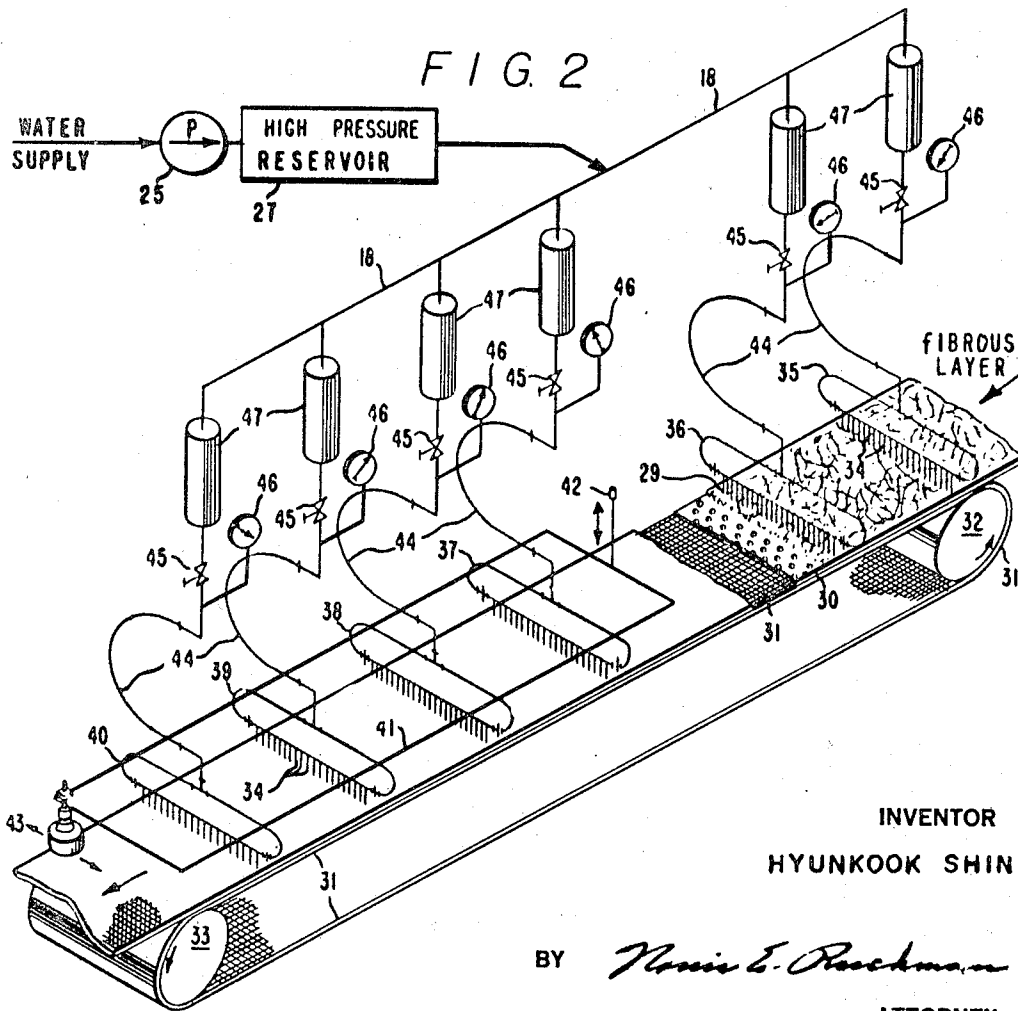
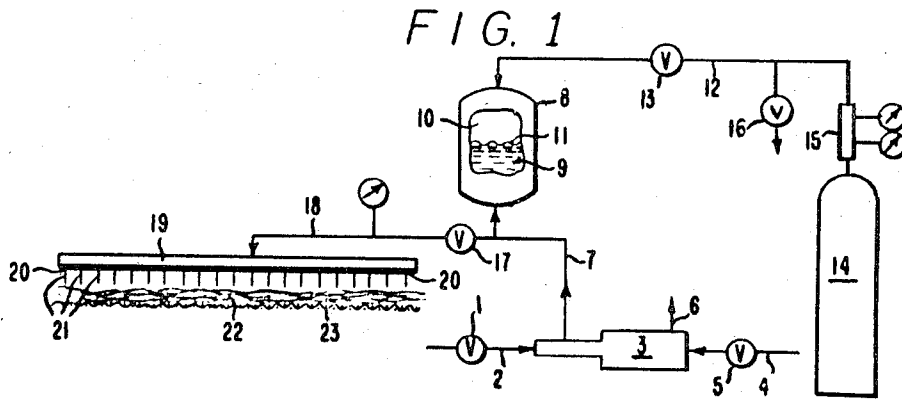


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HYUNKOOK SHIN
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STREAM OF POLYMER SOLUTIONS
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PRODUCTION OF NONWOVEN FABRICS WITH JET STREAM OF POLYMER SOLUTIONS

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Continuation-in-part of application Ser. No. 575,674, Aug. 29, 1966. This application June 30, 1967, Ser. No. 656,616

Int. Cl. D04h 3/12

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4 Claims

ABSTRACT OF THE DISCLOSURE

Improvements are disclosed in production of nonwoven fabrics directly from a layer of textile fibers or filaments by high-energy treatment with fine liquid jet streams to entangle the fibers into a strong structure. In the improved process the liquid is an aqueous solution of a polymer having a molecular weight of at least 10,000. Preferably a 0.5% aqueous solution of the polymer has a relative viscosity (RV) of at least 2.0 at 25° C. Other concentrations of polymer can be used, provided that the solution has at least 1.2 RV; preferably it is less than 20 RV. Aqueous solutions of polyethylene oxide, polyacrylamide or polyvinylpyrrolidone are shown to be much more effective than water alone for entangling fibers to produce strong, stable nonwoven fabrics. Fabrics of double the basis weight are successfully produced. For fabrics which are successfully produced with jet streams of water, the use of polymer solution provides a notable reduction in the total treatment energy required for the same fabric stability, e.g., as evaluated by resistance to pilling in use. The solutions also make it possible to process heavier denier material than can be processed successfully with water alone.

