

United States Patent

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3,439,678 4/1969 Thomas..... 128/284
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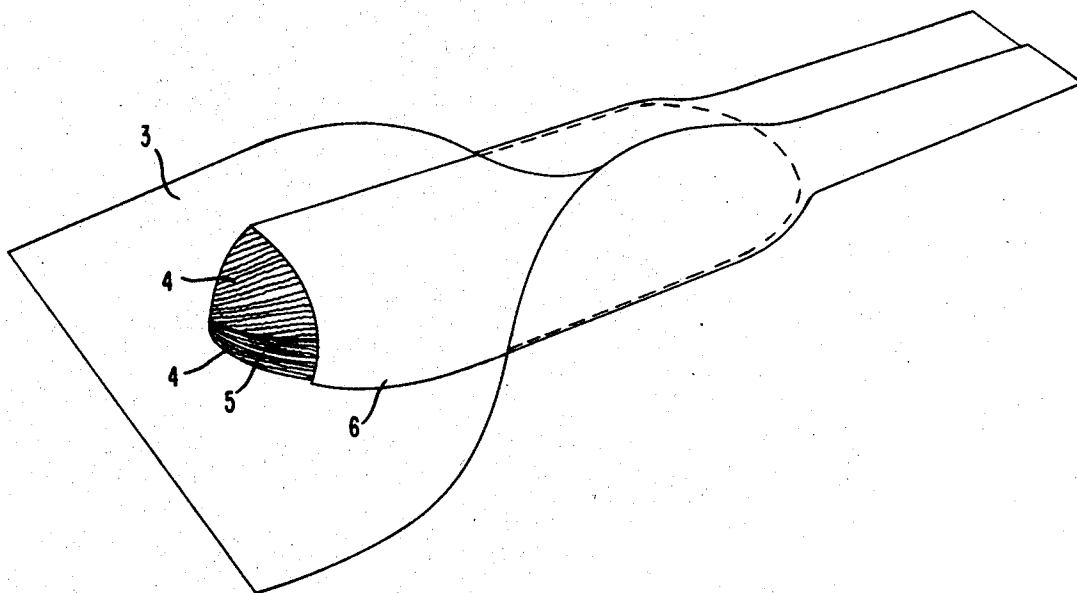
[54] FLUSHABLE SANITARY NAPKINS 12 Claims, 2 Drawing Figs.

[52] U.S. Cl..... 128/290
[51] Int. Cl..... A61f 13/16
[50] Field of Search..... 128/284,
287, 290, 296

[56] References Cited

UNITED STATES PATENTS
3,067,746 12/1962 Bletzinger et al. 128/290
3,370,590 2/1968 Hokanson et al. 128/290
3,407,814 10/1968 George et al. 128/290

ABSTRACT: A genuinely flushable sanitary napkin having an absorbent core of loosely felted fibrated wood pulp; a fluid barrier of a cellulosic fiber substrate coated with a fluoropolymer for retaining body fluids within the core, and a soft, water dispersible wrapper exhibiting pleasing aesthetics during use for encasing the core and fluid barrier and for maintaining the napkin in a unitary assembly during use, the wrapper being a nonwoven fabric of randomly entangled cellulosic fibers chemically modified to allow easy disintegration of the fabric in tap water for promoting flushability of the entire napkin, the wrapper is further characterized as having a unique combination of tensile properties while dry, or when wet with tap water, or while wet with body fluids. Also disclosed is a fluid barrier of a cellulosic sheet substrate coated with a fluoropolymer, or paraffin wax, the sheet having a combination of wet and dry tensile properties which yields good performance during use of the napkin of which it is a part while promoting flushability of the napkin.



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3,542,028

FIG. 1

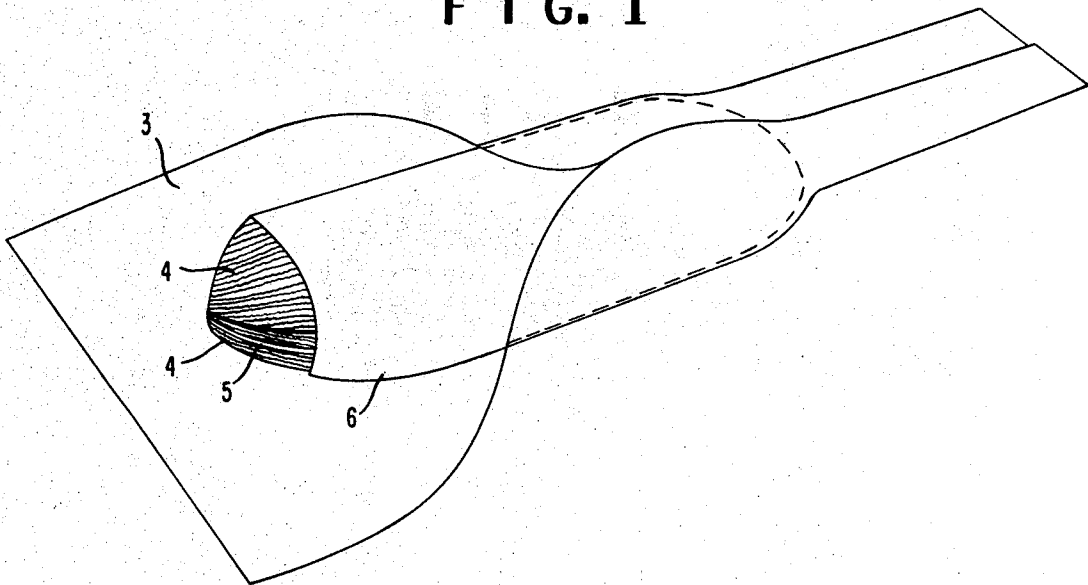
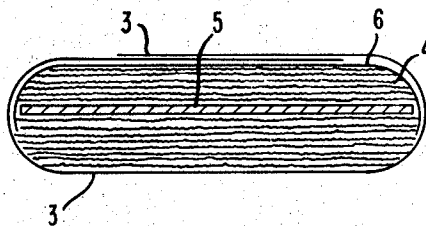


FIG. 2



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FLUSHABLE SANITARY NAPKINS

This application is a continuation-in-part of our copending application, Ser. No. 621,547 filed Mar. 8, 1967 now abandoned.

BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates to absorbent products in the form of sanitary napkins. More specifically, it provides an improved and novel sanitary napkin which is genuinely disposable by flushing in any home toilet facility.

Disposable sanitary napkins have been commercially available for many years and have met with great success and wide acceptance in the more industrialized countries of the world. A long-felt need has existed for a more convenient method for the disposal of soiled napkins. Numerous sewers have been stopped up in attempts to flush away conventional napkins to avoid the necessity of disposal by ordinary means. Although the absorbent core of the napkin is usually composed mainly of wood fluff, which by itself would be easily flushable, the moisture barrier and more especially the fabric cover of the prior art napkins are relatively unaffected by water so that they maintain their structural integrity in flushing. The fabric cover of the napkin of the present invention is water sensitive and has a unique combination of dry and wet tensile properties yielding good performance in use with the wet tensile properties sufficiently low to give genuine flushability in ordinary home toilet.

