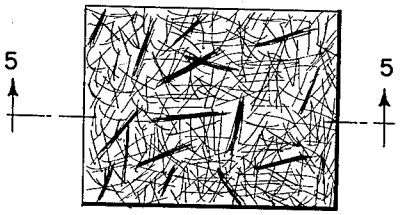
Nov. 7, 1961 F. S. WILCOX 3,007,840

PROCESS OF DISPERSING FIBROUS MATERIAL IN A FOAM AND RESULTING PRODUCT

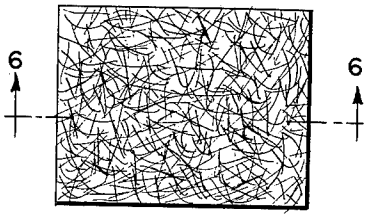
Filed April 3, 1958

FIG. 1



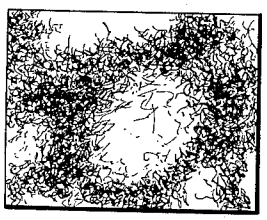
Mat of 1/4" 3.5 d. fibers deposited from aqueous dispersion

FIG. 2



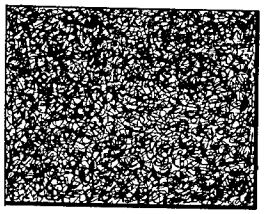
Mat of 1/4" 3.5 d. fibers deposited from a foam

FIG. 3



Mat of 1/4" 4.75 d. crimped fibers deposited from aqueous dispersion

FIG. 4



Mat of 1/4" 4.75 d. crimped fibers deposited from foam

FIG. 5



Cross-sectional illustration taken on line 5-5 of Fig.1 showing laminar arrangement of fibers

FIG. 6



Cross-sectional illustration taken on line 6-6 of Fig. 2 showing random arrangement of fibers

INVENTOR

FORREST S. WILCOX

BY P. J. Poindexter

AGENT

United States Patent Office 3,007,840
Patented Nov. 7, 1961

3,007,840

PROCESS OF DISPERSING FIBROUS MATERIAL IN A FOAM AND RESULTING PRODUCT

Forrest S. Wilcox, Newburgh, N.Y., assignor to E. I. du Pont de Nemours and Company, Wilmington, Del., a corporation of Delaware

Filed Apr. 3, 1958, Ser. No. 726,186
15 Claims. (Cl. 162—101)

This invention relates to a novel process of forming non-woven fibrous webs and improved products resulting therefrom.

In the making of paper it is well known to form a slurry of short fibers in water, run the fiber dispersion onto a porous screen, drain the water through the screen and deposit a web of fibers thereon. Such processes are basically the same since paper was first manufactured.

It has been a problem to form uniform water-laid webs from many synthetic fibers, particularly those which are crimped or strongly hydrophobic, such as, e.g., glass, polytetrafluoroethylene and various elastomeric fibers; also, all types of fibers having a relatively long length, i.e., greater than about ¼ to ½ inch (depending upon the denier and stiffness). Such fibers agglomerate, ball-up, and form large clumps when dispersed in water at the normal concentration used for paper making, i.e., .05% to .1% fiber and 99.95% to 99.9% water.

The primary object of this invention is the provision of a process for and product of uniform web made from fibers or fibers and fibrids having properties or physical shape which prevent the formation of a uniform non-woven web from an aqueous slurry of the fibrous material by prior art techniques. A further object is the provision of a uniform non-woven water-laid web (also know as a waterleaf) comprising strongly hydrophobic staple fibers. A still further object is the provision of uniform non-woven water-laid web comprising crimped fibers. A still further object is the provision of uniform non-woven, water-laid web comprising elastomeric staple fibers.

The above objects are accomplished by forming a dispersion comprising staple fibers in a foam and separating a non-woven web therefrom by depositing the foam, having the fibrous material dispersed therein, on a screen or other porous substrate; whereby the aqueous medium passes through the porous substrate and deposits a uniform web thereon. The resulting web is more uniform than those made heretofore from a dispersion of the fibrous material in a continuous liquid phase. Furthermore, waterleaves deposited from foam are not as laminar as those formed from dispersions in a continuous aqueous phase. The deposition of webs from a foam dispersion yields increased orientation of the fibers perpendicular to the plane of the web.