CROSS-REFERENCE TO RELATED APPLICATION

This is a continuation-in-part of my application Ser. No. 575,674, filed Aug. 29, 1966, now abandoned.

This invention relates to the production of nonwoven fabrics by treatment of fibrous layers with jet stream of liquid, and more particularly to jet treatments of fibrous layers with liquid solutions which provide important improvements over previous processes such as that disclosed in Evans application Ser. No. 550,209 filed May 16, 1966, and assigned to the assignee of the present application.

Although many patents have disclosed that layers of paper fibers or the like can be treated with low pressure water streams to rearrange the fibers, that result has been achieved by a mere washing action. More recently Guerin U.S. Patent No. 3,214,819, dated Nov. 2, 1965, discloses that liquid jet streams from needle-type jet devices can be used to pull fibers through a layer of fibrous material, in a manner analogous to the action of barbed needles in a needle loom, to produce felt-like nonwoven fabrics from a variety of natural and synthetic textile staple fibers.

The Evans application discloses a remarkable jet stream treatment for converting layers of textile fibers or filaments directly into patterned nonwoven products having a

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degree of durability, flexibility and stability of structure previously found only in woven or knitted fabrics. A wide variety of novel and useful products are disclosed, including many which closely resemble woven or knitted fabrics in appearance. In the process, water is jetted at a sufficiently high pressure, and impinged on the fibrous layer at a sufficiently high energy rate per unit area, to produce a unique form of fiber entanglement defined therein as tanglelacing. When a fibrous layer is supported on an apertured plate or screen and an adequate amount of treatment energy is applied by traversing the layer with such streams, the fibers are arranged into a pattern determined by the support and are locked into place by tanglelacing to form a strong, patterned nonwoven fabric.

The treatment energy expended during one passage of a fibrous web under a plurality of like jet streams in the preparation of a given nonwoven fabric, in horsepower hours per pound of fabric, may be calculated from the formula:

$$E_1 = 0.125 (YPG/sb) \text{ H.P.-hr./lb.,}$$

where:

Y=Number of streams per linear inch of treatment width,
P=Pressure at which the liquid is jetted in pounds per square inch gage (p.s.i.),

G=Average volumetric flow for one jet stream in cubic feet per minute,

s=Speed of passage of the web under the streams in feet per minute, and

b=The basis weight of fabric produced in ounces per square yard.

The total amount of energy expended in treating the web is the sum of the values calculated as above for each pass under such streams, if there is more than one pass. The value of G for use in the above formula can be determined from flow rate measurements. Evans teaches that treatment energy of at least 0.2 and preferably greater than 1 H.P.-hr./lb. of fabric should be used. The present invention permits the use of even lower energy when the web is particularly responsive.

The energy should also be applied to the fibers at a high rate per unit area (high energy flux). The energy flux of a stream, in foot-poundals per square inch per second, can be calculated by means of the formula:

$$EF = 77 PG/A \text{ ft.-poundals/in.}^2 \text{ sec.}$$

where P and G are as defined above, and A is the cross-sectional area of the streams in square inches, at a location just prior to impact with the fibrous web. This area can be determined from photographs of the stream with the web removed, or by means of micrometer probes. The maximum concentration of energy is obtained when A is substantially equal to the cross-sectional area of the stream immediately after formation in the jet device, which corresponds to the product of the area of the jet opening times the discharge coefficient. Evans teaches that the energy flux should be at least 23,000, and preferably at least 100,000 ft.-poundals/in.² sec.

Preferably the jet device produces a stream which is approximately cylindrical in shape, or is columnar with a low divergence angle. Drilled orifices are economical jet

devices, but should be carefully made for high efficiency. Evans discloses that streams suitable for tanglelacing are produced with such orifices of 0.003 to 0.030-inch diameter by jetting water at a pressure of at least 200 p.s.i. and preferably 500 to 5,000 p.s.i. However, other jet devices are suitable which provide an energy flux of at least 23,000 ft.-poundals/in.² sec. under the conditions of use.

In spite of the excellent properties of nonwoven fabrics made by treatment with water jets, there have been limitations associated with this process which, for example, required in some cases the use of excessive amounts of energy for desired products, or which placed an upper limit on the thickness of fibrous layers which might be processed, or which caused in certain high basis weight fabrics an incompleteness of processing at the lower, downstream, planes of the fabric. Moreover limitations were found in the size (denier) of filaments which might successfully be processed by the use of water jets.

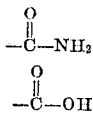
It has now been found that the use of certain aqueous polymer solutions, containing a fraction or very small percentage of the polymeric material in solution, will greatly extend the range of basis weights of fibrous layers which can be treated; double the basis weight or more may be processed successfully. Moreover at a lower range of basis weight, where either water or a polymer solution of this invention may be employed, a notable reduction (say, 50% reduction) is possible in total energy needed for a given degree of fabric stability (e.g., resistance to pill formation during use) when the solutions of this invention are used. This permits more economical production of these fabrics. Furthermore, a more uniform degree of fiber rearrangement is obtained with the said polymer solutions, so that stronger and also softer and more pliant fabrics may be made than is possible with previously used liquids. In addition, heavier denier fibrous material may be processed than with water alone. Other advantages of the present invention will become apparent from the subsequent discussion and examples.

In the process of the present invention, the fibers or filaments of fibrous layers are entangled to form strong nonwoven structures by treatment with jet streams of a liquid solution containing from 0.01% to 10% by weight of a polymer preferably having a molecular weight of at least 10,000. The liquid solution is jetted under pressure to form fine liquid streams having over 23,000 energy flux at the treatment distance, the streams are impinged against the fibrous material in a predetermined arrangement and the treatment is continued until fibers are highly entangled in treated portions of the fibrous material.

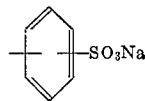
Preferably the liquid solution is an aqueous solution of a polymer selected from the class consisting of: (I) polyethers, and (II) substituted polyhydrocarbons consisting essentially of



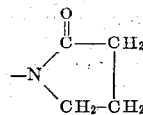
structural units wherein X is a hydrophilic group of the class



and salts thereof,



and



Suitable polyethers include poly(ethylene oxide), poly(propylene oxide) and their copolymers.

