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## PROCESS FOR PRODUCING NONWOVEN FABRICS

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5 Claims

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### ABSTRACT OF THE DISCLOSURE

Nonwoven fabrics suitable for diapers or other absorbent purposes, and which can be disposed of in sewage systems after use, are produced by entangling regenerated cellulose staple fibers with high pressure liquid streams to form a strong fabric, phosphorylating the cellulose fibers with a combination of urea and phosphoric acid or hydrogen phosphate salt to provide a degree of substitution of 0.10 to 0.40 and applying a buffered solution of an alkaline salt, e.g., to substitute sodium phosphate on the cellulose fibers. Such fibers become slippery when wet with tap water so that the fabric breaks up readily in turbulent water.

### REFERENCE TO RELATED APPLICATION

This is a continuation-in-part of my application Ser. No. 660,583 filed Aug. 15, 1967.

### FIELD OF INVENTION

This invention relates to a process for producing nonwoven fabrics comprising phosphate-modified regenerated cellulose fibers, and is more particularly concerned with production of nonwoven fabrics having adequate strength for use as diapers or other sanitary purposes and which are disposable in sewage systems after use.

Nonwoven fabrics can be prepared by treating a layer of regenerated cellulose fibers with liquid streams jetted at high pressure to entangle the fibers. When the fibers are entangled sufficiently to provide required fabric strengths, the fabric will not break up readily in water and is not disposable in sewage systems.

Phosphorylation of cellulose has been used to provide improved flame resistance. If carried too far, the cellulose may be degraded and even become soluble in water.

I have found that a combination of these treatments with proper control of the process conditions will provide fabrics of adequate strength when wet with body fluids and, in addition, provide fibers which slip apart readily when wet with ordinary city water so that the fabrics are dispersible or break up in turbulent water.

### SUMMARY OF THE INVENTION

This invention is an improvement in the production of textile-like nonwoven fabric 0.3 to 3 oz./yd.<sup>2</sup> in weight having an average strip tensile strength of at least 1 pound per inch of width from regenerated cellulose fibers. In accordance with the present invention, fibers 0.25 to 5 inches in length are entangled to form the fabric by treating a layer of the fibers with essentially columnar streams of liquid jetted at high pressure and the treatment is stopped when the wet fabric has yield resistance time R, as defined hereinafter, of 10 to 400 seconds. The fabric is then treated to decrease the wet coefficient of sliding friction of the fibers to a value  $f_w$  of from 0.3 to 1.3 by phosphorylating the cellulose fibers with a combination of urea and phosphoric acid or hydrogen phosphate salt in a mole ratio of 3:1 to 7.5:1 to provide a degree of substitution of 0.10 to 0.40 (calculated as  $-\text{PO}(\text{OH})_2$  sub-

stituents) and applying a buffered solution of an alkali metal salt to at least partially neutralize the phosphate substituents. The resulting fabric is dispersible in turbulent water but has a strip tensile strength of at least 0.1 pound per inch of width when wet with an aqueous solution containing 1% NaCl.

Details of the process are discussed subsequently and illustrated in the examples. In general, the conditions can be controlled to provide fabrics having break-up times of 5 to 100 seconds when stirred in distilled water (using the test procedure defined hereinafter) by adjusting the average fiber length, the extent of treatment with the water streams, and the extent of phosphorylation, to give a numerical total of the following terms which is greater than 1.6 and less than 4.6:

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

where R is the yield resistance time in seconds 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 total of less than 3.7. More preferably, the total should be less than 3.4 to yield products of higher water-dispersibility which can be flushed in household toilets.

The yield resistance time is a measure of the entanglement of the regenerated 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 0.75–1.56 inches.

The wet coefficient of sliding friction  $f_w$  is a measure of the chemical modification of the nonwoven. In general, for a given cellulosic nonwoven, the  $f_w$  increases as the extent of the modification with phosphate ester groups increases up to a certain level.

Nonwoven fabrics of regenerated cellulosic fibers are chemically modified with phosphate ester groups by impregnating the fabric with an aqueous solution made of urea and an acidic phosphate compound in the mole ratio of about 3:1 to 7.5:1 so that the fabric contains phosphoric acid or a hydrogen phosphate salt in an amount (calculated as  $\text{H}_3\text{PO}_4$ ) that is from about 0.2 to about 0.6 times the weight of the dry fibers, heating the fabric within the temperature range of 150° C. to about 220° C. until the regenerated cellulosic fibers have a degree of substitution (calculated as  $-\text{PO}(\text{OH})_2$  groups) within the range of about 0.10 to about 0.40 (preferably about 0.2 to 0.3), applying a buffered sodium salt solution to the fibers to convert the fabric at least in part to the sodium cellulose phosphate form and then drying the fabric.

The expression "hydrogen phosphate salt" includes salts of orthophosphoric acid such as disodium hydrogen phosphate, monosodium dihydrogen phosphate, diammonium hydrogen phosphate, and monoammonium dihydrogen phosphate.

Acidic reactions of this type cause excessive degradation of the cellulose fibers when the pH of the baked fabric is below 2.5. This can be avoided in the urea-

phosphoric acid system by maintaining at least 2000 parts per million (p.p.m.) of  $\text{NH}_3$  in the reaction atmosphere, or more preferably by having the reaction atmosphere substantially saturated with urea.

The degree of substitution of the products is calculated from the phosphorous content of acidified, salt-free samples assuming that all phosphorous is present as  $-\text{PO}(\text{OH})_2$  groups on the cellulose chain.

