



Europäisches Patentamt  
European Patent Office  
Office européen des brevets



⑪ Publication number:

**0 389 612 B1**

⑫

## EUROPEAN PATENT SPECIFICATION

⑯ Date of publication of patent specification: **22.06.94** ⑮ Int. Cl. 5: **D21H 13/10, D21H 11/00**

⑯ Application number: **89911338.5**

⑯ Date of filing: **29.09.89**

⑯ International application number:  
**PCT/US89/04292**

⑯ International publication number:  
**WO 90/04066 (19.04.90 90/09)**

### ⑯ HYDRAULICALLY ENTANGLED WET LAID BASE SHEETS FOR WIPERS.

⑯ Priority: **05.10.88 US 253805**

⑯ Date of publication of application:  
**03.10.90 Bulletin 90/40**

⑯ Publication of the grant of the patent:  
**22.06.94 Bulletin 94/25**

⑯ Designated Contracting States:  
**AT BE CH DE FR GB IT LI LU NL SE**

⑯ References cited:  
**EP-A- 0 108 621**  
**EP-A- 0 308 320**  
**US-A- 3 493 462**

**Database WPIL, no. 86-192792 Derwent Publications Ltd, London, GB**

⑯ Proprietor: **KIMBERLY-CLARK CORPORATION**  
**401 North Lake Street**  
**Neenah, Wisconsin 54956-0349(US)**

⑯ Inventor: **WATTS, Hugo P.**  
**6 Orwell Close**  
**Larkfield, Kent ME20 6UP(GB)**  
Inventor: **WATKINS, Sharon L.**  
**2300 Riverwood Lane,**  
**Apartment E**  
**Roswell, GA 30075(US)**  
Inventor: **EVERHART, Cherie H.**  
**100 Shadow Springs Drive**  
**Alpharetta, GA 30201(US)**  
Inventor: **VANDER WIELEN, Michael J.**  
**360 Roswell Farms Road**  
**Roswell, GA 30075(US)**

⑯ Representative: **Patentanwälte Grünecker,**  
**Kinkeldey, Stockmair & Partner**  
**Maximilianstrasse 58**  
**D-80538 München (DE)**

**EP 0 389 612 B1**

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid (Art. 99(1) European patent convention).

**Description**

The present invention relates to a hydroentangled coherent fibrous material, which may be used as wipers for industrial and other applications.

5 Nonwoven materials such as, for example, meltblown or spunbonded polypropylene may be used as wipers. In certain applications such as automobile finishing the wiper is usually moistened with one or more volatile or semi-volatile solvents such as, for example, isopropyl alcohol/water, n-heptane, naphtha, and C<sub>5</sub> to C<sub>7</sub> aliphatic hydrocarbons in order to remove grease, fingerprints and/or smudges from the automobile finish before painting or priming. Some solvents and/or other chemicals cause some components such as, 10 for example, low molecular weight polyolefins to leach out onto the wiped surface rendering that surface unsuitable for painting. Many nonwoven materials are hydrophobic and require treatment with one or more surfactants to become wettable. The surfactant may also be transferred to the wiped surface rendering that surface unsuitable for painting or priming.

Some nonwoven materials have a low tendency to shed fibers and may be used as wipers in 15 applications where lint and dust are undesirable such as, for example, micro-electronic manufacturing clean rooms. However, such wipes are typically treated with surfactants to provide the absorbance and clean wiping characteristics desired in such applications. Surfactant treatments typically comprise an anionic surfactant such as, for example sodium dioctyl sulfosuccinate which has a high metallic ion content. These metallic ions provide special problems since, if present in sufficient concentrations, they may adversely 20 affect the electrical properties of metal oxide semiconductors.

Additionally, certain nonwoven materials have a slow rate of electrical charge dissipation which results in static build-up. Static build-up on a wiper may cause problems such as, for example, discomfort for the user, hazards with flammable solvents or damage to sensitive electronic equipment.

Nonwoven materials used in wiping applications typically require some bonding to maintain the integrity 25 of the nonwoven web. Thermal bonding can reduce the content of "active" fibers available for absorption. Thermal bonding also results in a stiffer material which may scratch or abrade a soft surface such as newly applied paint. Chemical bonding offers potential problems with extractable bonding agents.

Nonwoven materials such as, for example, bonded carded webs and air laid webs can be hydroentangled into a coherent web structure and used as wipers. However, these materials typically have high 30 strength in only one direction because the fibers in the web are oriented in only one direction during the initial web forming process. That is, the materials have high strength in one direction such as, for example, the machine direction and relatively low strength in the cross machine direction. This inequality of strength is undesirable because the material is more likely to tear in the weak direction and because the material must be much stronger than necessary in one direction in order to meet minimum strength requirements in 35 the weak direction.

Composite hydroentangled materials containing staple fibers and wood pulp fibers are typically made by overlaying a wood pulp tissue layer on a staple fiber web and hydraulically entangling the two layers. Each side of the resulting hydroentangled material usually has a noticeably different level of abrasion resistance from the other side because of the way the material is produced.

40 Wood pulp and combinations of wood pulp and staple fibers can be processed to make paper tissue and paper items which may be used as wipers. Although these wipers have desirable absorbency, economy, and resistance to certain solvents and chemicals, they generally have low strength (particularly when wet), low toughness, low abrasion resistance and undesirable levels of lint. Such wipers also have poor visual and tactile aesthetics. For example, these materials are typically thin and sheet-like having a 45 thickness index of about 0.01 or typically less than 0.01. Some physical properties of these materials such as, for example, strength and abrasion resistance may be improved by adding binders. However, binders increase the cost of the wiper and may leave residue on the surface to be wiped.