2. Description of the Prior Art

The need for a flushable napkin was recognized early in the development of the art. British Pat. No. 282,447 attempts a partial solution by providing a flushable core and a repellent-treated barrier which are separated from the nonflushable cover fabric and flushed. The cover fabric remains to be disposed of otherwise. Other patents disclosing repellent-treated napkin barriers include the following U.S. Pat. Nos.: Atkinson, 2,643,656, dated Jun. 30, 1963; Harwood et al. 3,029,817, dated Apr. 17, 1962; Bletzinger 3,067,746, dated Dec. 11, 1962; and Torr, 3,070,095, dated Dec. 25, 1962. The last-mentioned patent also discloses the use of paper cover to achieve flushability for the whole structure with the resultant disadvantages of harshness, noisiness, possible weakness, and difficult flushability. Morse, U.S. Pat. No. 3,078,849, dated Feb. 26, 1963, discloses a napkin incorporating a fluid-sensitive, temporary barrier within the absorbent core for spreading the body fluids; but makes no provision for a water-sensitive cover fabric. No commercially successful flushable sanitary napkin has appeared on the market in the history of the art.

SUMMARY OF THE INVENTION

The present invention provides an economical, soft, absorbent, easily conformable, strong-in-use sanitary napkin which is fully flushable in home toilets by virtue of the easy water-dispersibility of the absorbent core, the repellent-treated barrier, and the novel water-sensitive nonwoven fabric cover. The flushable sanitary napkin of this invention comprises:

1. A flushable absorbent elongated wood pulp core covered on one side and both edges by;
2. A flushable barrier consisting of one or more cellulosic sheets to one or both sides of which has been applied in a substantially uniform manner, a fluoropolymer water repellent at the rate of 0.02 to 0.15 mg./cm.² or a household type paraffin wax at the rate of 0.07 to 0.25 mg./cm.², the assembly being enclosed in;
3. A flushable cover sheet of nonwoven fabric given integrity by random fiber entanglement in which from 10 to 100 percent of the fibers are water-sensitive fibers. Water-sensitive fibers are those with a wet coefficient of sliding friction of less than 0.90 (preferably less than 0.70) as measured on a nonwoven fabric consisting essentially of the salt free fibers being tested, which fibers must also be insoluble in synthetic urine.

Suitable water-sensitive 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.

Many of the water-sensitive fibers useful in this invention, such as chemically modified cellulose, hydrolyzed vinyl polymers, condensation polymers, etc., are disclosed in Belgian Pat. No. 706,510, granted May 14, 1968. Accordingly, the disclosure of Belgian Pat. No. 706,510, granted May 14, 1968, is herein incorporated by reference.

By flushable is meant the ability to pass the standard Flushability test to be described. It has been found that in order to ensure flushability, the nonwoven cover fabric should have an average wet tensile strength in distilled water of not more than about 0.1 lb./in. (18 g./cm.). At the same time, to ensure integrity in use, the fabric must have an average dry tensile strength of at least about 1.0 lb./in. (180 g./cm.) and preferably about 1.5 lb./in. (268 g./cm.) and at least about 0.05 lb./in. (9 g./cm.) average tensile strength when wet with a test solution termed "synthetic urine", to be described hereinafter. To ensure softness and comfort, the fabric should have a standard bending length of less than about 3 cm.

With reference to the fluid barrier, by the term fluoropolymer is meant vinyl-type polymers, copolymers, and segmented or graft copolymers in which at least a major portion of the vinyl monomer units contain monovalent perfluoroalkyl radicals $\text{CF}_3-(\text{CF}_2)_n-$ in which n is an integer from 2 to 13.

In addition to fluoropolymer type fluid barrier, a household type paraffin wax water repellent or other materials described herein may be used in the fluid barrier.

The term homogeneous water-sensitive fibers refers to fibers having an absorbency of 4 g./g. or more in the standard Absorbency test to be described. For some types of fibers, absorbencies of 10 to 15 g./g. or even more may be desirable.

In addition, the absorbent nonwoven fabrics disclosed in Belgian Pat. No. 706,510, granted May 14, 1968, are also suitable for use in this invention.

DESCRIPTION OF THE DRAWING

In the accompanying drawing, FIGS. 1 and 2 illustrate a product of this invention. FIG. 1 is a view of a sanitary napkin which has been unwrapped at one end to reveal the assembly of the inner core and barrier while FIG. 2 represents a cross section of the center of the same napkin fully assembled. The napkin comprises an absorbent core covered with a flushable fabric outer wrapper 3 which has end extensions forming attachment tabs in the usual manner. Within the cover is the absorbent core formed of a composite of a fluid distributing member 5 such as crepe wadding or embossed wood fluff batting, and loosely felted fibrated wood pulp 4 commonly known as fluff. Surrounding the core on the bottom and sides is creped tissue 6 which has been treated with a fluoropolymer or paraffin wax water repellent to act as a fluid barrier. The barrier tissue is made substantially coextensive, longitudinally, with the absorbent core.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

A successful and effective modern sanitary napkin must contain, in one form or another, three major components: an absorbent core, a fluid barrier, and an outer wrapper.

The absorbent core must soak up and immobilize the body fluids deposited on the napkin and should preferably have a physical structure contributing to shape retention in use. One or more layers of a fluid distributing material to cause the absorbed fluid to spread laterally, thus making more efficient use of the absorptive capacity, is desirable. An economical and efficient absorbent is found in wood fluff, a comminuted wood pulp compacted to a density of about 1.5 to 5 lbs./ft.³ (0.024 to 0.08 g./cm.³).

A fluid barrier is used to prevent the absorbed fluid from penetrating to the outside or edges of the napkin, which might result in soiled clothing or other embarrassment. The barrier must be fluid-proof under use conditions and must have sufficient integrity to resist rupture due to the normal movements of the wearer. A thin polyethylene film is often used for this purpose. The barrier may be located near the center of the "two-way" napkin (which may be used with either side next to the body) or it may be located near or at one face of the core in the "one-way" napkin. Obviously, the latter arrangement makes more efficient use of the absorbent core and eliminates unnecessary useless bulk.

The outer wrapper holds the components of the assembly in place, prevents disintegration and shedding of the absorbent, provides tabs as a means of attachment to a supporting belt, and is the major load-bearing member of the structure. Since it is worn next to the body it must be soft, conformable, and nonchafing. The outer wrapper must be strong enough to support the strain imposed by motions of the wearer in moving about, stooping, and the like. It must be soft and quiet and not emit papery sounds on bending and, further, it must not shed loose fibers when rubbed against the body while wet or dry, and it must transmit fluids readily. Commercially, sanitary napkin wrappers are often made of nonwoven fabrics in which textile fibers are held together by adhesive binders. Such fabrics have a weight of about 0.5 to 1.0 oz./yd.² (17 to 34 g./m.²).

A flushable sanitary napkin must meet all of the structural requirements already mentioned in order to perform satisfactorily in use. In addition, all of the individual components and the structure as a whole must disintegrate in the flushing water of an ordinary toilet. It is not necessarily sufficient that the individual components be disposable by flushing as the entire combination must be flushable as a unit. The bulky core of a sanitary napkin, if wrapped in sufficient toilet tissue to give mechanical integrity in use, would stop up ordinary toilets because the paper does not disintegrate easily enough, and the bulk of the entire unit is too large. On the other hand, in a flushable napkin the outer load-bearing wrapper and the fluid-impermeable barrier must maintain their strength and integrity in use even when wet with menstrual fluid. This difficult combination of properties plus easy biodegradability is attained for the first time by the novel combination of elements which constitutes the present invention.