Suitable hydrophilic-substituted polyhydrocarbons include poly(acrylamide), poly(acrylic acid), poly(sodium acrylate), poly(sodium styrene sulfonate), poly(vinyl pyrrolidone) and copolymers of the monomers of the above.

The best results are obtained with fibrous webs having a basis weight of at least 2 ounces/yd.².

The process can be used for preparing textile-like nonwoven fabrics or felt-like nonwoven fabrics, for seaming nonwoven fabrics together, and for cutting layers as they are being joined. It is most useful for preparing strong tanglelaced nonwoven fabrics, with or without a patterned configuration.

Tanglelaced structures are characterized by the presence of localized regions of fibers which are randomly entangled and interlocked with each other to provide strength and coherency to the structure. In these tanglelaced regions there is a high concentration of fibers and/or fiber segments which are in a tightly packed, highly interentangled relationship wherein fibers randomly turn, wind, twist back-and-forth and pass about one another in all three dimensions of the structure, both individually and severally, so as to be virtually inseparable. Tanglelacing can be evaluated numerically in several ways. For the purpose of the present invention a tanglelaced structure is defined as having an entanglement frequency (*f*) of at least 20 per inch with an entanglement completeness (*c*) of at least 0.5, when tested as described immediately prior to the examples.

In the drawings, which illustrate equipment for use in the invention,

FIGURE 1 shows a schematic view of one type of apparatus for carrying out the process of the invention, and

FIGURE 2 is a schematic isometric view of an apparatus for continuous processing of fibrous material.

The formation of suitable jet streams has been discussed above. During treatment the jet streams are directed preferably vertically, against the web or other fibrous layer as it is conveyed beneath the jet devices. As the streams enter the web surface, the flow is interrupted by the fibrous material and the energy of the stream is, in part, absorbed in rearranging fiber elements to produce the desired tanglelaced structure. The result may be that the fiber entanglement at the surface and for a distance into the web is high or entirely satisfactory but, as the energy flux in the jet stream diminishes, there may come a level in the fabric where insufficient kinetic energy remains in the stream to entangle the fibers. At this level and below this level the desired tanglelaced structure will not be obtained.

There exists, therefore, for any conditions of pressure, energy flux and treatment energy a maximum web thick-

ness or basis weight which may be formed into a tangled product. There is also an intermediate range of conditions wherein a tangled product results but in which less than maximum entangling is present, and a fabric of relatively low durability or prone to undesirable surface distortion may result.

By increasing the water pressure, or prolonging the jet treatment, or possibly by using a different jet device, one is able to increase the maximum web thickness which may be processed but these also reach a limit and, moreover, the cost of the treatment may increase to an undesirable level.

According to the present invention, the effectiveness of the jet streams are very largely increased by employing a treatment liquid, usually aqueous, having in solution certain types of polymeric materials in relatively low concentration, e.g., 0.01% to 10% by weight of the treatment liquid. The result is a surprisingly great increase in effectiveness for entangling fibers.

A wide variety of polymers may be used. Preferably such polymers should have a molecular weight sufficiently high that a 0.5% aqueous solution (freshly made) has a relative viscosity of at least 1.1 at 25° C. The relative viscosities of the polymer solutions usually decrease with use in the process of this invention; presumably due to degradation by the high shear. Used solutions having relative viscosities as low as 1.2 are still superior to water.

The concentration to be used will depend upon the chemical nature of the polymer and its molecular weight. Thus extremely high molecular weight polyethylene oxide may be used at concentrations as low as 0.01% while a low molecular weight specie of the same polymer may be used at a concentration of 10% or more. In general, the polymer solution should have a relative viscosity of from about 1.2 to 100 and preferably less than about 25.

In one aspect of this invention, a fibrous web of high basis weight up to 20 or 30 or more oz./yd.² is treated, while supported on a relatively fine screen, with jetted polymer solutions of this invention. Pressures used may vary from 200 p.s.i., or lower, to 2,000 p.s.i., or more. Usually, because of the remarkable efficacy of these solutions in rearranging filaments, the range of pressure chosen may be kept somewhat lower than in processes using water as the treatment liquid. Well-entangled structures of high basis weight are obtained in which the fibers are tangled at all levels. In general, they are patterned only by the relative motion of the jet stream and of the fibrous web. The great range in basis weight and the advantages of the presently claimed solutions over water as a jet liquid are clearly shown in Example I. A wide range of products of superior physical properties may be produced by this invention which could not be successfully made at all by prior jet stream processes.

It is usually desirable to rinse out the residual polymer solution from these products after treatment, since solid polymeric material, when present in the dried nonwoven fabric, tends to stiffen the product. However, the polymer may increase the strength and at least raise the modulus of the fabric, especially in the case of fabrics treated with a low level of power input. If these qualities are desirable for the intended use, the polymeric solution may be left in the fabric.

Of course, size or binder materials may be added during or after jet treatment, which may or may not, as desired, be chemically aftertreated to produce a resin bonded structure, and so enhance or modify the properties of the jet treated product.

The fine mesh-supporting screens, used for this process embodiment, impart a relatively smooth or lightly patterned surface to the fabric on the side next to the screen. The main patterning of these products of high basis weight is that of the jet stream pattern on the up-

stream side, a desirable pattern for many uses since it gives a pleasing hand and appearance and provides a surface of remarkable durability.

Of course, the treatment of this invention may be applied to both sides of a web by having jet streams impinge from both sides or by "flipping" the web between successive treatments. Such treatment will, in general, effectively increase the durability of the fabrics on both surfaces and tend to mask the difference between "upstream" and "downstream" appearance of the fabrics.

While the examples compare the use of water with the use of aqueous solutions of this invention, it is to be understood that a similar comparison of a solvent other than water, for example, kerosene or cyclohexane with solutions of polymers dissolved in this other exemplified solvent will similarly show the benefits of this invention. The examples illustrate the use of aqueous solutions merely as being most convenient and in general most economical of the various possible solvent systems which might be used.