Wipers may also be formed from woven materials. Depending on the material used, the wipers may 50 have desirable absorbency and strength but typically are expensive and must be reused in order to be economical. Reusable cloths are not desirable because they may retain foreign, possibly injurious objects from previous uses. Cloth made from natural fibers has the disadvantage that many natural fibers such as, for example, cotton have natural oils such as, for example, cotton oil that can be extracted by some solvents and deposited onto the wiped surface. Cloth made from man-made fibers such as, for example, polyester may not be able to absorb water unless the fibers are treated with a surfactant so that the fibers 55 are wettable. The presence of surfactants is undesirable for the reasons noted above.

## DEFINITIONS

The term "Peak Load" as used herein is defined as the maximum amount of load or force encountered in elongating a material to break. Peak Load is expressed in units of force, i.e., g<sub>f</sub>.

5 The term "Peak Energy Absorbed" (Peak EA) as used herein is defined as the area under a load versus elongation (stress versus strain) curve up to the point of "peak" or maximum load. Peak EA is expressed in units of work, i.e., kg-mm.

10 The term "Total Energy Absorbed" (TEA) as used herein is defined as the total area under a load versus elongation (stress versus strain) curve up to the point where the material breaks. TEA is expressed in units of work, i.e., kg-mm.

15 The term "Peak Percentage Elongation" as used herein is defined as relative increase in length of a specimen when a material is extended to up to the point of "peak" or maximum load. Peak percentage elongation is expressed as a percentage of the original length of the material, i.e., [(increase in length)/(original length)] X 100.

20 The term "Total Percentage Elongation" as used herein is defined as the relative increase in length of a specimen when a material is extended to up to the point where the material breaks. Total percentage elongation is expressed as a percentage of the original length of the material, i.e., [(increase in length)/(original length)] X 100.

25 The term "Thickness Index" as used herein is defined as the value represented by the ratio of the thickness and the basis weight of a material where the thickness is described in millimeters (mm) and the basis weight is described in grams per square meter (gsm). For example, the thickness index may be expressed as follows:

$$\text{Thickness Index} = [\text{thickness (mm)}/\text{basis weight(gsm)}]$$

30 The term "machine direction" as used herein is defined as the direction of travel of the forming surface onto which fibers are deposited during formation of composite nonwoven material.

35 The term "cross-machine direction" as used herein is defined as the direction which is perpendicular to the machine direction.

40 The term "Isotropic Strength Index" as used herein is defined as the value represented by the ratio of the peak load of a material in one direction such as, for example, the machine direction with the peak load of the material in the perpendicular direction, for example, the cross-machine direction. The index is typically expressed as the ratio of the machine direction peak load with the cross-machine direction peak load. Materials usually have an index of greater than one (1) unless a comparison of peak load in a particular direction is specified. An isotropic strength index near one (1) indicates an isotropic material. An isotropic strength index significantly greater than one (1) indicates an anisotropic material.

45 The term "staple fiber" as used herein refers to natural or synthetic fibers having an approximate average length of from about 1 mm to about 24 mm, for example, from about 6 mm to about 15 mm, and an approximate linear density of about 0,056 to about 0,333 tex (0.5 to about 3 den), for example, from about 0,078 to about 0,167 tex (about 0.7 to about 1.5 denier).

50 The term "Total Absorptive Capacity" as used herein refers to the capacity of a material to absorb liquid and is related to the total amount of liquid held by a material at saturation. Total Absorptive Capacity is determined by measuring the increase in the weight of a material sample resulting from the absorption of a liquid and is expressed, in percent, as the weight of liquid absorbed divided by the weight of the sample. That is, Total Absorptive Capacity = [(saturated sample weight - sample weight)/sample weight] X 100.

55 The term "Mop Up Capacity" as used herein refers to the capacity of a material to absorb liquid after the material has been saturated and wrung to simulate the multiple use of a wiper. The mop up capacity is related to the amount of liquid remaining in a material after liquid is removed from a saturated material by wringing. Mop up capacity is determined by measuring the difference between the saturated weight and the wrung out weight of a material sample and dividing that amount by the weight of the dry sample. It is expressed, in percent, as the weight of liquid removed from the sample by wringing divided by the weight of the dry sample. That is, [(saturated sample weight - wrung out sample weight)/weight of dry sample] X 100.

60 The present invention addresses the above-discussed problems by providing cloth-like nonwoven materials made from mixtures of wood pulp fibers and staple fibers randomly distributed and hydraulically entangled with each other to form a coherent entangled fibrous structure having a thickness index of at least 0.008 and a isotropic strength index of not greater than 1.5.

The materials of the present invention are made in a two step process. The materials are formed by conventional wet-forming techniques using an inclined wire. The materials are then hydroentangled using conventional hydroentangling techniques at pressures ranging from about 3,45 to about 13,8 N/mm<sup>2</sup> (500 to about 2000 pounds per square inch) and at speeds ranging from about 20 to about 300 meters per minute

5 to form a coherent web structure without the use of thermal or chemical bonding.