#### DESCRIPTION OF PREFERRED EMBODIMENTS

One class of preferred products of the process includes nonwoven fabrics of entangled fibers that will disentangle into smaller pieces in water under relatively mild agitation (such as used in the break-up time test) of 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 break-up 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 process in its narrower scope yields 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. When composed of homogeneously modified water-sensitive fibers, such fabrics preferably have single-layer (S.L.) absorbencies of at least about 2.0. The preferred products of this class are flushable in the form of diaper-size portions (such as 13 in. x 17 in.) in a household toilet without the need of predipping before flushing.

The products of the process are termed "fabrics" since they have the appearance, handle and elongation-at-the-break (at least 9% in at least one direction) of textile fabrics. The preferred products have an average elongation in both directions of at least 20%.

The fabrics are not papers and do not resemble papers at all in their essential properties.

Products of the process generally have average dry strip tensile strengths of at least 1 pound per inch of width with abrasion resistance which is adequate for many uses. The broad class of products includes products that have sufficient strength when wet with salt solution to be useful as absorbent pads, for example, while lacking the strength to be used for such products as diapers.

Another class of preferred products of the process 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 should have an average strip tensile strength of at least 0.1 pound per inch of width when wet with an aqueous solution containing 1% NaCl by weight. Preferably the strip tensile strength is about 0.2 lb./in. when wet with synthetic urine. Such products, when comprised of homogeneously modified cellulose phosphate fibers, are also characterized by an S.L. absorption of less than about 13.

By regenerated cellulose fiber is meant any artificial cellulose fiber formed by a solution process. The preferred fiber is viscose rayon of 1 to 3 denier per filament. Other suitable fibers include cuprammonium rayon, deacetylated acetate filaments, denitrated nitro-silk and the like. Regenerated fibers from alkaline solutions of low-substituted cellulose derivatives such as hydroxyethyl cellulose or methyl cellulose may also be used.

Preparatory to the formation of such nonwoven fabrics, the regenerated cellulose fibers are cut into staple lengths of 0.25 to 5 inches, with fibers from 0.75 to 1.56 inches being especially suitable. The fibers are preferably converted into a nonwoven web of random fiber orientation by air deposition of fibers, but wet (paper machine) methods may also be used for fibers up to about  $\frac{1}{2}$  inch (1.27 cm.) in length. Air deposition of fibers may be

accomplished with a Rando-Webber machine made by the Curlator Corporation of Rochester, N.Y., or other random web-forming means. Nonwoven fabrics for use in the process of this invention may also be prepared from more oriented webs made on a conventional carding machine. Such fabrics will show greater tensile strength in the machine direction MD than in the cross direction CD, suitable for use as sanitary napkin covers.

For applications in single-use disposable absorbent devices, it is preferable to keep the weight of the fabric as low as possible in order to keep the cost down. Preferred fabrics have fabric weights of about 0.3 to 3 oz./yd.<sup>2</sup> (10 to 100 g./m.<sup>2</sup>), with fabrics of about 0.8 to 1.5 oz./yd.<sup>2</sup> (27 to 50 g./m.<sup>2</sup>) being especially suitable for use in the construction of flushable sanitary napkins and diapers.

In order to give the light-weight nonwoven fabrics adequate strength and coherence for use as cover fabrics and supporting members for flushable adsorbent devices, it is necessary to give the fibers a high degree of cohesion or entanglement. This is most effectively accomplished in a preferred process using fine columnar streams of water delivered through a row of fine holes in a manifold under high pressure to the web of fibers supported by a wire screen or similar support. The process and apparatus involved are described in detail in Belgian Pat. 673,199, U.S. Pat. No. 3,403,862 to Dworjanyn and U.S. Pat. No. 3,434,188 to Summers.

The strong entangled-fiber nonwoven fabric obtained, when dried, is suitable for immediate use in the process of this invention.

The energy  $E_1$  expended during one passage under a manifold in the preparation of a given nonwoven fabric, in horsepower-hours per pound of fabric, may be calculated from the formula:

$$E_1 = 0.125 (YPG/sb)$$

where:

$Y$  = number of orifices per linear inch of manifold  
 $P$  = pressure of fluid in the manifold in p.s.i.g.  
 $G$  = volumetric flow in cu. ft./min./orifice  
 $s$  = speed of passage of the web under the streams in feet/minute  
 $b$  = the weight of the fabric produced in oz./yd.<sup>2</sup>.

The total amount of energy expended in treating the web is the sum of the individual energy values for each pass under each manifold if there is more than one.

The regenerated cellulose fibers to be modified need be soaked in the solution of urea-phosphoric acid or hydrogen phosphate salt only long enough to thoroughly wet them so that they will soak up and retain the total quantity of reagents required. Excess solution may be removed by centrifuging or the application of pressure. In the preferred continuous process, the fabric may be run through a dip tank of solution, and then between conventional squeeze rolls adjusted to leave the desired amount of liquid on the fibers. Alternatively, the exact amount of solution needed may be applied directly to the moving fabric by means of a conventional padder. In this case no excess solution need be removed.

The fibers which have been treated with the required amount of reagent solution may be dried slowly at room temperature or slightly elevated temperature. In the interest of controlling the reaction, it is preferred to dry the fabric before the high-temperature reaction operation.

The heating of the fibers to bring about reaction can be carried out by any known means which allows reasonably careful control of the time and temperature of reaction. Hot circulating air ovens, heated rolls, etc., may be used.

An appropriate choice of reaction conditions of 2 minutes at 157° C. is illustrated in Example 1. As with most chemical reactions, higher temperatures lead to

higher reaction rates and lower reaction times, while lower temperatures result in lower reaction rates requiring longer reaction times to achieve the same result. Increases in time or temperature or both may lead to several undesirable effects, if carried to extremes. The fibers may be unduly tendered or weakened or darkened in color. The degree of substitution by phosphoric ester groups may also be increased to a point where the fabric is weakened when wet with body fluids to such an extent that in-use integrity of the fabric is lost. In addition, the amount of permanently combined (non-ionic) nitrogen may increase and lower the percentage of ionizable substituent groups on the cellulose.

