United States Patent [19]

Santacecilia et al.

[11] **3,751,290**

[45] **Aug. 7, 1973**

[54] NONWOVEN FABRICS

[75] Inventors: **Remo Santacecilia; Edwin C. Sherburne,** both of Wilmington, Del.

[73] Assignee: **ICI America Inc.,** Wilmington, Del.

[22] Filed: **June 18, 1971**

[21] Appl. No.: **154,685**

[52] **U.S. Cl.** **117/140 A,** 117/138.8 F, 117/138.8 N, 117/143 A, 117/161 KP, 117/161 LN

[51] **Int. Cl.** **B32b 27/12,** C09d 3/50

[58] **Field of Search** .... 117/140 A, 161 KP, 117/161 UA, 143 A, 138.8 F, 138.8 N, 161 LN

[56] **References Cited**

UNITED STATES PATENTS

2,955,095	10/1960	Gollob	117/140 X
3,000,757	9/1961	Johnston et al.	117/140 X
2,774,687	12/1956	Nottebohm et al.	117/140 X
2,709,694	5/1955	Dietrich et al.	260/69
3,412,072	11/1968	Bouboulis et al.	260/77.5
2,699,435	1/1955	Auten et al.	260/70 X
2,696,504	12/1954	Yost et al.	260/70 X
3,133,897	5/1964	Inaba et al.	260/77.5 X
3,054,777	9/1962	Inaba et al.	260/77.5
3,359,243	12/1967	Criner	260/77.5
3,021,307	2/1962	Csendes	260/72

Primary Examiner—William D. Martin
Assistant Examiner—Harry J. Gwinnell
Attorney—Kenneth E. Mulford et al.

[57] **ABSTRACT**

A process for binding nonwoven textiles by impregnating a nonwoven textile with a liquid application medium containing a polymer selected from the group consisting of

1. a polyurea polymer characterized by the generalized formula

$$\left[-\overset{R}{\underset{}{N}}-\overset{O}{\overset{\|}{C}}-\overset{R}{N}-\left(R'O\right)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-\right]_{p'}$$

wherein m is 0 or 1, q is an integer from 1 to 13, p' is an integer from 3 to 100, R' is an alkylene group containing from 3 to 5 carbon atoms, each R'' is independently hydrogen or methyl and each R is independently selected from the group consisting of H and CH_2OH, with the proviso that at least one R is CH_2OH and

2. polyureaurethane polymers characterized by the generalized formula $(A)_x$ wherein x is an integer from 2 to 100 and each A is independently selected from the group consisting of

$$-\overset{R}{N}-\overset{O}{\overset{\|}{C}}-\overset{R}{N}-\left(R'O\right)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-$$

and

$$-\overset{O}{\overset{\|}{C}}-\overset{R}{N}-\left(R'O\right)_m\left(CH_2CHR''O\right)_n-$$

wherein m is 0 or 1, n is an integer from 2 to 14, q is an integer from 1 to 13, R' is an alkylene group having from 3 to 5 carbon atoms, each R'' is independently hydrogen or methyl, and each R is independently selected from the group consisting of hydrogen and CH_2OH, with the proviso that at least one A is

$$-\overset{R}{N}-\overset{O}{\overset{\|}{C}}-\overset{R}{N}-\left(R'O\right)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-$$

and at least one A is

$$-\overset{O}{\overset{\|}{C}}-\overset{R}{N}-\left(R'O\right)_m\left(CH_2CHR''O\right)_n-$$

and with the further proviso that at least one R is a CH_2OH group.

7 Claims, No Drawings

NONWOVEN FABRICS

This invention relates to nonwoven fabrics and to processes for binding nonwoven textiles. More particularly, this invention relates to the use of improved binder resins for nonwoven fabrics.

Nonwoven fabrics are structures comprising random or oriented fibers held together by adhesives. Nonwoven fabrics have achieved considerable success in the textile industry since they can be produced more rapidly and more economically than woven fibers. The preparation of nonwoven textile fabrics differs from the preparation of conventional textile fabrics in that no weaving is required. The nonwoven material is prepared by forming the textile fibers into a coherent sheet or web and then binding the fibers together to give the finished fabric. In order to impart adequate strength to the fabric, the fabric is impregnated with a liquid solution or emulsion containing a binding or adhesive agent. The adhesives or binders which have been employed for the preparation of nonwoven fabrics include polyvinyl alcohol, polyvinyl acetate, polyvinyl chloride, acrylic polymers and butadiene-acrylonitrile copolymers and butadiene-styrene copolymers.

In general, most binding agents impart adequate strength to the nonwoven textiles. However, nonwoven fabrics treated with such binders often display serious drawbacks, such as, color fastness, resistance to washing and dry cleaning, stiffness, and discoloration on treatment with heat, such as the heat applied during normal pressing or ironing. Accordingly, there is a need in the art for improved binders to impart improved softness and drape characteristics to nonwoven fabrics without sacrificing strength and the other properties mentioned above. Improved softness and drapeability in nonwoven fabrics are needed before they can compete successfully with woven and knit fabrics in many apparel items.