In another embodiment of this invention illustrated in Examples II through V and VII-IX, fibrous webs of relatively low basis weight, which may be successfully processed with water streams, are instead treated with solutions of the present invention at low or moderate levels of energy. Under these conditions notably better products in terms of surface stability (pill rating) after repeated wash cycling and/or notably higher tensile strengths are obtained by use of the presently claimed solutions than with water. If equally high levels of properties are desired with water-only treatment, considerably more energy (higher pressures or longer time of treatment) is necessary or, indeed, in some conditions, the properties would not be attainable with water regardless of the energy levels employed. This illustrates the economic advantages (in cost and rate of production) attributable to the practice of the present invention over the use of water for products of low and medium basis weight.

Example V shows the advantage of the presently claimed polymer solutions when the fibrous web contains no fibers below about 1½ inches in length. Such webs may be processed to good fabrics with water alone on 30 x 30 mesh screens at a higher energy level than shown for item *k* of the example. At the level of energy shown, much better surface stability is found when the polymer solution is used than is possible with water as the treating fluid. On the fine screen support used in item *m* of the example, it is very difficult to produce fabrics of really excellent durability with water alone even at the high energy level (although the fabrics of item *n* were amply strong and durable for many purposes). Use of the polymer solution at the fairly high energy level chosen produces excellent and surface-stable fabrics even at the low basis weight, and under the disadvantage of having no fibers shorter than 1½ inches included in the starting web.

The very efficient fiber rearrangement and energy conservation under treatment conditions ascribable to the polymer solutions of this invention provides stable well-entangled nonwoven fabrics at liquid pressures considerably lower than would be possible when using water alone.

Other advantages are illustrated for use of the polymer solutions of this invention. When paper products or webs of short staple fiber are treated the product is more efficiently tangled, softer and more flexible when produced with the polymer solutions than with water. It has also been found that webs treated on fine mesh screens give less trouble due to wash-away, i.e., massive fiber displacement, since they "tack down" more readily with the polymer solutions than with water.

Another advantage in the practice of this invention is in the jet entanglement of heavy denier fibers (Example VI). Fibers of 3 d.p.f. (denier per filament) entangle with difficulty when jet treated with water alone, requir-

ing rather high amounts of energy to attain a truly tangled structure. Even greater difficulty is encountered when, for example, fibers of 6 denier per filament are used. These coarse fibers may be entangled much more readily in accordance with the present invention, as shown in the example. The fiber entanglement frequency and completeness attained by treatment with the polymer solution of this invention is found to be within the limits characterizing a tangled product when 2.5 H.P.-hr./lb. energy is used.

When using water as the treatment liquid, neither this amount of energy nor up to three times as much energy will produce as high a level of entanglement frequency or completeness. The density and also fabric tenacity are likewise higher for this example than for the control product prepared by water treatment, even when using three times the energy for the latter product.

The efficient manner in which the presently claimed polymer solutions operate to rearrange fibers in webs may be further utilized for hydraulic stitching and seaming. A single jet or a small group or line of jets, when moved along a desired line with respect to a fibrous assembly, or held stationary (except for in-line oscillation) to define a line in the fibrous assembly (for instance, a sandwich of previously made tangled fabrics), may be used to stitch the assemblage together by entangling fibers from the top layer into and through the lower layers. Although similar results may be obtained on thin webs with water jet streams, for reasons already discussed, more efficient seaming is possible when using these solutions to make efficient sewing of a heavy web assembly possible. Simultaneous seaming and cutting of fibrous layers also may be done by this invention.

Under other conditions, the effective rearrangement by means of jets of these polymer solutions will result in forcing fiber ends from the fibrous web out on the downstream side so that a highly napped or fur-like fabric results. While this effect is to a degree characteristic of the downstream side of all hydraulically jet treated fabrics, especially those treated from one side only, the extent of pile or nap formation is increased markedly when the instant polymer solutions are used because of the more effective fiber displacement quality of these solutions. Such fabrics may be used as a "shaggy" pile fabric or sheared to uniform nap length.

Felt-like tangled fabrics of extremely high or even unlimited basis weight may be produced by a special technique of repeatedly adding web layers onto an initial jet treated structure. This is readily attained by use of the presently claimed polymer solutions. In order to approximate the same structure when using water alone (because of its less efficient fiber entangling action) three times the energy and many more passes and successive additions of smaller increments of fiber web or treatment operations are required. This again illustrates the marked improvement in process economy obtained by use of the present invention.

EQUIPMENT

A relatively simple form of equipment for treating fibrous webs with water at the required high pressure is illustrated in FIGURE 1. Water at normal city pressure of approximately 70 pounds per square inch (p.s.i.) (4.93 kg./cm.²) is supplied through valve 1 and pipe 2 to a high-pressure hydraulic pump 3. The pump may be a double-acting, single-plunger pump operated by air from line 4 (source not shown) through pressure-regulating valve 5. Air is exhausted from the pump through line 6. Water at the desired pressure is discharged from the pump through line 7. A hydraulic accumulator 8 is connected to the high-pressure water line 7. The accumulator serves to even out pulsations and fluctuations in pressure from the pump 3. The accumulator is separated into two chambers 9 and 10 by a flexible diaphragm 11. Chamber 10 is filled with nitrogen at a pressure of one-third to two-thirds of the desired operating water pressure and chamber 9 is

then filled with water from pump 3. Nitrogen is supplied through pipe 12 and valve 13 from a nitrogen bottle 14 equipped with regulating valve 15. Nitrogen pressure can be released from system through valve 16. Water at the desired pressure is delivered through valve 17 and pipe 18 to manifold 19 supplying orifices 20. Fine, essentially columnar streams of water 21 emerge from orifices 20 and impinge on the loose fibrous web 22 supported on apertured patterning member 23.