The term "fibrids" as used throughout the specification and claims is intended to designate a heterogeneous mass of particles, the particles having at least one dimension of 10 microns or less and of minor magnitude relative to its largest dimension, ribbon-like portions of the fibrous matter among said particles being no greater than 100 microns in width, said particles being non-rigid and small enough to pass through a 10 mesh screen, yet large enough so that 90% is retained by a 200-mesh screen when deposited from an agitated dilute suspensoid; said particles being further characterized by a freeness number of between about 100 and 750 when in the form of an aqueous slurry, and a capacity to form a waterleaf.

The polymeric fibrids useful in this invention are more fully described in copending U.S. application S.N. 635,876, filed January 23, 1957, by P. W. Morgan, now

abandoned, and they may be prepared in accordance with the procedures set forth therein.

FIGURE 1 of the drawing is a plan view of a non-woven web made from a conventional aqueous slurry of ¼ inch, 3.5 denier polyethylene terephthalate fibers dispersed in a continuous aqueous phase. FIGURE 2 is a plan view of a non-woven web made from the same fibers as illustrated in FIGURE 1, the difference being the web was formed from a dispersion of fibers in a foam instead of a continuous aqueous medium. FIGURE 3 is a plan view of a non-woven web of ¼ inch, 4.75 denier crimped polyethylene terephthalate fibers deposited from a conventional aqueous dispersion of the fibers. FIGURE 4 is a plan view of a non-woven web made from the same fibers illustrated in FIGURE 3, the difference being the web was formed from a dispersion of the fibers in a foam. FIGURE 5 is a cross section illustration taken on line 5—5 of the non-woven web illustrated in FIGURE 1 of the drawing which shows the planar or laminar arrangement of the fibers. FIGURE 6 is a cross-sectional illustration taken on line 6—6 of the non-woven web illustrated in FIGURE 2 of the drawing which shows the three dimensional arrangement of the fibers.

The following specific examples are given by way of illustration and not limitation, the parts and percentage figures are given on a weight basis unless stated otherwise.

Example I

To 1,000 parts of water was added 5 parts of "Triton X–100" (octylphenyl polyglycol ether) and 1 part of ¼ inch long, 3.5 denier, polyethylene terephthalate fibers which are self elongatable when heated. The above components were subjected to agitation for 30 seconds which converted substantially all the water from a liquid phase to a foam. The foam with the fibers dispersed therein was poured into a paper forming box having a screen in the bottom and containing sufficient water over the screen to cover it with about 2 inches of water. As soon as the aqueous foam containing the fibers was poured into the paper forming box, a vacuum was applied which pulled the water and foam from the box and the fibers were uniformly deposited on the screen in a three dimensional arrangement.

The wet web was removed from the screen and dried. The web was bulked by heating at 200° C. which caused the fibers to elongate an average of about 20% of their original length. The web was extremely uniform, weighed about 1.4 ounces per square yard and had a rating of +2 on arbitrary scale ranging from −2 to +2, where zero represents a degree of uniformity which would be acceptable.

A web formed in like manner from a dispersion of the same fibers in the same amount of water, but without the "Triton" foaming agent, had a rating of −1 in uniformity of fibers in the dry web. The dry web had considerable clumping of fibers as well as voids in the web.

Generally, in practicing this invention, in the case of relatively short fibers, i.e., less than about .5 inch, the order of addition for the components to form the dispersion of the fibers in the foam is not critical. The fibers may be added first to the container in which the fiber dispersion is formed followed by the addition of water and the foaming agent. Alternatively the fibers can be added to the foam. In the case of fibers having a length greater than about .5 inch, it is generally desirable to add the fibers to the preformed foam. It is sometimes desirable to treat the fibers with a foaming agent before dispersing them in water. In certain cases, it is advantageous to disperse the fibers, treated with foaming agent, in the water by mild agitation before

forming the foam by more vigorous agitation. The preferred order of addition of the components in forming the dispersion is dependent upon the length and denier of the fibers. The self elongatable (when heated) fibers used in the above example are prepared in accordance with copending application S.N. 718,114, filed February 28, 1958, by Kitson and Reese.

Essentially the same results were obtained when the following fibers were substituted on a pound for pound basis for the ¼ inch, 3.5 denier polyethylene terephthalate in the above Example I:

Example	Type of Staple Fiber	Denier	Staple Length, inches
II	Nylon [1]	15.0	1½
III	do [1]	3.0	¾
IV	Polyethylene Terephthalate	0.1	¼
V	Crimped Polyethylene Terephthalate (8–9 crimps per inch).	4.75	¼
VI	Crimped polyethylene terephthalate (16 crimps per inch).	3.0	½
VII	Polytetrafluoroethylene	6.0	½
VIII	Crimped Polyacrylonitrile (8–9 crimps per inch).	6.0	½

[1] Polyhexamethylene adipamide.