The absorbent core of the flushable napkin of this invention is of more or less conventional construction. The main component is wood fluff, which easily disintegrates in water with mild turbulence to form a flushable slurry. The wood pulp, in the form of dry sheet material, may be comminuted in a hammer mill and collected from an air suspension in the form of a loose batt as is well known in the art. The absorbent core may contain a fluid-distributing layer of flushable crepe wadding. Alternatively, a ridged pattern may be embossed in the surfaces of the wood fluff batts, whose ridged faces are then brought into face-to-face contact at the center of the absorbent core to encourage the spread of fluid as described in U.S. Pat. No. 2,952,260. While the use of a fluid-distributing layer of some sort can be used, it is not essential in the flushable product of this invention.

The fluid barrier of the flushable sanitary napkin of this invention is formed from a water-dispersible cellulosic sheet material, preferably crepe wadding or creped tissue, which is treated with a light application of a highly efficient water repellent or other materials described herein. Ordinary waxes or film-forming materials which have been proposed in the past have a stiffening and harshening effect on the barrier sheet and are therefore, sometimes undesirable. Such waxes and film-forming materials, however, are desirable when used at the low loading levels specified herein.

An especially efficient fluid barrier is formed by the application of fluoropolymers, and more particularly, by the use of the class of water repellents containing the fluoroalcohol esters of methacrylate polymers. Compositions suitable for

use are described in U.S. Pat. Nos. 3,068,187 and 3,171,861, Belgian Pat. No. 671,010 and French Pat. No. 1,462,332. It is preferred to use a solution or suspension of the repellent composition in an organic or nonaqueous liquid, since the use of an aqueous liquid may lead to the formation of pinholes or otherwise damage the crepe tissue to which it is applied.

The repellent materials are most conveniently applied by spraying a solution or suspension of the active ingredients onto the sheet material which is to form the fluid barrier. The amount of material applied varies to some extent with the particular repellent formulation chosen and the cellulosic sheet material used as a substrate. Too heavy an application may hinder the dispersibility of the barrier during flushing. Hence, some preliminary testing may be required for each specific set of materials. In general, the amount to be applied will fall within the range of 0.0001 g. to 0.0015 g./in.² (0.0000155 to 0.000232 g./cm.²) and preferably 0.0002 to 0.0005 g./in.² (0.000031 to 0.000077 g./cm.²) for the fluoropolymers and .0005 to .0015 g./in.² (0.000077 to .000232 g./cm.²) for the paraffin waxes.

The repellent may be applied to one or both sides of the barrier sheet, and the barrier may contain one to three or even more layers of cellulosic material, depending on the weight and tensile strength of the material chosen.

The barrier sheet is preferably of approximately the same length as the absorbent core with which it is used. The barrier should be at least wide enough to enclose one face and both edges of the absorbent core as shown in the drawings.

Optionally, the cellulosic sheet material which is to form the barrier may be of a width sufficient to completely wrap around the absorbent core with partial or complete overlap. In any case, the barrier repellent is applied to those areas of the sheet which are to cover the outside face and edges of the core. The repellent may be applied before the sheet is assembled with the core, or may be sprayed on the outside after the sheet has been laid over or wrapped around the core material.

The cellulosic sheet material which makes up the bulk of the repellent barrier is preferably the material known in the trade as crepe wadding. This is a loosely-structured, lightweight, extensible creped product made from wood pulp. It need not have great physical strength as it is supported in use by the external wrapper but the material should disintegrate quickly and easily in water.

A suitable cellulosic barrier substrate will have a machine-direction dry tensile strength in the range of about 0.3 to 0.6 lbs./in. (54 to 107 g./cm.) coupled with an elongation of 40 to 75 percent. These ranges are intended to indicate an area where satisfactory operation may be expected but are not intended to define the operable limits of the invention. There is a large number of cellulosic products on the market which can be adapted to use in barrier construction and which will be apparent to those skilled in the art. For example, a suitable cellulosic barrier substrate may also have a dry tensile strength in the principal direction of 0.4 to 0.8 lbs./in. and an elongation-at-break in the range of 10 to about 75 percent. The flushable nonwoven fabrics which are to be used as the outer wrapper may also be used to receive the repellent composition and form the inner barrier.

The outer wrapper of the flushable napkin of this invention is preferably made by the chemical modification of a nonwoven fabric of cellulose fibers held in place by random fiber entanglement. The chemical modification employed is one which makes the fibers water-sensitive and high-swelling in water.

The water sensitivity of the fibers cooperates with the entanglement-type of fiber-to-fiber fastening in the nonwoven fabric to yield a product which will disperse in water by fiber slippage and fiber breakage. In this way it is possible to provide an economical, lightweight, soft, drapeable fabric with high dry strength which is easily flushable in the ordinary toilet. In use, as the outer wrapper of the sanitary napkin of this invention, the front or outside area of the fabric is kept dry by the repellent barrier and thus retains full strength in use.

The outer wrappers suitable for use in the flushable napkin of this invention include the absorbent nonwoven fabrics described and disclosed in Belgian Pat. No. 706,510, granted May 14, 1968. Accordingly, the disclosure of Belgian Pat. No. 706,510, granted May 14, 1968, is herein incorporated by reference.

Such absorbent nonwoven fabrics have dry strength and integrity in the absence of resinous or fusion bonds comprising from 10 to 100 percent water-sensitive fibers characterized in that:

1. the water-sensitive fibers are less than 3 inches (7.6 cm.) in length, the balance of the fibers consisting of nonwater-sensitive fibers of less than 0.5 inch (1.3 cm.) in length;
2. said fabric has a dry coherence value (C_h dry) of at least 0.2 and a wet coherence value (C_h wet) of less than 0.3 both measured in the absence of resinous or fusion bonds; and
3. with the proviso that the ratio

$$\frac{C_h \text{ dry}}{C_h \text{ wet}} \geq 1.3$$

Preferably the fabric has a weight of 0.3 to 5 oz./yd.² (10 to 170 g./m.²) and most preferably has an average wet strip tensile strength of less than 0.1 lb./in. (18 g./cm.²). In a preferred embodiment, the fabrics have an elongation at break in two directions at right angles to each other of at least 20 percent, the C_h wet is less than 0.15 and the fabric contains at least 40 percent water-sensitive fibers. In another preferred embodiment, the fabric has an average tensile strength in synthetic urine as hereinafter defined of at least 0.05 lb./in. (9 g./cm.²), a C_h dry of at least 0.3, C_h wet not more than 0.15 and a ratio of C_h dry to C_h wet of at least 2. It is also preferred that the water-sensitive fibers have a denier per filament between 0.5 to 15 and a length of between 0.25 to 2 inches (0.6 to 5.1 cm.). The preferred water-sensitive fibers are those of cellulosic esters and ethers whose AOH compound is soluble in water at 20° to an extent of at least 3 percent. Two preferred products consist either (a) essentially of water-sensitive fibers or (b) are layered, the surface layer containing at least 80 percent nonwater-sensitive fibers and the center layer containing at least 70 percent water-sensitive fibers, the overall structure comprising 30 to 90 percent water-sensitive cellulosic fibers.

The coherence value (C_h) is a measurement of the degree to which the fabric comes apart by fiber-to-fiber slippage as opposed to fiber breakage whenever a long strip of the fabric is pulled apart and is thus a measure of interfiber friction from which the fabric derives its strength.