The streams are traversed over the web, by moving the patterning member 23 and/or the manifold 19, until all parts of the web to be treated are patterned and tangled at high energy flux. In general, it is preferred that the initial fibrous layer be treated by moving patterning layer 23 under a number of fine, essentially columnar streams, spaced apart across the width of the material being treated. Rows or banks of such spaced-apart streams can be utilized for more rapid, continuous production of tangled fabrics. Such banks may be at right-angles to the direction of travel of the web, or at other angles, and may be arranged to oscillate to provide more uniform treatment. Streams of progressively increasing energy flux may be impinged on the web during travel under the banks. The streams may be made to rotate or oscillate during production of the patterned, tangled fabrics, may be of steady or pulsating flow, and may be directed perpendicular to the plane of the web or at other angles, provided that they impinge on the web at sufficiently high energy flux.

Apparatus suitable for use in the continuous production of tangled, patterned fabrics in accordance with the present invention is shown schematically in FIGURE 2. A pump 25, which may be one of the types used for supplying water to high pressure steam boilers, is used to provide liquid at the required pressure. The liquid is stored in high pressure reservoir 27 until used. A fibrous layer 29, prepared by conventional means such as a card machine or Rando-Webber air-laydown equipment, is supplied continuously to a moving carrier belt 31 of flexible foraminous material, such as a screen. The carrier belt may also be the patterning member or, as illustrated, an apertured patterning member 30 may be supplied with the fibrous layer so that changes can readily be made in the pattern. The carrier belt is supported on two or more rolls 32 and 33 provided with suitable driving means (not shown) for moving the belt forward continuously. Six banks of orifice manifolds are supported above the belt to impinge liquid streams 34 on the fibrous layer at successive positions during its travel on the carrier belt. The fibrous layer passes first under orifice manifolds 35 and 36, which are adjustably mounted. Orifice manifolds 37, 38, 39 and 40 are adjustably mounted on frame 41. One end of the frame is supported for movement on a bearing 42, which is fixed in position. The opposite end of the frame is supported on oscillator means 43 for moving the frame back and forth across the fibrous layer to provide more uniform treatment.

High pressure liquid is supplied from the reservoir to the orifice manifolds through pipe 18. Each manifold is connected to pipe 18 through a separate line which includes flexible tubing 44, a needle valve 45 for adjusting the pressure, a pressure gage 46, and a filter 47 to protect the valve and jet orifices from foreign particles. As indicated on the gages in the drawing, the valves are adjusted to supply each successive orifice manifold at a higher pressure, so that the fibrous layer 29 is treated at increasingly higher energy flux during travel under the liquid streams 34. However, the conditions are readily adjusted to provide the desired patterning and tangling treatment of different initial fibrous layers.

Entanglement frequency and completeness tests

In preferred tests, nonwoven fabrics are characterized according to the frequency (f) and the completeness (c) of the fiber entanglement in non-bonded fabric, as deter-

mined from strip tensile breaking data using an "Instron" tester.

Entanglement frequency (f) is a measure of the extent of fiber entanglement along individual lengths of fiber in the nonwoven fabric. The higher the value of (f) the greater is the surface stability of the fabric, i.e., the resistance of the fabric to the development of pilling and fuzzing upon repeated laundering.

Entanglement completeness (c) is the proportion of fibers that break (rather than slip out) when a long and wide strip is tested. It is related to the development of fabric strength. A completeness (c) rating of 1 means that all of the fibers are being utilized in the development of fabric strength.

The products of the present invention have an entanglement frequency (f) of at least 20 per inch and an entanglement completeness (c) of at least 0.5.

Entanglement frequency (f) and completeness (c) are calculated from strip tensile breaking data, using strips of the following sizes:

Strip width symbol	Strip width (in.)	"Instron" gauge length (in.)	Elongation rate (in./min.)
w_0 -----	0.8	0	0.5
w_1 -----	0.3	1.5	5
w_2 -----	1.9	1.5	5

For patterned fabrics, strips are cut in two directions: (a) in the direction of pattern ridges or lines of highest basis weight (i.e., weight per unit area), and (b) in the direction at 90° to the direction specified in (a). In unpatterned fabrics any two directions at 90° will suffice.

In cutting the strips from fabrics having a repeating pattern of ridges or lines of high and low basis weight, integral numbers of repeating units are included in the strip width, always cutting through the low basis weight portion and attempting in each case to approximate the desired widths (w_0 , w_1 , w_2) closely. Ten or more specimens are tested at each strip width, using an "Instron" tester with standard jaw faces and the gauge lengths and elongation rates listed above. Average tensile breaking forces for each width (w_0 , w_1 and w_2) are correspondingly reported at T_0 , T_1 , and T_2 . It is observed that:

$$\frac{T_1}{w_1} < \frac{T_2}{w_2} < \frac{T_0}{w_0}$$

It is postulated that the above inequalities occur because:

- (1) there is a border zone of width D at the cut edges of the long gauge length specimens, which zone is ineffective in carrying stress; and
- (2) with zero gauge length, fibers are clamped jaw-to-jaw and all fibers carry stress up to the breaking point, while with long gauge length, some poorly-entangled fibers slip out without breaking. The proportion of stress-carrying fibers is called entanglement completeness (c).

Provided that (D) is less than $\frac{1}{2} w_1$, then:

$$\frac{T_1}{w_1 - 2D} = \frac{T_2}{w_2 - 2D} = c \frac{T_0}{w_0}$$

and D and c are:

$$D = \frac{w_1 T_2 - w_2 T_1}{2(T_2 - T_1)} \quad c = \frac{T_2 - T_1}{2T_0}$$

From testing various specimens, it is observed that when (c) is greater than 0.5, the value

$$D/\sqrt{d/1.5}$$

where d is the effective fiber denier, is a measure of the average distance required for fibers in the fabric to become completely entangled so that they cannot be separated without breaking. This value is practically independent of fiber length. The reciprocal of the value is the entanglement frequency (f) per inch, i.e.,

$$f = (1/D)\sqrt{d/1.5}$$

If the fabric contains fibers of more than one denier, the effective denier (d) is taken as the weighted average of the deniers.

Both (c) and (f) are determined in both major directions as defined above, and the geometric means (i.e. the square root of one half the sum of the squares) are reported as the proper values. In any determination of (f), if (f) turns out to be negative this is equivalent to a very high entanglement frequency and (f)=100 per inch is taken as the value to be used. When (c) is less than 0.5, it has been found that (D) and hence (f) may be influenced by factors other than entanglement. Accordingly, when (c) is less than 0.5, calculation of (f) as described above is not meaningful.