Example IX

A foamed fiber dispersion having the following composition was prepared:

	Parts by wt.
Water	1000
"Triton" X–100	5
Polyethylene terephthalate fibers (self-elongatable when heated) same as Example I	3

The above components were agitated for about 30 seconds. Substantially all the water was converted to foam and the fibers were dispersed throughout. The foamed fiber dispersion was transferred to a paper forming box having the screen covered with about 2 inches of water. As in the case of Example I, a vacuum was applied and a uniform web of fibers was deposited on the screen. The wet web was removed from the screen and dried. The web was bulked by heating for 30 seconds at 200° C. between platens which were just touching the web, but no measurable pressure was applied by the platens. The dried web weighed about 4.3 ounces per square yard and had a rating +2 on the arbitrary scale for uniformity.

Example X

A non-woven web was prepared from the following components:

	Parts by wt.
Water	1000
"Triton"	5
Fiber (same as Example I)	3
Aqueous dispersion of .6 part of copolyester fibrids in 99.4 parts of water	25
	1033

The copolyester is the reaction product of sufficient ethylene glycol to combine with a mixture of 80 parts of terephthalic and 20 parts of isophthalic acids.

The above components were subjected to vigorous agitation to convert the water to a foam and disperse the fibers and copolyester fibrids throughout the foam.

A uniform non-woven web was formed from the foamed fiber dispersion in the same manner as described for Example I. The copolyester fibrids were uniformly distributed throughout the web and the heating step caused them to bond to the fibers. The dried product had the appearance of a bonded web. It was somewhat bulky and particularly suited for use as interliner between an outer and inner ply of garment. The final sheet contained about 5% copolyester binder and 95% fiber.

In place of the aqueous dispersion of the copolyester

used in the above Example X, aqueous dispersions of non-fibrillated polymeric materials can be used as the binder for the fibers in the water-laid web, including elastomers, such as, e.g., copolymer of butadiene and acrylonitrile, neoprene and natural rubber; resinous materials, such as, e.g., terepolymers of acrylonitrile, methacrylic acid and butyl acrylate as well as the other terepolymers disclosed in U.S. Patent 2,787,603, plasticized polyvinyl chloride, copolymers of vinyl chloride and vinylidene chloride.

Useful products are also obtained when fillers, such as, e.g., silica, mica and various coloring materials, such as pigments, are dispersed in the foam along with the fibers in the above example.

Example XI

The following composition was prepared:

	Parts by wt.
Polyvinyl chloride (Geon 101)	5.0
Di(2 ethyl hexyl) phthalate	2.8
Pigments and fillers	2.0
Epoxidized soya bean oil (Paraplex G–62)	2.9
Methyl ethyl ketone	87.3
	100.0

The above composition was prepared in the well known manner by grinding the pigments and fillers in the di(2 ethyl hexyl) phthalate and the epoxidized soya bean oil. The polyvinyl chloride was dissolved in the methyl ethyl ketone at about 60° C. The pigment-plasticizer mill base was thoroughly mixed in the polyvinyl chloride solution. One part of the polyvinyl chloride solution at about 60° C. was poured as a fine stream into about 40–50 parts of water at about 25° C. while the water was vigorously agitated. Upon entering the turbulent water, the fine stream of pigmented and plasticized polyvinyl chloride was rapidly precipitated from solution as fibrids. The fibrids were well frazzled and of irregular shape. By means of a 100 mesh screen, the fibrids were filtered from the mixture of water and methyl ethyl ketone. While still on the screen, the fibrids were washed with water until substantially free of methyl ethyl ketone.

To 1,000 parts of water were added 2 parts of "Triton" X–100, 72 parts of the water wet fibrids (23.6 parts dry fibrids) and 4.4 grams of crimped polyethylene terephthalate fiber ½" long and 3.5 denier per filament having 8–9 crimps per inch. The water containing the foaming agent, fibers and fibrids was vigorous agitated to convert the water to a foam with the fibers and fibrids dispersed throughout. The foamed fiber-fiibrid dispersion was poured into a paper forming box while a vacuum was being applied to the bottom of the box. The fibrids and fibers were immediately deposited uniformly on the screen in the form of a web the fibers and fibrids being uniformly distributed amongst each other. The web was removed from the screen and dried. The dry web was pressed for 3 minutes at 350° F. at a pressure of about 75–100 pounds per square inch to fuse or weld the fibrids together without destroying the fibrous character of the fibers.