The wet-formed materials of the present invention contain randomly distributed mixtures of wood pulp fibers and staple fibers. Typical materials contain from about 50 to about 90 percent by weight staple fiber and from about 10 to about 50 percent by weight wood pulp fibers. Materials may contain up to about 100 percent staple fibers. The cloth-like nonwoven materials of the present invention have basis weights from

10 about 30 to about 150 gsm.

Staple fibers used in the invention may have a linear density in the range of about 0,078 to about 0,333 tex (0.7 to about 3 den) and an average length in the range of about 5 mm to about 18 mm. The staple fibers may be one or more of rayon, cotton, polyester, polyamides and polyolefins such as, for example, one or more of polyethylene, polypropylene, polybutene, ethylene copolymers, propylene copolymers and

15 butene copolymers. Long fiber wood pulps such as hardwood pulps are also particularly useful. Mixtures of long fiber and short fiber wood pulps may also be used.

In accordance with the present invention there is provided a cloth-like composite nonwoven material having strength, toughness, abrasion resistance, resistance to certain solvents, and good visual and tactile aesthetics.

20 The cloth-like nonwoven material is made from a dispersion of wood pulp fibers and staple fibers which is formed into a layer of randomly distributed fibers on a foraminous surface by conventional wet-laying techniques using an inclined wire. Exemplary wet-forming processes are described in, for example, US-A-2,414,833 to Osborne, the disclosure which is hereby incorporated by reference.

In the headbox of the wet-forming apparatus, the dispersion of fibers may be dilute, for example, 25 containing about 2.5 grams of dry fiber per liter of fiber and water mixture. The consistency of the uniform layer of fibers after formation on the foraminous surface may range from about 10 to about 30 weight percent fiber solids in water. For example, the consistency may be about 25 percent by weight solids. The uniform layer of fibers may be transferred to a different surface for entangling. The entangling surface may be, for example, a wire screen of from about 35 to about 100 mesh. The entangled material may be 30 transferred to another surface for patterning. Mesh size and/or the texture of the foraminous patterning surface can be varied to create different visual and tactile properties. A coarse mesh such as, for example, from about 14 to about 35 mesh can be used to impart a textile or cloth-like appearance and feel.

The newly formed layer of randomly distributed fibers is hydraulically entangled to form a nonwoven material. Exemplary hydraulic entangling processes are described in, for example, US-A-3,485,706 to 35 Evans, the disclosure of which is hereby incorporated by reference. For example, entangling may be effected with a manifold produced by Honeycomb Systems, Incorporated containing a strip having 0,127 mm (0.005 inch) diameter orifices, 16 holes per cm (40 holes per inch) and 1 row of holes. Other manifold configurations may also be used. The wet-formed materials may be run under the strip at speeds ranging from about 20 to about 300 meters per minute to be entangled by jets of liquid at pressures ranging from

40 about 3,45 to about 13,8 N/mm<sup>2</sup> (500 to about 2000 psi). It has been found that greater strength materials have been obtained by hydroentangling the base sheets at slower speeds and/or higher pressures. Additional passes through the hydroentangling equipment also yields improved strength.

Patterning may be accomplished by transferring the entangled material to a coarse mesh such as, for example, 14 to about 35 mesh and running the material under the hydraulic entangling apparatus at 45 pressures from about 1,38 to about 6,9 N/mm<sup>2</sup> (200 to about 1000 psi).

The nonwoven material formed by hydraulic entangling may be dried utilizing one or more conventional drying methods such as, for example, forced air, vacuum, heat or pressure. The nonwoven material may be dried on a foraminous surface such as, for example, a wire mesh. Alternatively, the nonwoven material may be dried on an un-textured surface by conventional drying methods. Materials dried on a foraminous 50 surface are softer and more drapeable than materials dried on an un-textured surface. Additionally, materials dried on a foraminous surface can be expected to have lower peak loads but greater peak elongations than materials dried on an un-textured surface.

In connection with this description certain test procedures have been employed to determine oil and water absorption capacity and rate, linting, abrasion resistance, static decay, drape stiffness, sodium ion 55 concentration, level of extractables, peak load, peak energy absorbed, total energy absorbed, peak elongation, and total elongation.

Lint tests were carried out using a Climet™ particle counter model CI-250 available from the Climet Instrument Company, Redlands, California. Test were conducted essentially in accordance with INDA

Standard Test 160.0 - 83 with the following changes: (1) the sample size was 152 x 152 mm (6 inches X 6 inches); and (2) the background count was not determined for each individual specimen tested. This test employed a mechanical particle generator which applied bending, twisting and crushing forces to sample specimens. Samples were placed in machine direction alignment in an enclosure and twisted through an angle of 150° for a distance of 106,7 mm (4.2 inches) at a rate of about 70 cycles per minute. The enclosure is connected by tubing to the particle counter which draws the particles to the counter at a rate of about 0,566 m<sup>3</sup> (20 cubic feet) per hour. The flow rate through the instrument sensor is 0,028 m<sup>3</sup> (1.0 cubic feet) per hour. Each count takes 36 seconds and represents the number of particles of the specified size in 0,00028 m<sup>3</sup> (0.01 cubic feet) of air.