Strip tensile strengths are determined on samples 2" long and 0.5" wide using an Instron testing machine at a rate of elongation of 50% per minute. The breaking force is normalized for the basis weight and reported as tensile strength to the nearest 0.1 unit. All samples are washed free of polymer and dried before testing.

Surface stability is determined by a laundering test. The test load consists of 6 industrial cotton wiping cloths (to provide a source of lint and to simulate normal washing conditions), and ten test samples. This load is washed in an agitator-type household washing machine, using a "cotton" or severe wash setting and a laundry detergent. The load is put through a complete wash, rinse and a damp dry cycle after which it is removed and dried for 20 minutes in a household dryer of the "tumble dry" type at maximum dryer temperature. The above procedure is repeated 15 times after which the sample is removed and subjectively rated for fuzz and pill resistance. A perfect rating is 5; the poorest is one. Ratings are reported for the downstream (i.e. side that faced the patterning screen) and upstream sides respectively.

Relative viscosity of a polymer solution is the ratio of the flow time for the polymer solution to the flow time for pure water in an Oswald-Cannon-Fenske type viscosimeter at 25° C.

EXAMPLE I

This example shows the use of this invention in entangling heavy webs.

The starting web of 3 ounces/yard² (101.7 g./m.²) basis weight is made by air deposition of an acrylic fiber of a blend of equal weight of 1.5 and 0.25 inch (3.8 and 0.63 cm.) staple (analyzing acrylonitrile/methyl acrylate/sodium styrene sulfonate 93.6/6/0.4) of 1.5 d.p.f.

Two thicknesses of the above web are placed on an 80 x 80 mesh screen (16% open area) and passed twice at a speed of 1 yard per minute (0.91 m./minute) under a row of substantially cylindrical, unbroken vertical jet streams of a liquid. The streams are produced by a row of funnel-shaped orifices spaced 20 per inch (per 2.54 cm.) located in a manifold about 2 cm. above the web. The treating liquid enters the cylindrical portion of the orifice 5 mils (0.13 mm.) in diameter and about 1 mil (0.025 mm.) long and exits as a stream from the frusto-conical portion which is 11 mils (0.28 mm.) long and has a diameter of 15 mils (0.38 mm.) at the exit edge of the cone. A pressure of 200 pounds/inch² (14 kg./cm.²) is first used. The web is inspected to see if the rearrangement of

fibers in the original, planar web has been accomplished on the bottom side of the web (facing the screen). If the rearrangement has not extended through the web the procedure is repeated with a fresh web at higher pressure using increments of about 200 p.s.i. (14 kg./cm.²) until the desired rearrangement is obtained or the maximum pressure attainable of 1,500 p.s.i. (105 kg./cm.²) is tried.

The above procedure is repeated starting with 3, 4, 5 or more plies of the original web and/or a 10 oz./yd.² (339 g./m.²) web to give higher basis weights.

Following are aqueous solutions used in addition to water as the jetting liquid. Molecular weights are those reported by the manufacturer.

Polymer	Molecular weight	Percent in solution	Relative viscosity
(A) Poly(ethylene oxide):			
A-1	600,000	0.5	3-4.3
A-2	50,000	3.0	6.3
A-3	14,000	10	6.2
(B) Poly(acrylamide)	3,000,000	0.5	3.3
(C) Poly(vinyl pyrrolidone)	360,000	2.0	8.7
(D) Poly(acrylamide/acrylic acid) Copolymer "Polyhail 295"		0.3	23

Results are shown in Table I. Under the conditions used it is seen that the maximum basis weight that can be entangled with water (item 1) at the maximum pressure of 1,500 p.s.i. (105 kg./cm.²) is 9 oz./yd.² (305 g./m.²). The use of polymer solutions in items 2-7 permits much higher basis weight webs to be entangled from top to bottom. Another advantage of the invention is that items 2-7 can produce equivalent results to water—where water is operable—at a significantly lower pressure, thus reducing the costs of the process.

The energy expended for the production of items 1-8 ranges from 0.04 to 0.6 H.P.-hrs./lb. [0.056 to 0.85 Cal./g. (kilogram-calories/g.)].

Similar results are obtained when the fine supporting screen used above is replaced with a coarser screen (as coarse as 3 x 3 mesh) or a perforated plate with the exception that the side of the product facing the screen is somewhat patterned and has an embossed appearance.

TABLE I

Item	Polymer	Percent in Solution	Minimum pressure required for basis weights below (p.s.i.)					
			6 oz./yd. ²	9 oz./yd. ²	12 oz./yd. ²	15 oz./yd. ²	21 oz./yd. ²	26 oz./yd. ²
1	None		500	1,000	1 X	1 X		
2	A-1	0.5	150	300		600		1,000
3	A-2	3.0	150	300		500	950	
4	B	0.5	200		900	1,100		
5	C	2.0	200			800	1,300	
6	D	0.3	300	600	1,000	1,050		
7	A-3	10	200	400	700	1,000		

¹ Could not be entangled even at 1,500 p.s.i. (105 kg./cm.²).

EXAMPLE II

Nonwoven fabrics are prepared using the starting web, polymer solution (A-1), screen and orifices of Example I by passing the web at 1 y.p.m. (0.91 m.p.m.) under the jets as follows:

- (1) 2 passes at 500 p.s.i. (35 kg./cm.²) with a 14 x 18 mesh top screen,
- (2) reverse the web,
- (3) 1 pass at 200 p.s.i. (14 kg./cm.²) with no top screen,
- (4) 1 pass at 1,000 p.s.i. (70 kg./cm.²) with the jets oscillating.

The results with polymer solution and with water are given in Table II as items *c* and *d*.

The above general procedure is repeated replacing the orifices with a similar design having a 7 mil (0.177 mm.) diameter cylindrical portion spaced 20/inch (20/2.54 cm.) and using a combination of pressures of 500 and 1,000 p.s.i. (35 and 70 kg./cm.²) to expend the desired energy. Results are given at items *a* and *b* in Table II.