"Nonwoven fabric" means a structure having drape as measured by a bending length of less than 3.0 cm. in two directions at right angles to each other and elongation-at-break of at least 9 percent in at least one direction.

Water-sensitive fibers are those with a wet coefficient of sliding friction of less than 0.9 (preferably less than 0.70) as measured on a nonwoven fabric consisting essentially of the salt free fibers being tested, which fibers also must 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.

A C_h wet of less than about 0.30 characterizes behavior such that entangled fibers will break up into smaller pieces in water after dipping by the relatively mild forces of the turbulent flow in a toilet bowl.

The preferred products of this invention have C_h (wet) values of less than about 0.15. This class of products is flushable, in the form of relatively large fabrics (such as 13 in. × 17 in.) with reduced turbulence of water, without the need of predipping before flushing.

A potential problem remains in the possibility of weakening the suspending tabs by moistening with perspiration, and the necessity of maintaining reasonable fabric integrity in the area wet by the menstrual fluid. For these reasons it is preferred to

convert the cellulosic fibers to polyelectrolytes by attaching ionizing groups to the cellulose chain. Polyelectrolytes are generally soluble or highly swollen in water but have been found to be highly sensitive to the deswelling effect of dissolved ionizing salts. Body fluids such as urine, blood, menstrual fluid, and perspiration all contain dissolved ionizing salts in sufficient concentration to permit the water-dispersible cover fabric to maintain adequate strength and integrity in use when wet with such body fluids. Suitable polyelectrolyte cellulose derivatives made from rayon generally have a degree of substitution of from 0.2 to 0.4 and preferably from about 0.22 to about 0.30 groups per glucose unit, on the average.

The nonwoven fabrics used for chemical modification are preferably made from regenerated cellulose fibers such as rayon rather than natural fibers like cotton. The regenerated cellulose fibers are significantly more reactive to the modifying reagents used, and are adaptable to high-speed, continuous operation in the chemical conversion.

Any regenerated cellulose fibers can be used, but preferably, they are viscose rayon fibers of 1.5 to 2 denier per filament. These are preferably cut into staple lengths of 0.25 inch to 2 inches (0.635 to 5.08 cm.), with fibers from 0.75 inch to 1.56 inches (1.9 to 3.97 cm.), being especially suitable. The fibers are preferably converted into a nonwoven web of random fiber orientation by an air deposition process but the wet paper machine methods may be used for the shorter fibers. Air-deposition may be accomplished by means of a Rando-Webber machine made by the Curlator Corp. of Rochester, N.Y. Nonwoven fabrics for use in the products of this invention may also be prepared from more oriented webs made on a conventional carding machine. Such fabrics are used with the machine (oriented) direction of the fabric aligned with the long direction of the final napkin.

The thin web or batt of rayon fibers is preferably given integrity and tenacity by a process of entanglement over a screen or similar support through the use of fine columnar streams of water delivered at relatively high pressures. The process and apparatus involved are described in detail in Belgian Pat. Nos. 673,199 and 706,510. The product obtained, when dried, is suitable for immediate chemical conversion to the flushable form.

The preferred chemical derivatives of cellulose for use as flushable fabrics include the sodium salts of the partial carboxymethyl ether, the partial monoester of phosphoric acid, and the partial monoester of succinic acid. Other less preferred derivatives include the hydroxyethyl ether and the sodium salts of: the carboxyethyl ether, the monoester of sulfuric acid, the monoester of phthalic acid, and the monoesters of other dibasic organic acids. All of these derivatives can be made by methods known in the art and the three preferred derivatives can easily be made by direct, rapid, continuous-process, high-temperature methods. These methods are illustrated in the examples.

As illustrated in the examples, the water-sensitive nature of the fabrics of this invention requires special treatment for the fabrics when wet with water. High-swelling fabrics containing substituents which are salts of weak acids may be converted to a low-swelling free-acid form by treatment with a dilute solution of a strong acid like H₂SO₄. In this free-acid form, the fabrics may be washed free of impurities in plain water quickly and without damage. They may then be converted to the more useful high-swelling form by treatment with a slightly alkaline buffer such as disodium phosphate to reform 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 saltfree 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 form of the fabric with an aqueous alcohol solution of a buffer or dilute alkali, followed by an alcohol or acetone wash and drying with heat.

The nonwoven fabric used for chemical modification may contain a small amount of less reactive or noncellulosic fibers. For example, cotton, nylon, 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 one-half inch (1.27 cm.) or less in length. It should be noted that the addition of such unreactive fibers to the fabric tends to reduce the biodegradability of the product which 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 percent relative humidity for at least 24 hours before testing under these conditions.

Tensile strengths and elongations are measured on 1.0 × 2 inch (2.54 × 5.08 cm.) samples at an elongation of 50 percent 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.² (g./m.²) 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. MgSO₄, 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 × 3-inch (7.62 × 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 liters/minute for a 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 about 7 percent.

The flushability of a napkin or piece of fabric is determined by dropping it into the 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-inch (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 cover fabric samples termed "Flushable" by the above test usually have a dispersibility of at least about 20 percent in 2 minutes, in the small-scale dispersion test. Preferred fabrics have a dispersibility of at least 40 percent.

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 a 1 × 6-inch sample on a Drape-Flex Stiffness Tester made by Fabric Development Tests, Brooklyn 32, N.Y. The procedure followed in making this test is given in ASTM method D1388-55T.

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 (lg. in 3,000 g. liquid). The sample is removed from the liquid and spread to cover a 5 × 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.². Pressure is applied for 5 minutes after which the sample is removed and weighed, giving the net weight. The sample is then dried to constant weight using a Nobel and Woods sheet dryer at 100°C. Absorbency equals the water absorbed which is the wet weight minus the dry weight divided by the dry weight of the sample. 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.

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 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 a 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 less than about 0.3 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 4.1 cm. wide strips. The strips are then cut to about 5 and 2.5 cm. long adjacent samples. The longer samples is broken using 3.8 cm. initial distance between the rubber-coated jaws of the Instron Tester. The shorter sample is broken using zero initial distance between the jaws. 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 around 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 12.7 mm. per minute.

Salt-free fabrics for use in coherence value determinations are prepared by extracting with a solution made from 60 percent methanol and 40 water by volume. About 100 g. of the fabric (divided into 4 portions of 25 g. each) 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 three washes). Three additional

treatments of the 4 portions simultaneously 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 is avoided.

Rayon fibers of 1.56 inches (3.97 cm.) length and 1.5 denier per filament are made into a batt of randomly oriented fibers by an air deposition process using a Rando-Webber machine. The batt has a weight of about 0.8 oz./yd.² (27 g./m.²) and it is made into a nonwoven fabric by the process of Belgian Pat. No. 673,199. In this process, the batt of random fibers is supported on a screen having 24 wires per inch (2.54 cm.) in each direction with 16 percent open area and is passed at 5 yards (4.57 meters) per minute under 5 rows of oscillating jets supplied with water at 200, 300, 400, 500 and 600 lbs./in.² (14.1, 21.1, 28.1, 35.2, and 42.2 kg./cm.²) respectively. Each row of jets contains 40 holes/inch (40 holes/2.54 cm.) which are 0.005 inch (0.127 mm.) in diameter. The columnar streams of water deliver energy at the rate of 0.96 horsepower-hour per pound (0.96 horsepower-hour per 0.454 kg.) of fibers. The dried nonwoven fabric has a weight of about 0.8 oz./yd.² (27 g./m.²) and a dry tensile strength of 6.17 lbs./in. (1.1 kg./cm.).