Applicant believes, without intending to be bound by any theory, that it is the ionized groups on the cellulose chain which are responsible for high swelling in water while the fabric is particularly sensitive to deswelling by the presence of dissolved ionized salts in the water. All body fluids such as urine, blood, or menstrual fluid contain an appreciable concentration of dissolved ionizing salts such as sodium chloride. It is theorized that it is the effect of these ionized salts which represses the swelling of the polyelectrolyte fibers in body fluids and allows the fabrics made from them to maintain adequate strength in use. When, after use, the fabrics are placed in large excess of flushing water in an ordinary toilet, the absorbed ionized salts are diluted to the extent that the fibers are free to swell. This results in fiber breakage or fiber slippage or both, with resulting disintegration of the fabric into easily flushable pieces or free fibers.

When the swelling or absorbency of the fibers in water reaches too high a value, the deswelling effect of dissolved ionized salts in body fluids may not be strong enough to keep the flushable fabric in a sufficiently strong condition to maintain it in an undamaged state during use. Excessively high absorbency or even solubility may result from the application of more severe reaction conditions such as the use of too much phosphoric acid or the use of long baking times at high temperatures as mentioned above.

When an attempt is made to apply the process of this invention to natural cotton fibers, a flushable fabric is not obtained. Even reaction conditions which convert rayon fibers to a water-soluble condition fail to make nonwoven cotton fabrics flushable, as illustrated in Example 2.

The fibers or fabric obtained by the operation of the process of this invention are contaminated with reactants, by products, and various decomposition products at the end of the high-temperature reaction. It is desirable to remove these impurities by a washing operation. It is possible to remove a major portion of the impurities by washing the fabric with alcohol and drying with heat. The alcohol washing medium removes the impurities without appreciably swelling the modified fibers and a soft, flexible fabric of good drape and hand is produced. The strongly-ionizing free acid and ammonium salt groups on the cellulose make the fibers high swelling in water.

A higher swelling product is obtained, however, if all the acid phosphate groups are converted to the sodium salt form. This is most easily accomplished in an aqueous treatment. Moreover, the use of aqueous washing and purifying media offers obvious economies over the use of alcohol or other organic washing media which are more costly and require investment in complicated recovery facilities. In order to minimize fiber swelling in the aqueous media, the modified fibers or fabric may be first treated with a dilute solution of a strong acid such as 5% sulfuric acid. This converts all of the phosphate partial ester groups to the free acid form, which is a lower swelling form. The fibers or fabric, whether acid-treated or not and preferably supported on a wire screen, are then washed free of acid and other impurities with water. They are then converted to the more useful high swelling form by treatment with a slightly alkaline buffer such as disodium phosphate to form the sodium salt of the acid

groups. In order to prevent excessive swelling and possible solution or other damage in the alkaline medium, the buffer is mixed with a solution of a deswelling ionized salt such as sodium sulfate. Other strongly ionizing salts such as ammonium sulfate or sodium citrate may also be used, but sodium sulfate appears to be the most effective and economical, and the most desirable as a minor residual solid in the final fabric. In order to prevent stiffening or harshening during drying, the fabric is dried directly while still wet with the deswelling buffered salt solution. Mechanical working during drying helps to soften the fabric and remove excess free salt. If a salt-free final product is desired, it may be obtained in a softened form by extracting the buffered salt solution with aqueous alcohol or acetone, rinsing in dry alcohol or acetone, and drying with heat. Alternatively, the use of salt may be minimized by neutralizing the free acid groups in the fabric with an aqueous alcohol solution of a buffer or dilute alkali, followed by an alcohol or acetone wash and drying with heat.

When nonwoven fabric is used for chemical modification, it may contain a small amount of less reactive or non-cellulosic fibers. For example, cotton, nylon wood pulp or polyester fibers may be mixed with the rayon staple prior to formation of the initial nonwoven batt or web. The amount of unreactive fiber will depend on length of the fibers used, the degree of entanglement, and the degree of substitution imposed on the cellulosic fiber. The unreactive fibers should be  $\frac{1}{2}$  inch (1.27 cm.) or less in length. It should be noted that the addition of some unreactive fibers to the fabric tends to reduce the biodegradability of the product. This may be undesirable where the products are to be disposed of in septic tanks.

#### TESTING PROCEDURES

Samples used for tensile tests, and fabric weight are conditioned at 70° F. (21° C.) and 65% relative humidity for at least 24 hours before testing under these conditions.

Tensile strengths and elongations are measured on 1.0 x 2 inch (2.54 x 5.08 cm.) samples at an elongation of 50% per minute on an Instron testing machine. The results are in pounds/inch (grams/centimeter), herein designated as lbs./in. (g./cm.).

Samples are soaked for 5 minutes in distilled water at 21° C. and then clamped in the tester and broken in air to determine wet tensile strength in water. In determining wet tensile strength in synthetic urine or other liquid, the soaking is done in synthetic urine or the test liquid specified.

Fabric weights are expressed in ounces/square yard (grams/square meter), herein designated as oz./yd.<sup>2</sup> (g./m.<sup>2</sup>) and are based on the weight of the air-dry fabric. Correction is made for salt content on high swelling fabrics containing sodium sulfate.

For the purposes of this invention and to evaluate the effects of body fluids on the fabrics, a salt solution termed "synthetic urine" with a composition similar to human urine (10 g. NaCl, 24 g. urea, 0.6 g. MgSO<sub>4</sub>, and 0.7 g. calcium acetate monohydrate per 964.7 g. distilled water) is employed.

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, weighs 11.73 grams and is rotated at 500 revolutions per minute. A 3 x 3-inch (7.62 x 7.62-cm.) 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 liter/minute for a period of 2 minutes. The effluent liquid from the upper side 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%.

