-- ether --.

Signed and sealed this 13th day of July 1971.

(SEAL)
Attest:

EDWARD M. FLETCHER, JR.
Attesting Officer

WILLIAM E. SCHUYLER,
Commissioner of Patents