The dry fabric is thoroughly wet out in an aqueous reagent bath containing 2.1 percent of sodium hydroxide and 15.8 percent of sodium monochloroacetate. Excess reagent is pressed out of the fabric with squeeze rolls so that the fibers retain 1.1 times their own weight of liquid. The moist fibers are immediately dried and caused to react by passing them through a circulating-air oven 0.46 meters long at a rate of 1.53 meters per minute while at a temperature of approximately 140°C. The carboxymethylated fabric is then passed under a spray of 5 percent sulfuric acid to convert it to the low-swelling free acid form after which it is thoroughly washed with water. It is then converted to the high-swelling sodium salt form in a deswelling neutralizing bath containing 8.5 percent sodium sulfate and 2 percent disodium phosphate. The fabric is passed between squeeze rolls to remove as much salt solution as possible, cut into convenient lengths, and dried at 40°C. in a laundry drier containing several baseballs 9.7 cm. in diameter to soften the fabric by mechanical working and to remove excess salt. The dried fabric is cut into 7 × 19-inch (17.8 × 48.3 cm.) pieces for use as the wrapper elements of sanitary napkins. The fabric has the following properties:

Fabric weight—1.5 oz./yd.² (51 g./m.²).
Tensile strength, dry (MD)—3.34 lb./in. (597 g./cm.).
Tensile strength, wet (MD) tap water—0.07 lb./in. (12.5 g./cm.).
Tensile strength, wet synthetic urine—0.23 lb./in. (41.1 g./cm.).
Absorbency—About 10 to 15 g./g.
Degree of Substitution—About 0.25.

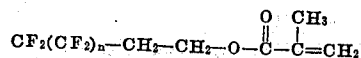
In the construction of a sanitary napkin, a wood fluff core having embossed layers to encourage the distribution of absorbed fluid, with a bulk density of about 3 lbs./ft.³ (0.048 g./cc.) is used. A batt of the wood fluff about 0.75 inch (1.9 cm.) thick, 2.5 inches (6.35 cm.) wide, and 7.6 inches (19.3 cm.) long is wrapped in a crepe tissue wrapper about 7.6 inches (19.3 cm.) long and 7.75 inches (19.7 cm.) wide so that the overlap forms a double thickness on part of the back side. These two thicknesses are glued together with a light continuous line of poly(vinyl alcohol) adhesive. The crepe tissue used is found to be too weak to test when wet with water. The physical properties measured on 1 × 1-inch [2.54 × 2.54 cm.] samples of the dry sheet in the tensile tests are as follows:

Sheet weight—0.57 oz./yd.² (19.3 g./m.²).
Tensile Strength—(MD)—0.41 lb./in. (73.2 g./cm.).
Tensile Strength—(XD)—0.15 lb./in. (26.8 g./cm.).
Elongation—(MD)—58%.
Elongation—(XD)—14%.
Work-to-break—(MD)—0.105 in. lbs./in.² (18.8 g./cm.²).

Work-to-break—(XD)—0.014 in. lbs./in.² (1.5 g./cm.²).
Bending Length—(MD)—1.48 cm.
Density—1.155 g./cm.³
Thickness—0.0049 in. (0.124 mm.).

The crepe tissue on the back side and edges of the wrapped absorbent core are sprayed with an organosol of a fluoropolymer water repellent so that 0.0064 g. of solid remain after the solvent has evaporated. The corners of the assembly are cut off to give rounded ends as shown in the drawing and the core assembly is then wrapped in a piece of the water-sensitive outer wrapper fabric. The edges of the outer wrapper on the back of the napkin are secured in place with small spots of an adhesive polyvinyl alcohol at intervals along the overlap.

The repellent organosol used in constructing the repellent barrier is made in accordance with the teachings of Belgian Pat. No. 671,010. The fluoromonomer is a mixture of methacrylate species having the general formula



wherein the species n being 5, 7 and 9 are in relative weight proportions of 3:2:1 respectively and constitute at least 90 percent of the monomer mixture; higher species constitute up to 10 percent.

In preparing the fluoropolymer the following ingredients are used ("Buton" 100 is a commercially available product of Enjay Company of 60 W. 49th Street, New York, N.Y.).

Part A	
35 ("Buton" 100) Butadiene/styrene copolymer	Parts by wt. 31.50
Fluoromonomer	63.00
Wetted benzoyl peroxide—67% in water	3.84
Toluene	139.44
Part B	
40 Azonitrile catalyst	4.04
Toluene	6.77
Fluoromonomer	115.50
Part C	
45 Methyl chloroform	685.91
	1,050.00

The ingredients of Part A are charged in order given with agitation into a reactor and heated to 95–97°C. and held at this temperature for 2 hours and 15 minutes.

The components of Part B are premixed and added to Part A reaction product. Combined Parts A and B are reheated to 95–97°C., held at this temperature for 2 hours and 15 minutes and then cooled to 65°C.

Part C is added to Parts A-B over a 10-minute period, maintaining the temperature at about 65°C. The composition is agitated for 20 minutes at 65°C. and then cooled to room temperature and filtered. The resulting mixture, containing approximately 20 percent nonvolatile material, is diluted to about 0.5 percent concentration with methyl chloroform before use.

In an experimental wear test, all of the napkins perform in a satisfactory manner, comparable to commercial nonflushable sanitary napkins. The test napkins are disposed of in the standard flushability test without difficulty.

EXAMPLE II

Rayon fibers of 0.75 inch (1.9 cm.) length and 1.5 denier per filament are made into a batt having a weight of 1.2 oz./yd.² (40.7 g./m.²), and entangled into a nonwoven fabric by the method of example I, using 0.96 horsepower-hours per

pound of fiber, the fiber being supported on a screen having 20 wires per inch (2.54 cm.) in both directions and 36 percent open area. The dried nonwoven fabric has a tensile strength of 6.6 lb./in. (1,180 g./cm.) in the machine direction and 4.6 lbs./in. (821 g./cm.) in the cross direction.

The dry fabric is cut into pieces about 20 inches (50.8 cm.) long and 10 inches (25.4 cm.) wide. These are soaked for 2 minutes in a 17 percent solution of succinamic acid at about 50°C. The fabric is removed and pressed between blotters until it retains three times its weight of succinamic acid solution. The fabric is then dried and a chemical reaction is effected by placing the sheet in a circulating hot-air oven for 4 min. during which time the air temperature is raised from 135°C. to 163°C. The cellulose is thus converted to the free acid form of the partial hemisuccinate ester at a degree of substitution of 0.3 acid succinate groups per glucose unit. It is converted to the high-swelling sodium salt by soaking it for 5 minutes in a buffered salt solution containing 3 percent disodium phosphate and 17 percent sodium sulfate and maintained at a pH of 8.5 to 9.0 by the addition of small amounts of sodium hydroxide solution as needed. The fabric is then squeezed as free of salt solution as possible, air-dried, and mechanically worked by hand to remove excess salt. The final fabric has the following properties:

Dispersibility (tap water)—46%.
Absorbency (dist. water)—9.5 g./g.
Tensile Strength, dry—greater than 5 lbs./in. (above 893 g./cm.).
Tensile Strength, wet (tap water)—0.019 lb./in. (3.4 g./cm.).
Tensile Strength, wet (syn. urine)—0.218 lb./in. (39 g./cm.).
Elongation, wet (tap water)—13.4%.
Elongation, wet (syn. urine)—33.6%.