The flushability of a napkin or piece of fabric is determined by dropping it into a bowl of a household toilet (Model F-2122 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 glass pipe 2.33 feet (71 cm.) long and 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 diamond perforations, formed into a cylinder one foot (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 inch (0.63 to 1.9 cm.) long and about 0.3 inch (0.76 cm.) apart, and bending the cut sections to stand perpendicular to the walls of the cylinder. One flushing gives a flow of 18 to 20 liters of water in 20 seconds. The toilet is flushed twice for each sample, if necessary. If no more than minor fragments of fibers are retained by the obstruction, the sample is termed "Flushable."

It has been observed that fabric samples termed "Flushable" by the above test usually have a dispersibility of at least about 20% in 2 minutes, in the small-scale dispersion test. Preferred fabrics have a dispersibility of at least 40%.

The liquid absorbency of a fiber or fabric is determined by soaking a small sample in an excess of the liquid at 25° C. unless otherwise designated (1 g. in 3,000 g. liquid). The sample is removed from the liquid and spread to cover a 5 x 5 cm. area on a 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.<sup>2</sup>. Pressure is applied for five minutes after which the sample is removed and weighed, giving the net 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 or synthetic urine, the dry weight must be corrected for solids dissolved in the liquid absorbed. All absorbencies are measured in distilled water unless otherwise noted.

#### 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. x 3 in. sample in a 100-mesh screen basket is immersed for 1 minute in 150 ml. of a 0.1% 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 x 5 in.) and the enclosed sample are covered with a 5 in. x 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 desiccator 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. x 6 in. 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. x 3 in. 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 x 1 x 0.063-in. 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% 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 the sled at a rate of 2 in. 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% 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% of the water-sensitive fibers. Fabric pattern or structure variation has been found to have little effect on the sliding friction with essentially planar fabrics.

Dispersible products have been made of fibers which have  $f_w$  values (as measured on an entangled and chemically modified nonwoven fabric containing 100% of the fibers) of from 0.3 to 1.3. Values of about 0.5 to 1.1 are usually preferred.

The  $f_w$  values of the unmodified regenerated cellulose fibers 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 1.36-inch average fiber length to 1.44 for a 0.38-inch average fiber length. At a near maximum entangling condition, the  $f_w$  for the same fiber lengths ranges from 1.77 to 1.90, respectively. When rayon nonwoven fabrics are modified to a watersensitive fiber form, the  $f_w$  is no more than about 80% of the starting rayon  $f_w$  values for the lesser dispersible products and can be as low as 40% (or lower) of the starting values for the highly dispersible products.

#### YIELD RESISTANCE TIME

The yield resistance time (abbreviated YRT or R), a measure of the degree of entanglement of the fibers in a fabric, is determined by measuring the time required for a plunger rubbing on the fabric supported at its edges to produce a given deflection or yield in the fabric. The test is run on an 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 weight 1982 grams but are counterbalanced 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. x 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 pick-up 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).

#### BREAK-UP TIME (B.U.T.)

The break-up time is determined by dropping a 3 in. x 3 in. 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 7/8 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-in. 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 1 1/8 inches. B.U.T. is the time in seconds 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 1

Crimped rayon staple fiber of 0.75 inch (1.9 cm.) length and 1.5 denier per filament is fed in sequence to a Garnett feeder, a Garnett card, a "Rando-Feeder," and a "Rando-Webber" to form a 1 oz./yd.<sup>2</sup> (34 g./m.<sup>2</sup>) web. The rayon fibers are entangled to give strength and integrity to the web using the method of Belgian Patent 673,199. In this process, the web of rayon fibers is supported on a screen containing 24 wires per inch (2.54 cm.) in both directions, with 16% open area, and passed at 5 yards (4.57 meters) per minute under 4 rows of stationary jets supplied with 65° C. water at 200, 500, 700 and 800 lbs./in.<sup>2</sup> (14.1, 35.2, 49.2, and 56.2 kg./cm.<sup>2</sup>), respectively. Each row of jets contains 40 holes/inch (40 holes/2.54 cm.), each hole being 0.005 inch (0.127 mm.) in diameter. The columnar streams of water deliver energy at the rate of 1.03 horsepower-hours per pound (2.27 horsepower-hours per kilogram) of fibers. The dried nonwoven fabric has a weight of 1.10 oz./yd.<sup>2</sup> (37.3 g./m.<sup>2</sup>). The tensile strength is 5.08 lbs./in. (907 g./cm.) in the machine direction (MD) and 2.97 lbs./in. (530 g./cm.) in the cross direction (CD). The fabric has a calculated YRT value of about 130 seconds.

The nonwoven fabric is uniformly padded with 2.02 times its weight of a reagent solution prepared by dissolving 100 parts of commercial urea pellets in 56.5 parts of deionized water at 185° F. (85° C.) and adding 43.5 parts of commercial 85% phosphoric acid to the solution. The ratio of phosphoric acid to urea in the reagent solution is 1/2.7 and the fabric retains 0.37 times its weight of H<sub>3</sub>PO<sub>4</sub>. The wet fabric is fastened on a tenter frame and

passed through a circulating air oven with exposure to balanced-flow, low-velocity air containing at least 2000 p.p.m. NH<sub>3</sub> at 315° F. (157° C.) for 2 minutes. The resulting cellulose phosphate ester fabric is cut into pieces which are washed in 5% sulfuric acid, squeezed free of excess liquid, and neutralized to the high swelling form in a buffered salt solution containing 2% disodium phosphate and 8% sodium sulfate, adjusted to a pH of about 8.5. The pieces of fabric are squeezed free of excess liquid, pressed between blotters to reduce the amount of retained salt solution, and dried at 40° C. in a home laundry dryer containing several baseballs of 9.7 cm. diameter to soften the fabric by mechanical working and remove excess salt. The final product has the following properties:

Degree of substitution—0.23  
 Dispersibility—20.7%  
 Absorbency—7.35 g./g.  
 Fabric weight—1.33 oz./yd.<sup>2</sup>  
 Tensile strength (MD), dry—3.64 lb./in.  
 Tensile strength (MD), wet (tap water)—0.34 lb./in.  
 Tensile strength (MD), wet (syn. urine)—0.375 lb./in.