At both energy levels it is seen that the use of the polymer solution affords a significantly more durable product than does the use of water under the same conditions.

The procedure for items *a* and *b* is repeated using lighter starting web and treating to different energy levels. A plot of energy consumed versus tensile strength of the product is made and the energy values interpolated to obtain a standard tensile strength as shown for items *e* and *f* on Table II. It is seen that the use of this invention decreases the energy consumption for a given property level by at least 50%.

EXAMPLE III

A web containing 65% of the 1.5" (3.8 cm.) acrylic staple fiber of Example I and 35% of 0.25" (0.63 cm.), 1.5 d.p.f. rayon staple fiber is entangled by passing on a 30 x 8 mesh screen (35% open area) at 1.5 yards (1.37 meters) per minute under jets of an aqueous solution of polymer A-1 of Example I using 7 mil (0.177 mm.) diameter orifices of Example 2 for a combination of treatments as given below to give item *g* of Table II. The procedure is repeated with the use of water as the jetting liquid to give item *h*. The superior integrity of item *g* over the *h* is evident.

- (1) 1 pass at 500 p.s.i. (35 kg./cm.²) with a 14 x 18 mesh screen on top,
- (2) Repeat 1 with no top screen and jets oscillating,
- (3) Reverse the web on the bottom screen,
- (4) Repeat 1 without a top screen,
- (5) 1 pass at 1,000 p.s.i. (70 kg./cm.²) with jets oscillating.

EXAMPLE IV

Webs of the composition of Example I are entangled by passing at 1.5 yards (1.37 meters) per minute on a 150 x 150 mesh screen (37% open area) under jets of an aqueous solution of polymer A-1 (Example I) from the 7 mil diameter orifice of Example II using the entangling schedule of Example III to make item *i* of Table II.

The procedure is repeated using water as the jetting liquid to make item *j*. The greatly improved tensile properties of item *i* versus *j* are also reflected in the superior surface stability.

EXAMPLE V

This example shows the use of all long staple on two different patterning screens.

Webs containing 100% of the 1.5" (3.8 cm.) acrylic staple fiber of Example I are entangled by passing on 30 x 30 mesh screens (27% open area) or 80 x 80 mesh screens (16% open area) at 1.5 yards (1.37 meters) per minute under jets of the polymer solution of Example IV from the 7 mil diameter orifices of Example II a number of times at 500 and 1,000 p.s.i. (35 and 70 kg./cm.²) to give items *k* and *m*. The procedures for items *k* and *m* are repeated using water to give items *l* and *n*. As shown in Table II the use of this invention under these circumstances gives moderate improvements in tensile strength and very large improvements in durability of the product as compared to samples made with water under the same conditions.

EXAMPLE VI

This example shows the use of higher denier fibers in the process.

The fiber used for items *o* and *p* is a self-crimping, bi-component acrylic fiber of a composition as shown in Example III of U.S. Patent No. 3,092,892 with a staple

length of 1.5" (3.8 cm.) and a denier per filament of 6.

A web of the fiber on a 40 x 40 mesh screen (21% open area) is passed at a speed of 2 yards (1.82 meters) per minute under jets of an aqueous solution of polymer A-1 (Example I) from the 7 mil diameter orifices of Example II for a number of treatments at different pressures up to a maximum of 1,500 p.s.i. (105 kg./cm.²) to give item *o* of Table II. The procedure is repeated exactly but using water to give item *p*.

Item *q* is made in a similar manner but using 3 d.p.f. fiber (of the chemical composition of Example I) in a lower basis weight web. Item *r* is made in the same manner with water and the treatment is repeated with water to give item *s*.

From Table II it is seen that use of this invention affords a superior product in tensile strength, frequency of entanglement, entanglement completeness and surface stability over products made with water at the same (or 3 times larger) energy consumption.

EXAMPLE VIII

This example shows the improvement in surface stability of a nonwoven fabric made with polymer solutions.

The starting web of Example VII is treated with the jets and treatment sequence of Example VII to give an energy input of 1.2 H.P.-hrs./lb. (1.7 Cal./g.) based on the average final weight of 2.5 oz./yd.² (85 g./m.²).

The surface stability ratings after 3 cycles of laundering are given in Table IV as items *a*, *b* and *c* for the different polymer solutions and water for two different patterning screens. The values for water treated products (*a*) are the average of 3 different preparations.

All samples are strong, having a strip tensile strength (average of both directions) of from 3.1 to 4.2 (lb./in.)/(oz./yd.²), [16.3 to 22.2 (g./m.)/(g./m.²)] for the different treatments.

TABLE II

Item	Process conditions				Product properties				
	Starting basis weight		Additive	Percent	Energy used		Tensile strength (MD)		Surface stability
	Oz./yd. ²	(G./m. ²)			H.P., hrs./lb.	(Cal./g.)	Lb./in./oz./yd. ²	(G./cm./g./m. ²)	
a	3	(102)	A-1	0.5	1.3	(1.8)			4.8/4.8
b	3		None		1.3				3.8/2.3
c	3		A-1	0.5	0.7	(1.0)			4.5/2.0
d	3		None		0.7				Destroyed
e	2	(67)	A-1	0.38	2.0	(2.8)	3.0	(15.7)	>3.5/3.5
f	2	(67)	None		4-5	(5.6-7.0)	3.0	(15.7)	≥3.5/3.5
g	3	(102)	A-1	0.5	1.3	(1.8)			4.3/4.8
h	3		None		1.3				2.0/2.5
i	2	(67)	A-1	0.38	2.0	(2.8)	4.3	(22.6)	5/5
j	2		None		2.0		1.9	(10.0)	2/2
k	2	(67)	A-1	0.38	2.7	(3.8)	4.0	(21)	4.0/4.5
l	2		None		2.7		3.7	(19.5)	1.5/2.0
m	2		A-1	0.38	4.7	(6.6)	4.7	(24.6)	4.5/3.0
n	2		None		4.7		3.7	(19.5)	2.3/1.8
o	6	(204)	A-1	0.5	2.5	(3.5)	4.0	(21)	30 0.92
p	6		None		2.5		2.8	(14.2)	17 0.79
q	5	(170)	A-1	0.5	2.9	(4.1)	5.2	(27.4)	38 0.99
r	5		None		2.9		4.1	(21.6)	20 0.96
s	5		None		8.7	(12.2)	4.1	(21.6)	30 0.92