The fabric is used in the construction of sanitary napkins as in example I. In a use test, the napkins perform fully as well as the usual commercial sanitary napkin. All the test napkins flush easily in the experimental test toilet facility.

EXAMPLE III

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.² web. The rayon fibers are entangled to give strength and integrity to the web using the method of Belgian Pat. No. 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 percent open area, and passed at 5 yards (4.57 meters) per minute under 4 rows of oscillating jets supplied with 65°C. water at 200, 500, 700, and 800 lbs./in.² (14.1, 35.2, 49.2, and 56.2 kg./cm.²) 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 (0.454 kg.) of fibers. The dried nonwoven fabric has a weight of 1.10 oz./yd.² (37.3 g./m.²) and a tensile strength of 5.08 lbs./in. (907 g./cm.) in the machine direction with a tenacity of 2.97 lbs./in. (530 g./cm.) in the cross direction.

The nonwoven fabric is uniformly padded with 2.02 times its weight of a reagent solution prepared by dissolving 100 parts of shotted commercial urea in 56.5 parts of deionized water at 185°F. (85°C.) and adding 43.5 parts of commercial 85 percent phosphoric acid to the solution. The web fabric is fastened on a tenter frame and passed through a circulating air oven with exposure to balanced-flow low-velocity air at 315°F. (157°C.) for 2 minutes. The resulting cellulose phosphate ester fabric is cut into pieces which are washed in 5 percent sulfuric acid, squeezed free of excess liquid, and neutralized to the high-swelling form in a buffered salt solution containing 2

percent disodium phosphate and 8 percent 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 as in example I. The final product has the following properties:

Degree of substitution—0.23.

Dispersibility—20.7%.

Absorbency—7.35 g./g.

10 Fabric weight—1.33 oz./yd.² (45 g./cm.²).

Tensile strength (MD), dry—3.64 lb./in. (650 g./cm.).

Tensile strength (MD), wet tap water—0.034 lb./in.

(6.1 g./cm.).

15 Tensile strength (MD), wet syn. urine—0.375 lb./in. (67 g./cm.).

The fabric is used as the wrapper fabric in the construction of sanitary napkins as in example I. These napkins easily pass the standard flushability test.

EXAMPLE IV

The napkins of this example are constructed solely to evaluate the barrier structure and are not flushable as an assembled unit. They are made using a wood pulp core as in example I. The nonflushable outer cover fabric is a resin-bonded nonwoven scrim removed from a commercial napkin. The repellent barrier is similar to that in example I, except that the portion of the crepe tissue which is to cover the back and sides of the core is sprayed with repellent on both sides before it is wrapped around the wood fluff core. In this case, the repellent contains as active ingredient, a segmented copolymer of about 76 percent of 2-(N-ethyl, heptadecafluoro-1-octanesulfonamide)-ethyl methacrylate and about 24 percent methyl methacrylate, as described in U.S. Pat. No. 3,068,187. A total of about 0.034 g. of repellent per gram of substrate is applied to the barrier sheet.

The napkins are subjected to a static loading test designed to be more severe than any actual conditions to be encountered in use. The napkin is placed, with the outside down, on a resilient felt flock protected with a thin polyethylene film. A 9-pound (4.1 kg.) lead block is placed on top of the napkin, subjecting it to a pressure of about 0.5 lb./in.² (35 g./cm.²). Through a 1-inch (2.54 cm.) hole in the center of the lead block, oxalated bovine blood is introduced into the napkin at the rate of 10 cc./min. After 20 cc. has been added in 2 minutes, the bottom is inspected, and the test is resumed if no stain shows. After 30 ml. of blood has been added, the lead block is shifted to apply pressure uniformly to the wet area for 10 seconds. If the barrier does not pass a stain under these conditions it is considered more than adequate for general use. The napkins of this example do not show any stain on the back side in this test. The repellent barriers easily disintegrate in mildly circulating water, indicating easy flushability.

EXAMPLE V

A nonwoven entangled rayon fabric is made by a process closely similar to that in example I using a supporting screen having 20 wires per inch (2.54 cm.) in both directions. The dried fabric has a weight of 0.95 oz./yd.² (32.2 g./m.²) and the tensile strength is 3.2 lbs./in. (572 g./cm.) with 31 percent elongation in the machine direction while the cross direction exhibits a tensile strength of 2.24 lbs./in. (400 g./cm.) with 72 percent elongation.

A reagent solution is prepared containing 4.50 percent sodium hydroxide, 20 percent sodium monochloroacetate and 0.75 percent "Kelzan", a polysaccharide thickener made by the Kelco Company of Chicago, Illinois. This solution is applied uniformly to the nonwoven fabric by means of a pad roll etched with 2.5 mil (0.0635 mm.) lines so that the fabric picks up 1.87 g. reagent per gram of air-dry cellulose. The fabric is passed through a circulating hot air oven at 136°—144°C. at 6 feet (1.83 meters) per minute, for a reaction time of 15

seconds. The fabric is next passed under a shower of 5 percent sulfuric acid, containing 15 percent sodium sulfate followed by a shower of solution containing 17 percent sodium sulfate and 4 percent disodium phosphate. In order to ensure complete removal of reagents and byproducts, the treatments with dilute acid and with buffered salt solution are repeated. The final fabric is squeezed and tumble-dried as in example I. The dried fabric has the following properties:

Degree of substitution—0.32.

Absorbency—10.2 g./g.

Dispersibility—59.1%.

Fabric weight—about 1.5 oz./yd.² (51 g./m.²).

Tensile strength, dry (MD)—4.56 lbs./in. (815 g./cm.).

Tensile strength, dry (XD)—1.55 lbs./in. (277 g./cm.).

Tensile strength, wet (MD) tap water—0.044 lbs./in. (7.9 g./cm.).

Tensile strength, wet (XD) tap water—0.002 lbs./in. (0.4 g./cm.).

Tensile strength, wet (MD) syn. urine—0.13 lbs./in. (23.2 g./cm.).

Tensile strength, wet (XD) syn. urine—0.10 lbs./in. (17.9 g./cm.).

The fabric is cut into pieces 7 inches (17.8 cm.) wide and 19 inches (48.2 cm.) long which are used as wrapper in the construction of flushable sanitary napkins. The absorbent core of the napkins is wood fluff wrapped in crepe tissue as in example I. In this case, however, a separate barrier is constructed of crepe tissue having the following properties:

Sheet weight—0.6 oz./yd.² (20.4 g./m.²).

Thickness—0.0074 in. (0.188 mm.).

Bursting strength—6.0 p.s.i. (422 g./cm.²).

Bending length—2.8 cm. (MD) and 1.1 cm. (XD).

Tensile strength, MD—0.25 lb./in. (44.7 g./cm.).

Tensile strength, XD—0.14 lb./in. (25 g./cm.).

Elongation, MD—70.6%.

Elongation, XD—12%.

The crepe tissue is treated on both sides with the fluoropolymer repellent of example IV, so that the sheets retain about 5 percent of their original weight of repellent material. The sheets are about 7.5 inches (19 cm.) long and are assembled into a barrier three sheets thick with a 5-sheet thick overlap at the center. The barrier is 11 inches (28 cm.) long, and 3.5 inches (8.9 cm.) wide so as to cover the back and sides of the wrapped absorbent core. These assemblies are enclosed in the flushable nonwoven fabric wrappers, after two notches about 1.25 inch (3.18 cm.) wide and 4 inches (10.5 cm.) long are cut out of each end of the wrapper fabric to reduce the bulk of the fastening tab. In experimental test usage, none of the barriers permit the passage of menstrual fluid and the napkins are easily flushable in the standard flushability test.