The fabric is used as the outer wrapper fabric in the construction of sanitary napkins having a wood fluff and crepe wadding absorbent core. These easily pass the standard flushability test.

#### EXAMPLE 2

This example shows that a nonwoven cotton fabric, even when modified by the use of stronger reaction conditions, does not become flushable through the phosphoric acid-urea treatment.

El Paso combed cotton is passed through the "Rando-Webber" to form a 1 oz./yd.<sup>2</sup> (34 g./m.<sup>2</sup>) web. The web is given strength and coherence by entangling the fibers by a method similar to that in Example 1. In this case, the screen and web of cotton fibers is passed at 5 yds. (4.57 m.) per minute under 3 rows of stationary jets supplied with water at 500, 700 and 800 lbs./in.<sup>2</sup> (35.2, 49.2 and 56.2 kg./cm.<sup>2</sup>), respectively, delivering energy at 1 horsepower-hour per pound (2.2 horsepower-hour per kg.) of fabric. A cover screen is placed over the web of fibers during passage under the first manifold to prevent disruption of the sheet. The resulting entangled cotton non-woven fabric has a dry tensile strength in the machine direction (MD) of 3.0 lbs./in. (536 g./cm.). In a number of trials, the cotton fabrics are given various pretreatments intended to increase the reactivity or internal availability of the cotton fibers to the reagents. The fabric is cut into 8 in. x 8 in. (20.3 cm. x 20.3 cm.) pieces and pretreated as indicated in Table I, the various symbols having the following meanings:

A—Sample dewaxed by boiling for 1 hour in 0.1% Na<sub>2</sub>PO<sub>4</sub> and 0.1% "Tide," a home laundry detergent. Washed in distilled water.

B—Sample boiled in distilled water for 1 hour. Water displaced by rinsing 3 times in fresh portions of methanol, then rinsing 3 times in fresh portions of benzene. Air-dried.

The dry fabrics are immersed in a reaction mixture containing 248 parts of urea, 108 parts of 85% H<sub>3</sub>PO<sub>4</sub>, and 144 parts of water (ratio of H<sub>3</sub>PO<sub>4</sub> to urea, 1/2.7). The fabrics are then couched or lightly blotted with absorbent paper to achieve the various reagent retentions listed in the table. The wet fabrics are then reacted in a circulating hot-air oven at 150° C. for 15 minutes. The fabrics are next washed in 4% sulfuric acid, and converted to the sodium salt in a buffered solution containing 17% Na<sub>2</sub>SO<sub>4</sub> and 3% Na<sub>2</sub>HPO<sub>4</sub>, at pH 8.5. The fabrics are finally blotted free of excess liquid and dried in a home laundry dryer at 40° C. The wet tensile properties and chemical analyses are shown in Table 1. The test results show that cotton fabrics retain a water-wet tensile strength far too high to permit disintegration in tap water, even when the fibers have been pre-activated and treated with

a large amount of reagent. None of the modified cotton fabrics are flushable. Rayon fabrics, treated with a similar excess of reagent (outside the scope of this invention) become too weak to maintain unitary fabric form in synthetic urine or body fluids.

TABLE I

Sample No.	Pre-treatment	Reagent retained by fabric, g./g.		Anal. percent P*	Wet tensile strength, lbs./in. (g./cm.)	
		Total	H <sub>3</sub> PO <sub>4</sub>		Synth. urine, MD/CD	Tap water, MD/CD
1	(None)	0.806	0.148	2.3(2.9)	2.00/1.30(357/232)	
2	do	4.44	.815	3.8(4.1)	2.24/1.40(400/250)	
3	A	.862	.158	2.9(3.4)		0.99/0.23(177/41)
4	A	3.80	.698	3.6(4.0)		1.19/1.08(212/193)
5	A+B	1.09	.200	3.8		0.79/0.66(139/118)
6	A+B	6.85	1.26	4.6		1.39/1.32(248/336)
(Rayon)		1.70	.312	3.8(4.6)	0.29/0.26(52/46)	
Do		5.15	.945	(5.8)	TWTT**	
Do		1.44	.264	3.7(4.6)	0.26/0.20(46/36)	
Do		5.88	1.08	8.7(6.4)	TWTT**	

\*1st figure for percent P is based on a distilled water washed sample dried. The value in parenthesis is on the as-made sample containing soluble salts from the process.

\*\*TWTT=Too weak to test.