EXAMPLE VII

This example illustrates the use of other polymers. The starting web is a random web of 3 oz./yd.² (101.7 g./m.²) basis weight made of the acrylic fiber/rayon fiber blend of Example III. The web is entangled by passing on an 80 mesh screen at 4 y.p.m. (3.66 m.p.m.) under jets of a liquid using 7 mil (0.177) diameter orifices of Example II for a combination of treatments as given below. The total treatment gives an energy input of 1.0 H.P.-hrs./lb. (1.4 Cal./g.).

Treatment

- (1) 1 pass at 1,000 p.s.i. (70 kg./cm.²) with a 14 x 18 mesh screen on top of the web,
- (2) repeat 1 with no top screen and jets oscillating,
- (3) reverse the web on the bottom screen,
- (4) repeat 1,
- (5) repeat 2.

Results obtained with solutions of polyvinyl pyrrolidone (of Example I) and water are shown as Items *a* and *b* of Table III.

The process is repeated using 500 p.s.i. (35 kg./cm.²) in treatment steps 1 and 4 to give an energy input of 0.67 H.P.-hr./lb. (0.94 Cal./g.). Results obtained with Poly(acrylamide) (of Example I), a commercial grade of poly(sodium styrene sulfonate) and water are given as Items *c-e*, respectively, in Table III.

TABLE III

Item	Polymer in solution	Percent in solution	Relative viscosity	Tensile strength (MD/CD)	
				Lb./in./oz./yd. ²	G./cm./g./m. ²
a	Poly(vinyl pyrrolidone)	2.0	6-6.5	4.8/5.2	25/27
b	None	0	1	3.9/4.8	20/25
c	Poly(acrylamide)	0.5	2.4-3.5	3.3/3.8	17/20
d	Poly(sodium styrene sulfonate)	0.5	2.9-7.3	3.2/3.8	17/20
e	None	0	1	2.4/2.1	13/11

EXAMPLE IX

The original webs of Example VIII are entangled by passing on an 80-mesh or 20-mesh screen at 0.9 y.p.m. (0.82 m.p.m.) under the jets of Example VIII for a combination of treatments as given below. The total treatment gives an energy input of 1.5 H.P.-hrs./lb. (2.1 Cal./g.) based on the average 2.0 oz./yd.² (68 g./m.²) weight of the products.

Treatment

- (1) One pass at 300 p.s.i. (21 kg./cm.²) with a 14 x 18 mesh screen on top of the web.
- (2) Repeat 1 with no top screen.
- (3) Repeat 2 with jets oscillating.
- (4) Reverse the web on the bottom screen.
- (5) Repeat 1.
- (6) Conditions of step 2 for 2 passes.

The surface stabilities after 2 laundry cycles are reported in Table IV as items *d*, *e* and *f*. Two distinct preparations were made with water for item *d*: Both samples made on the 80-mesh screen were destroyed by 2 launderings, and one sample made on the 20-mesh screen was destroyed by 2 launderings while the other had a surface rating of 1.0/1.0.

The products are strong, having a strip tensile strength (average of 2 directions) ranging from a low of 2.4 for water prepared samples to a high of 3.5 (lb./in.)/(oz./

yd.²), [12.6 to 18.5 (g./cm.)/(g./m.²)], for the solution treatments.

TABLE IV

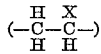
Item	Polymer in solution	Percent	Relative viscosity	Surface stability	
				20 x 20 screen	80 x 80 screen
a	None	0	1	1.9/2.9	1.4/2.6
b	Poly(ethylene oxide)	0.5	1.7	4.2/4.5	2.0/3.0
c	Poly(acrylamide)	0.5	3.0	4.0/4.5	3.5/4.7
d	None	0	1	1.0/1.0	(¹)
e	Poly(sodium styrene sulfonate)	1.5	8-17	2.7/4.0	3.5/4.7
f	Poly(acrylamide/acrylic acid) copolymer.	0.3	15-50	1.3/2.5	2.2/2.5

¹ Destroyed by test.

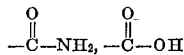
Since many different embodiments of the invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited by the specific illustrations except to the extent defined in the following claims.

I claim:

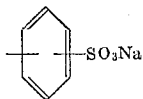
1. An improvement in the process for producing non-woven products by advancing a layer of fibrous material having a basis weight of at least 2 oz./yd.² and impinging a plurality of liquid jet streams upon the advancing layer at sufficiently high energy to cause intermingling and entanglement of fibers and form a self-coherent web, wherein the improvement comprises impinging said advancing layer with jet streams of an aqueous solution of a high molecular weight polymer selected from the class consisting of (I) polyethers and (II) substituted polyhydrocarbons consisting essentially of



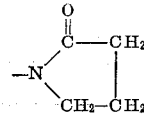
structural units wherein X is a hydrophilic group of the class



and salts thereof,



15 and



20

said polymer being present in an amount which provides an aqueous solution having a relative viscosity of 1.2 to 2.5 when determined at 25° C., and said polymer having a molecular weight such that a 0.5% aqueous solution of the polymer has a relative viscosity of at least 1.1 at 25° C.

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2. The process defined in claim 1 wherein the advancing layer of fibrous material is supported on a patterning member and is traversed with streams having over 23,000 energy flux at the treatment distance to form a patterned nonwoven fabric.

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3. The process defined in claim 1 wherein the denier per filament of said fibrous material exceeds 4.0.

35

4. The process defined in claim 1 wherein said layer of fibrous material has a basis weight of at least 15 oz./yd.² and is treated on a supporting screen with substantially cylindrical streams, jetted from orifices at a pressure of at least 1,100 pounds per square inch, to form a heavy weight product.

40

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