The fluid barriers of the napkins of this invention can optionally be made by using the outer wrapper comprised of modified cellulosic fibers as the substrate and coating the inside portion of the wrapper that is to be away from the user directly with the fluoropolymer, thereby converting the outer wrapper into an effective barrier. The barrier, when so constructed restricts the napkin to use as a "one-way" type of napkin.

EXAMPLE VI

In the construction of a flushable sanitary napkin, an absorbent core fabricated from crepe tissue and wood fluff batts is used. The cores are formed by laying up three layers of wood fluff, each layer being contained between single plies of crepe tissue, and stamping out the individual cores, with a suitable die cutter, of about 7.5 inch long × 2.5 inch wide × 0.875 inch thick, ends rounded. A suitable wood fluff for these absorbent cores is prepared by processing pulp sheets of Weyerhaeuser

Soft Alpha M through a hammermill followed by further processing through a Rando-Webber (or similar air laydown) machine to give batts of material of 8 oz./yd.² basis weight and a thickness of three-eighths inch. In the absorbent cores, the crepe tissue carriers of the wood fluff are aligned with the creping direction of the tissue running parallel to the long dimension of the core. Suitable tissues, of the grade known as white facial toilet tissue, have the following specifications:

10

Basis weight—0.47–0.57 oz./yd.²

Tensile strength (MD)—0.41–0.71 lb./in.

Tensile strength (XD)—0.12–0.30 lb./in.

Elongation (MD)—10–58%.

Elongation (XD)—5–14%.

15

Work-to-break (MD)—0.09–0.105 in. lbs./in.².

Work-to-break (XD)—0.007–0.014 in. lbs./in.².

Beading length (MD)—1.48–2.2 cm.

Thickness—About 0.005 in.

20

The absorbent cores are wrapped in a single ply crepe tissue wrapper (of the preceding specifications) about 7.25 inches long and 14 inches wide. The tissue wrapper is rolled over core in such a way that a two ply wrapper results. The second ply of tissue is glued to the first ply, on the bottomside of the core, with a light continuous line of either an aqueous poly(vinyl alcohol) adhesive or a poly(vinyl acetate) emulsion type of adhesive.

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The crepe tissue on the back (bottom) side and edges of the wrapped absorbent core is sprayed with a solution of a household type paraffin wax water repellent (freezing point 122–124F.) in 1,1,1-trichloroethane (2 g. of wax in 98 ml. of solvent) so that 0.07 to 0.25 mg./cm.² of wax remains, on the treated area, after the solvent evaporates.

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This core assembly is then wrapped in a 7 inch wide and 19 inch long piece of cellulose phosphate ester fabric prepared as described below.

35

Crimped dull 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.² (34 g./m.²) web. The web of rayon fibers is supported on a screen containing 24 wires per inch (2.54 cm.) in both directions, with 16 percent open area, and passed at 6 yards (5.5 meters) per minute under 4 rows of jets supplied with 60°C. water at 300, 600 and 700 lbs./in.² (21.2, 42.4 and 49.2 kg./cm.²) 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 dried nonwoven fabric has a weight of 1.05 oz./yd.² (35.6 g./m.²). The tensile strength is 4.1 lbs./in. (476 g./cm.) in the cross direction.

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The nonwoven fabric is uniformly padded with 1.95 times

its weight of a reagent solution prepared by dissolving 100

parts of commercial urea pellets in 62.4 parts of tap water,

heated to maintain about 45°C., and adding 37.6 parts of commercial 85 percent phosphoric acid to the solution. The ratio

of phosphoric acid to urea in the reagent solution is 1/3.12 and the fabric retains 0.31 times its weight of H₃PO₄. The wet

fabric is supported on a meshed wire conveyor and passed

through a circulating air oven with exposure to through-flow,

low velocity air at 376°F. (191°C.) for 0.25 minutes. The

heated air is recirculated and fresh air addition is minimized to

maintain a concentration of urea vapor in the air. The resulting

cellulose phosphate ester fabric supported on a screen

conveyor is passed at 12 yd./min. (11.0 m./min.) under three

sequential, uniformly distributed countercurrent flows of 0.6

percent H₂SO₄ issuing at 0.85, .50 and 0.35 gal./min./inch

(1.26, 0.74 and 0.52 l./min./cm.) of width 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 two sequential, uniformly distributed

flows of 1 percent Na₂SO₄/2 percent Na₂HPO₄/3 percent sodium hexametaphosphate containing 0.2 percent Du Pont Paper

white SP solution and 0.05 percent Merpol* ON adjusted to

75

pH 6.0 both issuing at 0.45 lb./min./inch (0.08 kg./min./cm.) of width and into the nip of a squeeze roll set at 30 lb./linear inch (5.4 kg./cm.). The fabric is dried at 110°C. in a tenter dryer and mechanically softened by passing it through sets of meshing rolls. The resulting fabric has the following properties:

Degree of substitution—0.26 (est.).
Fabric weight—1.5 oz./yd.² (51.5 g./m.²).
Tensile strength (MD), dry—3.5 lb./in. (630 g./cm.).
Tensile strength (XD), dry—2.0 lb./in. (366 g./cm.).
Elongation, dry (MD)—13.4%.
Elongation, dry (XD)—62.2%.
Tensile strength (MD), 1% NaCl—.24 lb./in. (g./cm.).
Water—.02 (est.).

The edges of the outer wrapper described above are secured in place with small spots of an adhesive poly(vinyl alcohol) or poly(vinyl acetate) at intervals along the overlap.

In experimental wear tests, the napkins perform in a satisfactory manner, comparable to commercial nonflushable sanitary napkins. The test napkins are disposed of in the standard flushability test without difficulty.

Other solvents may be used for the spray application of the paraffin wax water repellent barrier. Suitable solvents are V.M. & P. Naptha, paraffin distillate, and ethylene dichloride, among others. The paraffin wax may also be used to treat the tissue substrate through the use of hot melt printing techniques. For example, a hand gravure roller containing 180 pyramidal shaped cells per inch has been used in the laboratory to apply the paraffin wax, at a temperature of 190°—210°F., to a single ply of the various tissues at a loading of about 0.62 to 1.09 mg./cm.² per pass of the roller. At this loading, the tissue has a slight waxy feel but is not stiffened noticeably. Although this printed tissue is not quite as water repellent as the 0.07 to 0.25 mg./cm.² waxed tissue obtained by spray application (as judged by beading of droplets of water on the surfaces), it may be rendered equally repellent by a post heat treatment at 81° to 150°C. In none of these application methods is a continuous film of wax applied, as judged visually under the microscope. When a loading of about of 20 to 27 mg./in.² of the wax is applied to the tissue by the printing technique (repeated passes), the tissue becomes quite waxy and is stiffened somewhat.