10 Grab Tensile Test were conducted essentially in accordance with Method 5100 of Federal Test Method Standard No. 191A, utilizing samples of the entangled material having a width of about 102 mm (4 inches) and a length of about 152 mm (6 inches). The samples were held at opposite ends by a 6,45 cm<sup>2</sup> (one square inch) gripping surface. The samples were tested with an Intellect II Model tensile testing apparatus available from Thwing Albert and with an Instron Model 1122 Universal Testing Instrument, each having a 15 76,2 mm (3 inch) jaw span and a crosshead speed of about 305 mm (12 inches) per minute. Values for peak load, peak energy absorbed, peak percentage elongation, total energy absorbed and total percentage elongation were determined.

15 The rate of electrical charge dissipation of the material was determined essentially in accordance with Method 4046 of Federal Test Method Standard No. 101B. Test results were obtained with an Electro/Tech™ Calibrated Electrostatic Charge Detector with High Voltage Sample Holder using rectangular samples measuring 140 x 89 mm (5-1/2 inches X 3-1/2 inches).

20 The rate that the material absorbed oil was determined as follows: A sample measuring 300 mm in the cross-machine direction and about 150 mm in the machine direction was placed flat on the liquid surface of an oil bath containing SAE 20W/50 motor oil. A stopwatch was used to record the time for the sample to completely wet-out, that is, total saturation of 99 percent of the surface area of the sample. Non absorbent streaks of the material are not acceptable under the definition of complete wet-out but non absorbent individual fibers are acceptable. The rate that the material absorbed water was determined by the same procedures utilized for oil except that distilled water was used instead of oil.

25 The capacity of the material to absorb oil was determined as follows: A dry 15 cm X 30 cm standard felt available from the British Paper and Board Industry Federation, London, England was submerged for at least 24 hours in an oil bath containing SAE 20W/50 motor oil. The weight of a 10 cm X 10 cm material sample was determined to the nearest 0.01 gram. The sample was then submerged in the oil bath over the piece of felt until the sample was completely saturated (at least 1 minute). The felt and sample were removed and suspended over the bath until the observed drainage of oil from the sample was complete. 35 i.e., when the sample assumed a single overall color or appearance. The drained sample was weighed to the nearest 0.01 gram and the total absorptive capacity was calculated.

30 The mop up capacity of the material was determined from the sample in the total absorptive capacity test by folding the saturated sample in half, and then in half again. The sample was then grasped between the thumb and fore finger on opposite edges and twisted as far as possible to wring oil from the sample. 40 The oil was allowed to drain while the sample was twisted. When no further oil drained from the twisted sample the sample was untwisted. The sample was weighed to the nearest 0.01 gram and the mop up capacity was determined.

35 The capacity of the material to absorb and mop up water was determined by the same procedures utilized for oil except that distilled water was used instead of oil.

45 The drape stiffness measurements were performed using a Shirley Stiffness Tester available from Shirley Developments Limited, Manchester, England. Test results were obtained essentially in accordance with ASTM Standard Test D 1388 except that the sample size was 25,4 x 203,2 mm (1 inch X 8 inches) with the larger dimension in the direction being tested.

50 The levels of (1) extractables in isopropyl alcohol, 1,1,1-trichloroethane and distilled water and (2) the concentration of sodium ions was determined by the following procedure. Duplicate samples of the wipes weighing approximately 2 grams were refluxed for 4 hours in 200 mL of solvent using a soxhlet extraction apparatus. The solvent was evaporated to dryness and the percent extractables was calculated by determining the difference in the weight of the container before and after evaporation. The percent extractables is expressed as weight percent of the starting material. The quantity of sodium in the sample 55 was determined by measuring the concentration of sodium ions in water obtained from the soxhlet extraction apparatus after the water extractables test. A Perkin-Elmer Model 380 atomic absorption spectrophotometer was used to measure the sodium ion concentration in the water.

The abrasion resistance of the material was determined essentially in accordance with British Standard Test Method 5690: 1979 with the following changes: (1) the abrasion machine used was available under the trade designation Martindale Wear and Abrasion Tester Model No. 103 from Ahiba-Mathis, Charlotte, North Carolina; (2) the samples were subjected to 100 abrasion cycles under a pressure of 1.3 pounds per square inch (psi) or 9 kilopascals (kPa); (3) a 38.1 mm (1.5 inch) diameter abradant was a cut from a 914.4 x 101.6 x 1.27 ( $\pm 0.127$ ) mm (36 inch X 4 inch X 0.050 ( $\pm 0.005$ ) inch) piece of glass fiber reinforced silicone rubber having a surface hardness of 81A Durometer, 81 $\pm$ 9 Shore A available from Flight Insulation Incorporated, Marietta, Georgia, distributors for Connecticut Hard Rubber; and (4) the samples were examined for the presence of surface fuzzing (fiber lofting), pilling, roping, or holes. The samples were compared to a visual scale and assigned a wear number from 1 to 5 with 1 indicating little or no visible abrasion and 5 indicating a hole worn through the sample.

#### EXAMPLE 1

A mixture of about 50 percent by weight hardwood pulp available from the Weyerhauser Company under the trade designation Grade Regular and about 50 percent by weight uncrimped polyester staple fiber of 0,167 tex x 12 mm (1.5 denier x 12 mm), was dispersed to a consistency of about 0.5 percent by weight solids and then formed into handsheets of about 75 gsm on a standard 94 x 100 mesh plastic screen.