## EXAMPLE 3

Nonwoven fabric, prepared identically to the fabric of Example 1, is uniformly padded with 2.24 times its weight of a reagent solution prepared by dissolving 100 parts of commercial urea pellets in 57.6 parts of tap water, heated to maintain about 45° C., and adding 42.4 parts of commercial 85% phosphoric acid to the solution. The ratio of phosphoric acid to urea in the reagent solution is 1/2.8 and the fabric retains 0.40 times its weight of H<sub>3</sub>PO<sub>4</sub>. The wet fabric is fastened on a tenter frame and passed through a circulating air oven with exposure to balanced flow, low velocity air at 325° F. (163° C.) for 1.5 minutes. The resulting cellulose phosphate ester fabric supported on a screen conveyor is passed at 1 yd./min. (.92 m./min.) under sprays of 30° C. tap water issuing at 0.25 gal./min./inch of width (0.37 l./min./cm.) and into the nip of rubber covered squeeze rolls set at 30 lb./linear inch (5.4 kg./cm.). The fabric picks up 1.1 parts of water per part of fabric. The fabric next passes from the nip onto a screen conveyor and underneath sprays of 17% Na<sub>2</sub>SO<sub>4</sub>/3% Na<sub>2</sub>HPO<sub>4</sub> adjusted to pH 8.5 issuing at 0.45 lb./min./inch of width (0.08 kg./min./cm.) and into the nip of a squeeze roll where it is plied with 140 lb./ream (6.25 oz./yd.<sup>2</sup>, 212 g./m.<sup>2</sup>) bleached sulfite blotting paper. The fabric retains 1.15 parts of salt solution per part of fabric. The fabric is cut into pieces which are dried at 40° C. in a home laundry dryer (without the baseballs used in Example 1). The resulting product has the following properties:

Degree of substitution—0.25 (4.56% P)  
 Dispersibility—32.1%  
 Absorbency—8.27 g./g.  
 Fabric weight—1.42 oz./yd.<sup>2</sup> (salt free, bone dry) (48.2 g./m.<sup>2</sup>)  
 Tensile strength (MD), dry—3.42 lb./in. (611 g./cm.)  
 Tensile strength (MD), wet (tap water)—TWTT<sup>1</sup>  
 Tensile strength (MD), wet (syn. urine)—0.354 lb./in. (63.3 g./cm.)

<sup>1</sup> Too weak to test.

The fabric is used as the outer cover in the construction of flushable diapers having wood fluff and crepe wadding absorbent cores. These diapers are readily flushable in the standard flushability test.

## EXAMPLE 4

This example illustrates a continuous process.

A nonwoven fabric of 1.0 oz./yd.<sup>2</sup> (34 g./m.<sup>2</sup>) weight is made with the fiber and apparatus of Example 1 under substantially the same water pressures.

The nonwoven fabric is dipped into a tank containing an aqueous solution of 18% H<sub>3</sub>PO<sub>4</sub> and 50% urea, passed between squeeze rolls set at 30 lb./linear inch (5.4 kg./cm.) pressure which leaves 1.58 lb. of aqueous solution per lb. of dry fabric on the fabric. The wet fabric is car-

ried on a screen conveyor through a circulating air oven which passes air at 200° C. at a velocity of about 200 feet (62 meters) per minute. Residence time in the oven is 15 seconds.

5 The baked fabric is then carried on a screen under-

neath sprays of tap water for washing. Excess water is removed by squeeze rolls set at a pressure of 60 lbs./linear inch (10.8 kg./cm.). The wet fabric is then carried under sprays of an aqueous solution containing 3% Na<sub>2</sub>SO<sub>4</sub> and 12% Na<sub>2</sub>HPO<sub>4</sub> adjusted to a pH of 8.5 issuing at a rate of about 1 lb./minute/inch of width (0.18 kg./min./cm.). Excess solution is removed by squeeze rolls set at the same pressure as the rolls for removing water. The fabric passes continuously through the above chemical modification steps at a speed of 12 feet (3.7 meters) per minute.

The wet, modified fabric is then passed at 9 feet (2.74 meters) per minute through an oven where it is subjected to air at 56° C. issuing from pairs of opposed slots transverse to the direction of fabric so that the fabric flutters between the opposed jets of air. The dry fabric is then softened by passing at the same speed 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 resulting product has the following properties:

Degree of substitution—0.23 (4.06% P)  
 Dispersibility—31%  
 Absorbency—5.1 g./g.  
 Fabric weight—1.07 oz./yd.<sup>2</sup> (salt free, bone dry) (36 g./m.<sup>2</sup>)  
 Tensile strength (MD), dry—5.0 lb./in. (900 g./cm.)  
 Tensile strength (MD), wet (tap water)—0.01 lb./in. (1.8 g./cm.)  
 Tensile strength (MD), wet (syn. urine)—0.37 lb./in. (64.5 g./cm.)

## EXAMPLE 5

A nonwoven fabric of 1.2 oz./yd.<sup>2</sup> (41 g./m.<sup>2</sup>) weight is made with the fiber and apparatus of Example 1 using a maximum water pressure of 600 p.s.i. (42 kg./cm.<sup>2</sup>) and a total energy of treatment of about 0.5 HP-hr./lb. of fiber (0.7 calories/gram). The fabric has a calculated YRT value of about 70.

The nonwoven rayon fabric is passed through a dip tank containing an aqueous solution of 50% urea and 18% H<sub>3</sub>PO<sub>4</sub> at room temperature with a residence time of about 10 seconds. The fabric is passed through a pair of rubber-covered nip rolls and squeezed to leave about 2.0 lbs. of aqueous solution per lb. of fabric on the fabric. The wet fabric is passed over about a 3-foot (0.9-meter) air gap to a commercial fabric oven (Benz) where it is subjected to opposed jets of air at 164° C. for a period of about 91 seconds. The treated fabric which passes continuously from the feed roll through the padding tank and the oven is wound up at about 6.3 feet (1.9 meters) per minute. The hot air is recirculated and controlled so as to contain at least 2000 p.p.m. of NH<sub>3</sub>.

The general procedure and apparatus of Example 4 are used to wash the fabric with tap water and then to convert it to the salt form by spraying with an aqueous solution containing 3% Na<sub>2</sub>HPO<sub>4</sub>, 17% Na<sub>2</sub>SO<sub>4</sub> and 1% sodium hexametaphosphate adjusted to a pH of 6.0. The fabric is then dried and softened as in Example 4 using air of 35° C. to 40° C. in the dryer and 3 treatments through the grooved rolls at 3 to 5 feet (0.9 to 1.5 meters) per minute. The final product has the following properties:

Degree of substitution—0.26–0.30

Fabric weight—2.3 oz./yd.<sup>2</sup> salt free, dry (78 g./m.<sup>2</sup>)

Tensile strength (MD), dry—6.1 lb./in. (1100 g./cm.)