The product was a fiber reinforced plastic sheet about 21 mils thick and weighing about 20 ounces per square yard. It was useful as an upholstery material.

Example XII

A polytetrafluoroethylene gasket material was made in accordance with the following procedure.

A foam was prepared by agitating 2,000 parts of water and 4 parts of "Triton" X–100 foaming agent. To the foam was aded 32 parts of polytetrafluoroethylene fibers ⅝ inch long and 6.0 denier per filament, and 64 parts of water wet beaten polytetrafluoroethylene fibers (46.0 parts dry). The above ingredients were vigorously agitated for about 1 minute to disperse fibrous materials throughout the foam.

The beaten polytetrafluoroethylene fibers can be prepared by forming a suspension or paste of polytetrafluoroethylene and extruding it in the form of a filament rod or tube which is 1/16" to 1/8" in diameter. During this operation, fibers are formed in the extruded material. The extruded polytetrafluoroethylene is then cut into small lengths of, e.g., .25 to 1.0 inch in length and then worked in a "micropulverizer," hammer mill, shredder or similar device to break up the extruded material into finer fibers. A preferred process for producing the beaten polytetrafluoroethylene fibers is described in greater detail in copending application S.N. 426,041, filed April 27, 1954, by Edward F. Harford, now abandoned. The foamed dispersion was poured into a paper forming box with vacuum applied. A web composed of 41% polytetrafluoroethylene fibers and 59% polytetrafluoroethylene beaten fibrous binder was formed on the screen. The unbeaten fibers and beaten fibrous material were uniformly distributed amongst each other. After drying, the web was subjected to pressure of about 75–100 pounds per square inch at 350° F. for 15 minutes. The pressed sheet was about 42 mils thick and weighed about 55.4 ounces per square yard.

The pressed sheet was ideally suited for use as a gasket material where resistance to high temperatures and corrosive chemicals is required.

The amount of fibrous material dispersed in the foam is not critical; usually, however, concentration ranging from .01 to 10% are particularly useful in practicing this invention. However, the length of fiber will control the preferred concentration depending upon the degree of uniformity required for the final product. In the case of short fibers, i.e., less than 1/4 inch in length, concentrations greater than 10% fiber in the foam are useful in carrying out this invention.

The type of foaming agent is not critical. Satisfactory results have been obtained by using proprietary products other than "Triton" X–100, such as, e.g., "Ivory" soap chips; liquid foaming agents, such as, e.g., "Joy" and "Lux"; and solid foaming agents, such as "Fab," "Ad," and "Tide." Any foam producing surface active agent, such as, for example, those disclosed in the book entitled "Encyclopedia of Surface-Active Agents" by J. P. Sisley et al., published 1952 by Chemical Publishing Company, Inc., can be used in carrying out this invention. The selection of the foaming agent whether anionic, cationic or non-ionic for carrying out this invention is dependent upon its foam forming property. It will be obvious to those skilled in the art that the fluidity of the foam can vary over very wide limits, the only limitation being that the foam must be fluid enough to drain or be sucked through a screen fine enough to retain the fibrous materials. Best results are obtained when all the water is converted to foam.

It is to be understood that any of the conventional paper making machines can be used in practicing this invention, such as, e.g., Fourdrinier machines, continuous rotary paper making machines, or simple vacuum filters equipped with fabric, wool-felt or wire screen filters.

This invention is not limited to any particular type of fiber. Fibers other than those mentioned in the specific examples which are useful in carrying out the invention include various other synthetic fibers, such as, e.g., those derived from monochlortrifluoroethylene polymers, copolymers of tetrafluoroethylene and hexafluoropropene, cellulose acetate, glass, asbestos and metals.