A manifold available from Honeycomb Systems, Incorporated was utilized to entangle the handsheets. The handsheets were transferred to a standard 100 x 92 mesh stainless steel wire. The manifold was positioned approximately 12.7 mm (one-half inch) above the stainless steel wire mesh. The manifold contained a strip having 0.127 mm (0.005 inch) diameter orifices, 16 holes per cm (40 holes per inch) and 1 row of holes. The strip was inserted into the manifold with the conical shaped holes diverging in the direction of the wire. Entanglement was performed with the handsheet travelling at a speed of about 20 meters per minute.

The handsheets were entangled at pressures of 1,38; 2,76; 4,14; 5,52; 8,28; and 9,66 N/mm<sup>2</sup> (200, 400, 600, 800, 1200 and 1400 psi) on one side of the sheet and at pressures of 8,28 and 9,66 N/mm<sup>2</sup> (1200 and 1400 psi) on the opposite side of the sheet. The flow rate of the entangling water was 415 m<sup>3</sup>/h/cm (1.054 cubic meters per hour per inch) of strip. The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 70 gsm.

Samples of the entangled material having a width of about 102 mm (4 inches) were tested using an Intellect II tensile testing apparatus available from Thwing Albert and an Instron Model 1122 Universal Testing Instrument, each having a 76.2 mm (3 inch) jaw span and a crosshead speed of about 305 mm (12 inches) per minute. Values for Peak Load, Peak EA, Peak Percentage Elongation, TEA and Total Percentage Elongation for the dry samples are reported in Table 1 for the machine direction and the cross-machine direction. Similar data was collected for wet samples in the machine direction only and is also reported in Table 1.

#### EXAMPLE 2

A mixture of about 20 percent by weight hardwood pulp available from the Weyerhauser Company under the trade designation Grade Regular, about 40 percent by weight uncrimped polyester staple fiber of 0,167 tex x 12 mm (1.5 denier x 12 mm) and about 40 percent by weight uncrimped rayon staple fiber of 0,167 tex x 12 mm (1.5 denier x 12 mm) was dispersed and then formed into handsheets of about 75 gsm on a standard 94 x 100 mesh plastic screen.

The handsheet was entangled using the equipment and procedure of Example 1 on a standard 100 x 92 mesh stainless steel wire at pressures of 4,14; 6,21; 8,28 and 10,35 N/mm<sup>2</sup> (600, 900, 1200 and 1500 psi) on one side of the sheet and at pressures of 8,28 and 10,35 N/mm<sup>2</sup> (1200 and 1500 psi) on the opposite side of the sheet. The flow rate of the entangling water was 0,318 m<sup>3</sup>/h/cm (0.808 cubic meters per hour per inch) of strip. The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 73 gsm.

Samples of the entangled material having a width of about 4 inches were tested using the equipment and procedures of Example 1. Values for Peak Load, Peak EA, Peak Percentage Elongation, TEA and Total Percentage Elongation for the dry samples are reported in Table 2 for the machine direction and the cross-machine direction.

## EXAMPLE 3

5 A mixture of about 18.5 percent by weight hardwood pulp available from the Weyerhauser Company under the trade designation Grade Regular, about 78.5 percent by weight uncrimped polyester staple fiber of 0,167 tex x 12 mm (1.5 denier x 12 mm) and about 3 percent by weight polyvinyl alcohol binder fiber was dispersed and then formed continuously onto a foraminous surface at about 60 gsm. The web was formed utilizing a continuous inclined wire paper making machine. The web was dried over a series of steam heated cans. Polyvinyl alcohol was added to facilitate reeling and handling.

10 The dried web was re-wetted and then entangled using the equipment and procedure of Example 1 on a standard 100 x 92 mesh stainless steel wire employing 6 passes at pressures of 12,42 N/mm<sup>2</sup> (1800 psi) on each side of the sheet. The flow rate of the entangling water was 0,8 m<sup>3</sup>/h/cm (2.04 cubic meters per hour per inch) of strip. The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 53 gsm.

15 Samples of the entangled material having a width of about 102 mm (4 inches) were tested using the equipment and procedures of Example 1. Values for Peak Load, Peak EA, Peak Percentage Elongation, TEA and Total Percentage Elongation for the dry samples are reported in Table 3 for the machine direction and the cross-machine direction.

## EXAMPLE 4

20 A mixture of about 19 percent by weight hardwood pulp available from the Weyerhauser Company under the trade designation Grade Regular, about 39 percent by weight uncrimped polyester staple fiber of 0,167 tex x 12 mm (1.5 denier x 12 mm), about 39 percent by weight uncrimped rayon staple fiber of 0,167 tex x 12 mm (1.5 denier x 12 mm) and about 3 percent by weight polyvinyl alcohol binder fiber was dispersed and then formed continuously onto a foraminous surface at about 60 gsm. The web was formed utilizing a continuous inclined wire paper making machine. The web was dried over a series of steam heated cans. Polyvinyl alcohol was added to facilitate reeling and handling.

25 The dried web was pre-wetted and then entangled using the equipment and procedure of Example 1 on a standard 100 X 92 mesh stainless steel wire. Pre-wetting was done on one side at pressures of 1,38; 2,76 and 4,14 N/mm<sup>2</sup> (200, 400 and 600 psi). Entangling on that side was performed at pressures of 5,52; 6,9; 8,28 (800, 1000, 1200) and three passes at 10,35 N/mm<sup>2</sup> (1500 psi). The other side of the material was entangled by 3 passes at 10,35 N/mm<sup>2</sup> (1500 psi). The entangled sheets were air dried at ambient temperature. The dried material had a basis weight of about 53 gsm.