Tensile strength (MD), wet (tap water)—0.02 lb./in. (36 g./cm.)

Tensile strength (MD), wet (syn. urine)—0.77 lb./in. (140 g./cm.)

Wet coefficient of wet friction—0.72

### EXAMPLE 6

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.<sup>2</sup> weight made from 1.5 d.p.f. rayon staple of different lengths are entangled at 6 ypm. using the apparatus and method of Example 1 with 4 different treatments.

The different fiber lengths used are identified as follows:

(a) 1.36 in. average (75% 1–9/16 in., 25% 0.25 in.)

(b) 0.75 in. (100% 0.75 in.)

(c) 0.38 in. average (75% 0.25 in., 25% 0.75 in.)

15 p.s.i. pressure on the squeeze rolls (modification level A), while 27 p.s.i. is used on a second run (modification level C).

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

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% P, respectively.

The rayon fabrics used to make items 12, 13 and 14 (at modification level A), when modified at the C level have  $f_w$  values of 0.79, 0.90 and 0.93, respectively, with break-up time (B.U.T.) of 10, 11 and 15 seconds, respectively.

A portion of item 3 is soaked in 0.5% H<sub>3</sub>PO<sub>4</sub> 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% polyester fiber of 1.5 d.p.f. and 0.25-inch length and 50% 1.5 d.p.f. 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. value 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. values 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.

TABLE II

Item:	Fiber length	Entangling conditions	Sodium cellulose phosphate fabric							
			Rayon fabric		Modification level	Tensile, lbs./in.			B.U.T. sec.	
			Y.R.T. sec.	$f_w$		$f_w$	wet <sup>1</sup>	1% NaCl		Tensile Dry <sup>1</sup> lbs./in.
1.....	a	IV	255	1.77	A	0.73	.07	.30	3.9	44
2.....	a	IV	255	1.77	C	1.13	.20	.59	3.9	87
3.....	b	IV	207	1.75	A	0.91	.04	.40	3.2	37
4.....	b	IV	207	1.75	C	1.23	.27	.68	4.1	97
5.....	c	IV	98	1.90	A	0.98	.03	.19	2.0	33
6.....	c	IV	98	1.90	C	1.32	.13	.36	2.1	118
7.....	b	III	128	1.70	C	1.03	.21	.58	4.4	55
8.....	a	II	103	1.41	C	0.96	.14	.54	3.6	33
9.....	b	II	78	1.54	C	0.97	.16	.67	3.2	34
10.....	c	II	40	1.66	C	1.20	.06	.30	2.5	33
11.....	b	II	78	1.54	A	0.66	.02	.26	2.7	13
12.....	a	I	50	1.30	A	0.58	.004	.16	2.6	6
13.....	b	I	40	1.33	A	0.55	.004	.12	2.8	8
14.....	c	I	11	1.44	A	0.78	.003	.10	2.1	7

<sup>1</sup> Measured on 1.6 x 2-inch samples with 1.5-inch gauge length.

<sup>2</sup> Measured on samples containing salt.

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 nonwovens 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% urea and 16% H<sub>3</sub>PO<sub>4</sub>; squeezed to the desired pick-up by rolls; baked for about 15 seconds by passing through a 191° C. oven wherein the atmosphere contains at least 2000 p.p.m. of NH<sub>3</sub>; washed with sprays of a 0.5% aqueous solution of sulphuric acid; converted to the salt form with sprays of an aqueous solution adjusted to a pH of 6.0 containing 1% Na<sub>2</sub>SO<sub>4</sub>, 2% Na<sub>2</sub>HPO<sub>4</sub>, and 3% sodium hexametaphosphate; and then dried. One roll of fabric is run with

### EXAMPLE 7

A random web of 0.75-inch length, 1.5 d.p.f. rayon staple of 0.8 oz./yd.<sup>2</sup> weight is entangled at 6 y.p.m. using the apparatus and method of Example 1 using pressures of 200, 650 and 1000 p.s.i. on the three rows of orifices. The strong entangled nonwoven fabric has a calculated yield resistance time (Y.R.T. value) of about 150 seconds.

The nonwoven rayon fabric is passed through a tank containing an aqueous solution of 15.30% H<sub>3</sub>PO<sub>4</sub> and 49.63% urea at about 27° C. The wet fabric is pressed with rolls to leave 1.80 grams of solution for each gram of original fabric. The wet fabric is conducted on a belt at 5.5 y.p.m. through a double-chambered oven. The first chamber is 3 feet long and has a 9-inch long opening at the end of the chamber through which 190° C. air is blown down through the fabric at a linear velocity of about 500' a minute to dry the fabric. The dry fabric is carried through an insulated section about 12 inches long and into the second or reaction chamber. The fabric

is protected from downward flow of the hot air in the 3-foot long reaction chamber by a cover plate located about 0.75 inch above the fabric and having vertical sides extending to within about 0.12 inch from the top of the belt edge. A second solid plate is positioned close below the belt. The hot air flows around the sides of the cover plate and the bottom plate to a lower exit where it is recirculated. This provides an atmosphere for the fabric that is saturated with urea. The fabric temperature in the reaction chamber is 190° C. and the air temperature is somewhat higher (about 195° C.).

The reacted fabric is then washed by passing concurrent streams of 0.6% H<sub>2</sub>SO<sub>4</sub> at the total rate of 30 gallons/minute onto the fabric from two Fourdrinier paper machine-type of washers (with a headbox). The washed fabric is then treated with a concurrent stream of an aqueous 6.0 pH solution containing 1% Na<sub>2</sub>SO<sub>4</sub>, 2% Na<sub>2</sub>HPO<sub>4</sub>, 3% sodium hexametaphosphate and 0.5% of N-oleyl betaine (as a softening agent). The fabric is then dried by forced air.