Other paraffin waxes are suitable as water repellent barriers on the tissue. Blends of paraffin wax and microcrystalline waxes and/or ethylene/vinyl acetate-type copolymers with the wax are also operable. In addition, other barrier candidates which can pass a laboratory screening test similar to that described in example IV when sprayed onto the crepe tissue wrapper, include: "Zelan" S (durable water repellent) (0.54 mg./in.²), "Zepel" S-1 (fabric fluoridizer) (1.14 mg./in.²), TLF-2005 (a polyhydroxy titanium stearate) (0.78 mg./in.²), "Zonyl" S-13 (fluorochemical surfactant) (about 0.5—1.0 mg./in.²), poly(acrylic acid), (0.2 mg./in.²), and poly(vinyl alcohol) (0.15 mg./in.²).

EXAMPLE VII

Example VI is repeated except that an alternate core construction is used in which one layer of wood fluff is prepared from Weyerhaeuser soft Alpha M pulp. The core is contained between the same single plies of tissues and cut to the same dimensions described in example VI. The wood fluff batt has a basis weight of 21 oz./yd.² and a thickness of about 1 inch. In experimental wear tests, the napkins perform in a satisfactory manner and are disposed of in the standard flushability test without difficulty.

The flushable fabrics of examples I, II, III, V, VI and VII have a dry coherence value (C_h dry) of at least 0.2 and a wet coherence value (C_h wet) of less than 0.3, both measured in the absence of resinous or fusion bonds. In addition, the fabrics have a wet coefficient of sliding friction of less than 0.90.

The following procedure is used to determine the water-sensitive fiber frictional characteristics of the flushable fabrics used in this invention.

WATER-SENSITIVE FIBER FRICTIONAL CHARACTERISTICS

The coefficient of sliding friction, δ (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 inches $\times$ 6 inches (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 inch $\times$ 3 inches (2.5 $\times$ 7.6 cm.) is wrapped around a sled so that the bottom, front and back faces of the sled have one thickness of cloth and the top face has one to two thicknesses. A 5 gram brass weight is placed on top of the fabric. The sled consists of a 1 $\times$ 1 $\times$ 0.063 inches (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 inches (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 .

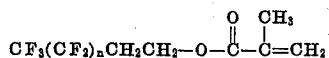
The test is made on a nonwoven fabric consisting of 100 percent of the water-sensitive fibers. Fabric pattern or structure variation has been found to have little effect on the sliding friction with essentially planar fabrics. We claim:

1. A flushable sanitary napkin comprising in combination an elongated absorbent core of loosely felted fibrated wood pulp, said core having a density in the range of 1.5 to 5 lbs./ft.³; a fluid barrier for retaining body fluids within the core; a wrapper encasing the core and fluid barrier for maintaining said napkin in a unitary assembly during use, the wrapper being a nonwoven fabric of randomly entangled, chemically-modified cellulosic fibers, of 1.5 to 3 dpf and a length within the range of 0.25 to 2.0 inches, selected from the group consisting of sodium carboxymethyl cellulose of 0.2 to 0.4 degree of substitution, the sodium salt of cellulose acid succinate of 0.2 to 0.4 degree of substitution, and the sodium salt of monocellulose dihydrogen phosphate of 0.15 to 0.35 degree of substitution; said wrapper being further characterized as a soft, water-dispersible, nonwoven fabric of 0.5 to about 2.0 oz./yd.² fabric weight with a bending length of less than 3 cm., having a tensile strength in synthetic urine within the range of about 0.1 to 0.5 lbs./in., and having a tap-water wet tensile strength within the range of 0 to about 0.09 lbs./in., the wrapper corresponding to a fabric weight within the range of 0.5 to about 1.0 oz./yd.² having a dry-strip tensile strength within the range of 1 to 5 lbs./in. in the principal fabric directions and the wrapper of a fabric weight within the range of about 1.0 to 2.0 oz./yd.² having a dry-strip tensile strength within the range of 3 to about 10 lbs./in.

2. The sanitary napkin of claim 1 wherein the fluid barrier is a cellulosic substrate coated with an organosol of a fluoropolymer to yield about 0.0001 to 0.0005 gram of fluoropolymer per square inch of barrier surface.

3. The sanitary napkin of claim 2 wherein the fluoropolymer is the polymerization product of:

- a. a fluoromonomer comprising about 85 percent by weight of the reaction mixture consisting essentially of a mixture of



wherein the compounds corresponding to n of 5, 7 and 9 are in the relative weight proportions of about 3:2:1 respectively and constitute at least 90 percent of the monomer mixture, the remainder of the monomer mixture being the compounds corresponding to n values greater than 10; and

- b. a styrene/butadiene copolymer which comprises about 15 percent of the reaction mixture.

4. The sanitary napkin of claim 2 wherein the fluoropolymer coating consists essentially of the segmented copolymer of:

- a. about 76 percent of 2-(N-ethyl, heptadecafluoro-1-octanesulfonamido)-ethyl methacrylate; and

- b. about 24 percent methyl methacrylate.

5. The sanitary napkin of claim 2 wherein the barrier substrate comprises a cellulosic crepe sheet having a dry tensile strength in the principal direction of 0.4 to 0.8 lbs./in. and an elongation at break in the range of 10 percent to about 75 percent at a basis weight of about 0.4 to 0.7 oz./yd.², the substrate having a bending length of 1 to 2.5 centimeters.

6. The sanitary napkin of claim 2 wherein the barrier substrate is the outer wrapper of said napkin.

7. The sanitary napkin of claim 1 wherein the fluid barrier is a cellulosic substrate coated with compounds selected from the group consisting of a paraffin wax, blends of paraffin wax and microcrystalline wax and blends of paraffin wax and an ethylene-vinyl acetate copolymer to yield about 0.0005 to 0.0015 gram of wax per square inch of barrier surface.

8. The sanitary napkin of claim 7 wherein the barrier substrate comprises a cellulosic crepe sheet having a dry tensile

strength in the principal direction of 0.4 to 0.8 lbs./in. and an elongation at break in the range of 10 percent to about 75 percent at a basis weight of about 0.4 to 0.7 oz./yd.², the substrate having a bending length of 1 to 2.5 centimeters.

9. The sanitary napkin of claim 7 wherein the barrier substrate is the outer wrapper of said napkin.

10. A flushable sanitary napkin comprising in combination an elongated absorbent core of loosely felted fibrated wood pulp, said core having a density in the range of 1.5 to 5 lbs./ft.³; a fluid barrier for retaining body fluids within the core, and a wrapper encasing the core and fluid barrier for maintaining said napkin in a unitary assembly during use; said wrapper being an absorbent nonwoven fabric weighing 0.3—5 oz./yd.², having dry strength and integrity in the absence of resinous or fusion bonds and comprising from 10 to 100 percent water-sensitive fibers characterized in that:

1. the water-sensitive fibers are less than three inches (7.6 cm.) in length, the balance of the fibers consisting of non-water-sensitive fibers of less than 0.5 inch (1.3 cm.) in length;

2. said fabric has a dry coherence value (C_h dry) of at least 0.2 and a wet coherence value (C_h wet) of less than 0.3 both measured in the absence of resinous or fusion bonds,

3. with the proviso that the ratio

$$\left(\frac{C_h \text{ dry}}{C_h \text{ wet}}\right) \geq 1.3$$

11. The sanitary napkin of claim 10 in which the nonwoven fabric comprising the wrapper has an average wet strip tensile strength of less than 0.1 lb./in.

12. The sanitary napkin of claim 11 in which the nonwoven fabric comprising the wrapper has an elongation at break in two directions at right angles to each other of at least 20 percent.

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