30 Samples of the dried and the entangled material having a width of about 102 mm (4 inches) were tested using an Intellect II tensile testing apparatus with a 76,2 mm (3 inch) jaw span and a crosshead speed of about 254 mm (10 inches) per minute. Values for Peak Load, Peak EA and Peak Strain are reported in Table 4 for the machine direction and the cross-machine direction for dry samples. Similar results are also reported in Table 4 for wet samples.

35 For comparative purposes, Table 5 lists the Thickness Index, Isotropic Strength Index, abrasion test results, and drape stiffness test results for the entangled material of Examples 2, the entangled and unentangled material of Example 4, and two commercially available materials which can be used for wiping. Wiper A is a hydraulically entangled nonwoven material having the trade designation Sontara, grade 8005 available E.I. duPont De Nemours and Company. Wiper B is made from a wood pulp/staple fiber blend formed by laying a wood pulp web over a staple fiber web and then hydroentangling the webs. Wiper B has 45 the trade designation Mohair Bleu and is available in France from Maury of Nantes, France and from Sodave of Angers, France. Table 5 also lists the thickness index and the isotropic strength index for the identified materials.

40 As can be seen from Table 5, the hydroentangled materials from Examples 2 and 4 have a greater thickness index than the unentangled material of Example 4, Wiper A and Wiper B. The materials from Examples 2 and 4 also have a greater isotropic strength index than Wipers A and B.

45 Table 6 provides results of testing for the absorption rate, total absorptive capacity and mop-up capacity of the material from Example 4 for oil and water. The material of Example 2 had a total absorptive capacity and mop-up capacity for both oil and water which is significantly greater than the values for Wiper B.

50 Tables 7, 8 and 9 provide test results for the materials of the present invention and for various other wipers that are commercially available in Europe. Wiper CW1 is made of a meltblown polypropylene fabric. Wiper CW2 is a laminate of spunbonded polypropylene/meltblown polypropylene/spunbonded polypropylene. The wiper available under the trade designation MIRACLE WIPES is made of hydroentangled

staple and cellulosic fibers. The wiper available under the trade designation CLEAN ROOM WIPERS is made of wet formed staple and cellulosic fibers. The wiper available under the trade designation DURX is made of hydroentangled staple and cellulosic fibers. The wiper available under the trade designation LABX is made of wet-formed staple and cellulosic fibers. The wiper available under the trade designation

5 TEXWIPE is made of a 100 percent cotton woven fabric. The wiper available under the trade designation MICRONWIPE is made of hydroentangled staple and cellulosic fibers. The wiper available under the trade designation TEXBOND is made of a spunbonded nylon fabric. The wiper available under the trade designation TECHNI-CLOTH is made of hydroentangled staple and cellulosic fibers.

For comparative purposes, Table 7 lists the results of extractable tests and sodium ion tests for the material of Example 2 and for some of the above-mentioned wipers. Also included in Table 7 are results for two materials made according Example 2. Material H contains about 80 percent by weight rayon staple fibers and about 20 percent by weight wood pulp. Material F contains about 80 percent by weight polyester staple fibers and about 20 percent by weight wood pulp. Table 8 lists the results of electrical charge dissipation tests for the material of Example 2, Wiper A and for some of the above-mentioned wipers. Table 15 9 lists the results of Climet™ lint tests for the materials from Example 2, the entangled and untangled material from Example 4, Wiper A, and for some of the above-mentioned wipers.

As shown in Table 7, the materials of the present invention have levels of extractables which compare favorably with many commercial wipers. From Table 8, it can be seen that the materials of the present invention without any anti-static treatment have a static decay which is comparable with many commercial 20 wipers. From Table 9, it can be seen that the materials of the present invention have relatively low lint levels and compare favorably with many commercial wipers.

Thus, it is apparent that the present invention provides a wiper that satisfies problems associated with previous wipers. While the invention has been described in conjunction with specific embodiments, the disclosed embodiments are intended to illustrate and not to limit the invention.

25

TABLE 1

DRY	MACHINE DIRECTION	CROSS-MACHINE DIRECTION
30	Peak Load (g)	11,677
	Peak Energy Absorbed (kg-mm)	106
	Peak Strain (%)	68.5
	Total Energy Absorbed (kg-mm)	198
	Total Strain (%)	131
35	WET	
	Peak Load (g)	7214
	Peak Energy Absorbed (kg-mm)	117
	Peak Strain (%)	120
	Total Energy Absorbed (kg-mm)	196
40	Total Strain (%)	217

45

TABLE 2

DRY	MACHINE DIRECTION	CROSS-MACHINE DIRECTION
50	Peak Load (g)	9125
	Peak Energy Absorbed (kg-mm)	55
	Peak Strain (%)	46
	Total Energy Absorbed (kg-mm)	114
	Total Strain (%)	100

55

TABLE 3

DRY	MACHINE DIRECTION	CROSS-MACHINE DIRECTION
5 Peak Load (g)	5035	4081
Peak Energy Absorbed (kg-mm)	63	71
Peak Strain (%)	89	118
Total Energy Absorbed (kg-mm)	124	99
Total Strain (%)	174	160