One of the outstanding advantages of the present invention is that it permits the formation of uniform water-laid webs from staple fibers at least twice as long as those employed in conventional prior art paper making techniques. In the case of prior art conventional techniques, it has been difficult and somewhat impractical to make uniform water-laid webs from fibers having a length greater than about .5 to .75 inch. In practicing this invention, there is no particular lower limit to fiber length, useful products can be obtained from fibers as short as .01 inch in length. The upper limit of fiber length is dependent upon the denier (density) and stiffness of the fiber. For example, for a .1 denier fiber, the length can be as high as 1/4 to 1/2 inch and for 15 denier fiber, the length can be 1.5 to 2.0 inches or higher depending upon the degree of agitation and type of agitation. The following table illustrates the preferred fiber length for the denier indicated:

Denier	Fiber length, inches
0.5	1/8
1.5	1/4
3.0	3/8
6.0	5/8
15.0	3/4

Also, the present invention permits the formation of uniform water-laid webs made from crimped fibers, which has not been possible by prior art techniques. Another outstanding advantage is that only a fraction of the time required for dispersing fibers in liquid water by conventional processes is required in practicing this invention. A still further advantage is that much less water is required to disperse a given weight of fibers by the process of this invention than by prior art processes. Another outstanding advantage of the present invention is that it permits the formation of water-laid fibrous webs in which the fibrous materials are in a three dimensional arrangement, i.e., less planar or laminar than water-laid webs made by prior art techniques.

While there are above disclosed but a limited number of embodiments of the structure, process and product of the invention herein presented, it is possible to produce still other embodiments without departing from the inventive concept herein disclosed, and it is desired therefore that only such limitations be imposed on the appended claims as are stated therein, or required by the prior art.

The embodiments of the invention in which an exclusive property or privlege is claimed are defined as follows:

1. The process which comprises forming a fluid dispersion of hydrophobic fibers at least about one quarter inch in length in a foam substantially free of a liquid phase and forming a non-woven web from said dispersion.

2. The process of claim 1 in which the fibrous material comprises nylon.

3. The process of claim 1 in which the fibrous material comprises polyacrylonitrile.

4. The process of claim 1 in which the fibrous material comprises polyethylene terephthalate.

5. The process of claim 1 in which the fibrous material comprises crimped staple fibers.

6. The process of claim 1 in which said fibrous material comprises a fluorohydrocarbon polymer.

7. The process of claim 1 in which said fibrous material comprises a tetrafluoroethylene polymer.

8. The process which comprises forming a fluid foam comprising water, a foaming agent and hydrophobic fibers at least about one quarter inch in length, said foam being substantially free of a liquid phase, and separating said fibrous material in the form of a web from said foam.

9. The process which comprises forming a fluid foam comprising water and a foaming agent, dispersing hydrophobic fibers at least about one quarter inch in length in said foam, said foam being substantially free of a liquid phase, applying said fiber containing foam to a porous medium, filtering said foam through said porous medium to deposit a web of said fibers thereon, and removing said web from said porous medium.

10. The process which comprises dispersing hydrophobic fibers at least about one quarter per inch in length in water in the presence of a foaming agent, foaming

substantially all the water, and separating said fibers in the form of a web from said foam.

11. The process which comprises dispersing hydrophobic fibers at least about one quarter inch in length and a non-fibrous particulate polymeric material in an aqueous foam substantially free of a liquid phase, separating a non-woven web from said foam, heating said web at a temperature above the fusion temperature of said particulate material and below the fusion temperature of said fibrous material and pressing said web.

12. The process of claim 11 in which the non-fibrous polymeric material comprises an elastomeric material.

13. The process of claim 11 in which the non-fibrous polymeric material comprises a resinous material.

14. The process which comprises dispersing hydrophobic fibers at least about one quarter inch in length and a non-fibrous particulate material in an aqueous foam substantially free of a liquid phase, and forming a web from said foam.

15. The process which comprises forming a foamed fluid dispersion comprising hydrophobic fibers at least about one quarter inch in length and fibrids, said dispersion being substantially free of a liquid phase, and forming a non-woven web from said dispersion.

References Cited in the file of this patent

UNITED STATES PATENTS

1,740,280	Bryant	Dec. 17, 1929
1,821,856	Spafford	Sept. 1, 1931
1,841,785	Bryant	Jan. 19, 1932
1,870,279	Bryant	Aug. 9, 1932
1,966,437	Bryant	July 17, 1934
2,260,557	Burton	Oct. 28, 1941
2,273,313	Clapp	Feb. 17, 1942
2,481,959	Wahlin	Sept. 13, 1949
2,626,213	Novak	Jan. 20, 1953
2,626,214	Osborne	Jan. 20, 1953
2,730,479	Gibson	Jan. 10, 1956
2,810,646	Wooding et al.	Oct. 22, 1957

OTHER REFERENCES

Casey: "Pulp and Paper," vol. I, Interscience Publishers, N.Y., 1952, page 364.