The dry fabric has the following properties:

Weight (including 8.2% salts)—1.13 oz./yd.<sup>2</sup>  
Tensile strength (MD), dry—3.0 lb./in.  
Tensile strength (MD) 1% NaCl solution—0.11 lb./in.  
Break-up time (B.U.T.)—5.1–5.9 seconds

The fabric has a calculated degree of substitution of about 0.25 and coefficient of wet friction  $f_w$  of below 0.5.

#### EXAMPLE 8

A random web of 0.75-inch length, 1.5 d.p.f. rayon staple of 1.0 oz./yd.<sup>2</sup> is hydraulically entangled at 6 y.p.m. using the apparatus and method of Example 1 using pressures of 300, 600 and 700 p.s.i. on the 3 rows of orifices. The strong, entangled, nonwoven fabric has a calculated (Y.R.T.) value of about 80.

The nonwoven rayon fabric is passed through a tank containing an aqueous solution of 50% urea and 18% NaH<sub>2</sub>PO<sub>4</sub>. The fabric is pressed with rolls to leave about 2 grams of solution per gram of original fabric. The wet fabric is passed on a conveyor belt through an oven where 190° C. air is passed through the fabric for 30 seconds. The air is recirculated and reheated with low additional make-up air so that the air contains urea and 9900 parts per million of NH<sub>3</sub>.

The dry reacted fabric is washed with aqueous buffering solution of Example 7 that also contains 0.2% of a commercial paper brightening agent. This treatment replaces the two-step procedure of Example 7 and yields the sodium cellulose phosphate fabric. The fabric is air dried. Properties of the fabric are given below:

Tensile strength, dry (MD)—1.4 lb./in.  
Tensile strength, 1% aqueous NaCl, (MD)—0.16 lb./in.  
Tensile strength, distilled water, (MD)—0.02 lb./in.  
Degree of substitution—0.20  
Break-up Time (B.U.T.)—8 seconds

The product has a calculated coefficient of wet friction  $f_w$  of less than about 0.5.

I claim:

1. In the production of textile-like nonwoven fabric 0.3 to 3 ounces per square yard in weight having an average dry strip tensile strength of at least 1 pound per inch of width, wherein regenerated cellulose fibers 0.25 to 5 inches in length are entangled to form the fabric by treating a layer of the fibers with essentially columnar streams of liquid jetted at high pressure; the improvement for producing fabric that is dispersible in turbulent water but has a strip tensile strength of at least 0.1 pound per inch of width when wet with an aqueous solution containing 1% NaCl; wherein the improvement comprises (A) stopping said treatment with the liquid streams when the wet fabric has a yield resistance time (R) of 10 to 400

seconds and (B) decreasing the wet coefficient of sliding friction of the fibers to a value  $f_w$  of from 0.3 to 1.3 by phosphorylating the cellulose fibers with a combination of urea and phosphoric acid or hydrogen phosphate salt in a mole ratio of 3:1 to 7.5:1 to provide a degree of substitution of 0.10 to 0.40, calculated as —PO(OH)<sub>2</sub> substituents, and applying a buffered solution of an alkali metal salt to at least partially neutralize the phosphate substituents.

2. The process defined in claim 1 wherein step (B) comprises preparing an aqueous solution of urea and phosphoric acid in a mole ratio of 3:1 to 7.5:1, applying the solution to said fabric to impregnate the fabric with an amount calculated as H<sub>3</sub>PO<sub>3</sub> of from 0.2 to 0.6 times the weight of dry cellulose fibers, heating the impregnated fabric within the temperature range of 150° to 220° C. in an atmosphere containing at least 2000 parts per million of ammonia until the degree of substitution is sufficient to provide a wet coefficient of sliding friction  $f_w$  of 0.3 to 1.3, and applying an aqueous buffered solution of sodium salt to the fabric to form sodium cellulose phosphate.

3. The process defined in claim 1 wherein regenerated cellulose fibers having an average length (L) of 0.75 to 1.56 inch are formed into a chemically modified nonwoven fabric weighing 0.8 to 1.5 ounces per square yard, using an extent of treatment in step (B) which provides a degree of substitution of 0.2 to 0.3 and a wet coefficient of sliding friction  $f_w$  of about 0.5 to 1.1 to produce a dispersible fabric having a strip tensile strength of 0.2 to 1.0 pounds per inch of width when wet with an aqueous solution containing 1% sodium chloride, 2.4% urea, 0.06% magnesium sulfate and 0.07% calcium acetate monohydrate, by weight.

4. The process defined in claim 1 wherein a chemically modified nonwoven fabric having a break-up time of 5 to 100 seconds when stirred in distilled water is produced by adjusting the average fiber length (L), the extent of treatment in step (A) as determined by the yield resistance time (R), and the extent of treatment in step (B) as determined by the wet coefficient of sliding friction  $f_w$ , to give a numerical total of the following terms which is greater than 1.6 and less than 4.6:

$$1.38 + 0.0180R - 1.60L + 0.731f_w \\ + 0.00292RL - 0.0000480R^2 \\ + 0.476L^2 + 0.847f_w^2$$

5. The process defined in claim 1 wherein said buffered salt solution is an aqueous solution of about 1% to 25% Na<sub>2</sub>SO<sub>4</sub> and 2% to about 15% Na<sub>2</sub>HPO<sub>4</sub>, by weight, the solution having a pH within the range of about 6 to about 8.5 and being applied to the fabric in a quantity of from 1 to 2 times the dry fabric weight.

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