10

TABLE 4

DRY	MACHINE DIRECTION	CROSS-MACHINE DIRECTION
15 Peak Load (g)	4529	4133
Peak Energy Absorbed (kg-mm)	83	79
Peak Strain (%)	39	49
20 WET		
Peak Load (g)	4014	
Peak Energy Absorbed (kg-mm)	62	
Peak Strain (%)	37	

25

TABLE 5

		EXAMPLE 2	EXAMPLE 4	BASE SHEET EXAMPLE 4	WIPER A	WIPER B
30	Isotropic Strength Index	1.04	1.096	1.0	2.37	1.45
	Thickness (mm)	0.79	0.73	0.36	0.44	0.31
35	Basis Weight (gsm)	73	53	60	65	75
	Thickness Index	0.011	0.014	0.006	0.007	0.004
	Drape Stiffness (cm)					
40	Side 1		3.6		3.4	7.7
	Side 2		3.2		2.8	4.8
	Martindale Abrasion					
45	Rating	Side 1	1	2	2	3
		Side 2	1	2	1	1

45

50

55

TABLE 6

	WATER	EXAMPLE 4	WIPE B
5	Absorption Rate (sec.)	1.0	<1
	Total Absorptive Capacity (%)	553	347
	Mop-Up Capacity (%)	258	151
10	OIL		
	Absorption Rate (sec.)	9.0	7.0
	Total Absorptive Capacity (%)	596	230
	Mop-Up Capacity (%)	250	33

TABLE 7

	EXTRACTABLES				
	PRODUCT/CODE	% in isopropyl alcohol	% in 1,1,1-trichloroethane	% in hot water	hot water leachable sodium (ppm)
	CW1	0.8	0.7	2.5	43
	CW2	0.8	3.5	0.2	47
	Miracle Wipes® 1003	<0.1	1.7	0.2	20
20	Clean Room Wipers® 8025	2.1	1.6	1.0	391
	Durx® 670	<0.1	<0.1	0.3	41
	Durx® 770	0.2	0.1	0.4	177
25	Labx® 123	<0.1	<0.1	1.1	206
	Texwipe™ 309	0.3	<0.1	3.1	47
30	Micronwipe® 500	<0.1	<0.1	3.4	1030
	Texbond™ 909	5.4	3.4	0.3	1640
	Techni-Cloth® 609	<0.1	4.3	1.4	43
35	Techni-Cloth® II 1009	<0.1	0.4	2.5	43
	Example 2	0.1	0.1	1.5	126
	Wiper Material H	0.2	0.1	1.0	133
	Wiper Material F	0.2	0.1	0.6	116

40

45

50

55

TABLE 8

ELECTRICAL PROPERTIES		
	PRODUCT/CODE	static decay (sec)
5	CW1	14.7
	CW2	19.8
10	Miracle Wipes® 1003	0.3
	Clean Room Wipers® 8025	4.5
	Durx® 670	5.8
	Labx® 123	1.3
	Texwipe™ 309	1.1
15	Micronwipe® 500	0.9
	Texbond™ 909	6.5
	Techni-Cloth® 609	15.2
	Techni-Cloth® II 1009	1.6
	Example 2	7.0
20	Wiper A	No Dissipation
	Wiper B	3.6

Notes: 1. Higher static decay times indicate increased tendency for static charge accumulation.

TABLE 9

25

CLIMET LINE (# PARTICLES)			
	PRODUCT/CODE	10 $\mu\text{m}$	0.5 $\mu\text{m}$
30	CW1	0.4	112
	CW2	0.1	9
	Miracle Wipes® 1003	0.2	56
	Clean Room Wipes® 8025	0.2	4
35	Drux® 670	0.4	442
	Labx® 123	0.4	428
	Texwipe™ 309	2.6	5130
	Micronwipe® 500	0.7	498
40	Texbond™ 909	0.0	7
	Techni-Cloth® 609	0.6	358
	Techni-Cloth® II 1009	0.4	8
	Example 2	2	65
	Example 4 (Entangled)	0.8	76
45	Example 4 (Base Sheet)	2	72
	Wiper A	0.8	51
	Wiper B	0.2	286
	Example 1	0.2	328

## Claims

50

1. A hydraulically entangled coherent fibrous structure comprising:  
from 0 to 50 percent by weight wood pulp fibers; and from 50 to 100 percent by weight of staple fibers;  
and  
wherein the structure has a basis weight of from 30 gsm to 150 gsm, a thickness index of at least 0.008  
and an isotropic strength index less than 1.5.
2. The structure of claim 1 wherein the structure comprises:  
from 10 to 50 percent by weight wood pulp fibers; and from 50 to 90 percent by weight of staple fibers.

3. The structure of claim 1 or 2 wherein the staple fibers have a linear density in the range of about 0.078 to about 0.333 tex (0.7 to about 3 den) and an average length in the range of about 5 mm to about 18 mm.
- 5 4. The structure of any one of claims 1 to 3 wherein the staple fibers comprise one or more of rayon, cotton, polyesters, polyolefins, and polyamides.
- 5 5. The structure of any one of claims 1 to 4 wherein the material has an oil absorption capacity of at least about 300 percent.
- 10 6. The structure of any one of claims 1 to 4 wherein the material has a water absorption capacity of at least about 375 percent.
- 15 7. The structure of any one of claims 1 to 6 wherein the material has a sodium ion content of less than about 150 parts per million.
8. The structure of any one of claims 1 to 7 wherein the material is capable of being elongated 104 percent in at least one direction.
- 20 9. The structure of claim 8, wherein the material has a water absorption capacity of at least about 375 percent.
10. The structure of claim 8 or 9 wherein the material has a hot water leachable sodium ion content of less than about 150 parts per million.
- 25 11. The structure of any one of claims 1 to 10 wherein the level of materials extractable in isopropyl alcohol is 0.2 percent, by weight and the level of materials extractable in 1,1,1-trichloroethane is 0.1 percent, by weight.

30 **Patentansprüche**

1. Hydraulisch verschlungene, kohärente Faserstruktur, die von 0 bis 50 Gew.-% Holzpulpefasern und von 50 bis 100 Gew.-% Stapelfasern enthält, und wobei die Struktur ein Grundgewicht von 30 g bis 150 g/m<sup>2</sup>, einen Dickenindex von mindestens 0,008 und einen isotropen Festigkeitsindex geringer als 1,5 aufweist.
- 35 2. Struktur nach Anspruch 1, wobei die Struktur umfaßt von 10 bis 50 Gew.-% Holzpulpefasern und von 50 bis 90 Gew.-% Stapelfasern.
- 40 3. Struktur nach Anspruch 1 oder 2, wobei die Stapelfasern eine lineare Dichte im Bereich von etwa 0,078 bis etwa 0,333 tex (0,7 bis etwa 3 den) und eine mittlere Länge im Bereich von etwa 5mm bis etwa 18mm aufweisen.
- 45 4. Struktur nach einem der Ansprüche 1 bis 3, wobei die Stapelfasern eine oder mehrere der Materialien Rayon, Baumwolle, Polyester, Polyolefine und Polyamide enthalten.
5. Struktur nach einem der Ansprüche 1 bis 4, wobei das Material eine Ölabsorptionskapazität von mindestens etwa 300% aufweist.
- 50 6. Struktur nach einem der Ansprüche 1 bis 4, wobei das Material eine Wasserabsorptionskapazität von mindestens etwa 375 % aufweist.
7. Struktur nach einem der Ansprüche 1 bis 6, wobei das Material einen Natriummionengehalt von weniger als etwa 150 ppm aufweist.
- 55 8. Struktur nach einem der Ansprüche 1 bis 7, wobei das Material in mindestens einer Richtung um 104% ausdehnungsfähig ist.

9. Struktur nach Anspruch 8, wobei das Material eine Wasserabsorptionskapazität von mindestens etwa 375% aufweist.
10. Struktur nach Anspruch 8 oder 9, wobei das Material einen in heißem Wasser auslaugbaren Natrium-5 mionengehalt von weniger als etwa 150 ppm aufweist.
11. Struktur nach einem der Ansprüche 1 bis 10, wobei der Gehalt an Materialien, die in Isopropylalkohol extrahierbar sind 0,2 Gew.-% beträgt und wobei der Gehalt an Material, das in 1,1,1-Trichloräthan extrahierbar ist 0,1 Gew.-% beträgt.

10

## Revendications

1. Structure fibreuse cohérente enchevêtrée par voie hydraulique comprenant : de 0 à 50 % en poids de fibres de pâte de bois ; et de 50 à 100 % en poids de fibres coupées ; et dans laquelle la structure a un grammage de 30 g/m<sup>2</sup> à 150 g/m<sup>2</sup>, un indice d'épaisseur d'au moins 0,008 et un indice de résistance isotrope inférieur à 1,5.
2. Structure selon la revendication 1, qui comprend : de 10 à 50 % en poids de fibres de pâte de bois ; et de 50 à 90 % en poids de fibres coupées.
3. Structure selon les revendications 1 ou 2, dans laquelle les fibres coupées ont une densité linéaire dans l'intervalle d'environ 0,078 à environ 0,333 tex (0,7 à environ 3 den) et une longueur moyenne dans l'intervalle d'environ 5 mm à environ 18 mm.
4. Structure selon l'une quelconque des revendications 1 à 3, dans laquelle les fibres coupées comprennent de la rayonne et/ou du coton et/ou des polyesters et/ou des polyoléfines et/ou des polyamides.
5. Structure selon l'une quelconque des revendications 1 à 4, dans laquelle la matière a une capacité d'absorption d'huile d'au moins environ 300 %.
6. Structure selon l'une quelconque des revendications 1 à 4, dans laquelle la matière a une capacité d'absorption d'eau d'au moins environ 375 %.
7. Structure selon l'une quelconque des revendications 1 à 6, dans laquelle la matière a une teneur en ions sodium inférieure à environ 150 parties par million.
8. Structure selon l'une quelconque des revendications 1 à 7, dans laquelle la matière est susceptible d'être allongée de 104 % dans au moins une direction.
9. Structure selon la revendication 8, dans laquelle la matière a une capacité d'absorption d'eau d'au moins environ 375 %.
10. Structure selon les revendications 8 ou 9, dans laquelle la matière a une teneur en ions sodium lessivables par l'eau chaude inférieure à environ 150 parties par million.
11. Structure selon l'une quelconque des revendications 1 à 10, dans laquelle le taux de matières extractibles par l'alcool isopropylique est de 0,2 % en poids, et le taux de matières extractibles par le 1,1,1-trichloéthane est de 0,1 % en poids.

45

50

55