Accordingly, it is an object of this invention to provide nonwoven fabrics which exhibit a high degree of softness and excellent drapeability and still possess good textile strength, color fastness, and resistance to washing and dry cleaning.

It is an object of this invention to provide a process for the preparation of nonwoven fabrics having improved properties.

It is an object of this invention to provide a process for binding nonwoven textiles for the preparation of nonwoven fabrics having improved softness and drapeability without sacrificing properties such as tensile strength, color fastness, and resistance to washing and dry cleaning.

The foregoing art objects and still further objects are accomplished in accordance with the present invention by providing improved adhesives or binders for the preparation of nonwoven fabrics having improved properties. The binders are methylolated polyurea polymers and methylolated polyureaurethane polymers. The methylolated polyurea polymers may be characterized by the generalized formula

$$\left[-\overset{R}{\underset{|}{N}}-\overset{O}{\underset{\|}{C}}-\overset{R}{\underset{|}{N}}-\left(R'O\right)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-\right]_{p'}$$

wherein m is 0 or 1, q is an integer from 1 to 13, p' is an integer from 3 to 100, R' is an alkylene group containing from 3 to 5 carbon atoms, each R'' is independently hydrogen or methyl, and each R is independently selected from the group consisting of hydrogen and CH_2OH with the proviso that at least one R is CH_2OH. The methylolated polyureaurethane polymers may be characterized by the generalized formula $(A)_x$ wherein x is an integer from 2 to 100 and each A is independently selected from the group consisting of

$$-\overset{R}{\underset{|}{N}}-\overset{O}{\underset{\|}{C}}-\overset{R}{\underset{|}{N}}-\left(R'O\right)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-$$

and

$$-\overset{O}{\underset{\|}{C}}-\overset{R}{\underset{|}{N}}-\left(R'O\right)_m\left(CH_2CHR''O\right)_n-$$

wherein m is 0 or 1, n is an integer from 2 to 14, q is an integer from 1 to 13, R' is an alkylene group having from 3 to 5 carbon atoms, each R'' is independently hydrogen or methyl and each R is independently selected from the group consisting of hydrogen and CH_2OH, with the proviso that at least one A is

$$-\overset{R}{\underset{|}{N}}-\overset{O}{\underset{\|}{C}}-\overset{R}{\underset{|}{N}}-\left(R'O\right)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-$$

and at least one A is

$$-\overset{O}{\underset{\|}{C}}-\overset{R}{\underset{|}{N}}-\left(R'O\right)_m\left(CH_2CHR''O\right)_n-$$

with the proviso that at least one R is CH_2OH. The preferred compounds are those wherein substantially all of the R groups are CH_2OH.

Methylolated polyurea polymers and methylolated polyureaurethane polymers which may be used as binders in accordance with the present invention are described in detail in U. S. Pat. application Ser. No. 880,892, filed Nov. 28, 1969, the disclosure of which is specifically incorporated hereinto by reference. Briefly, the binders which are used in accordance with the present invention may be prepared by reacting urea with a composition comprising from about 10 to about 100 parts of a diamine and from 0 to 90 parts of an amino alcohol. The reaction of the urea with the diamine results in the formation of urea linkages whereas the reaction of the urea with the amino alcohol results in the formation of urethane linkages. Accordingly, if the reaction mixture is free of amino alcohol, the resulting polymer is a polyurea polymer whereas if the reaction mixture contains some amino alcohol, the resulting polymer is a polyureaurethane polymer. The methylol groups may be introduced into the polymers by reacting the aforesaid reaction products with formaldehyde, paraformaldehyde, or other convenient sources of formaldehyde. The reaction conditions used are those conventionally used in the art for methylolating active hydrogen containing compounds.

The polyurea polymers may be prepared by reacting about equal molar amounts of urea and a diamine at a temperature from about 140° to about 220° C. The diamine used may be any compound characterized by the generalized formula

$$H_2N\,(RO)_m(CH_2CHR''O)_n\,CH_2CHR''\text{-}NH_2$$

wherein m is 0 or 1, n is an integer from 2 to 13, R is an alkylene radical having from 3 to 5 carbon atoms, and each R'' is independently hydrogen or methyl. Illustrative examples of these diamines include triethylene glycol diamine, hexaethylene glycol diamine, bis-2-aminopropyl ether of diethylene glycol, and decaethylene glycol diamine.

The polyureaurethane polymers which are methylolated to prepare a binder in accordance with this invention may be prepared by reacting urea with substantially equal molar amounts of a mixture of an amino alcohol and a diamine at a temperature from 150° C. to about 220° C. The diamine used may be any of those described above. The amino alcohol used may be any amino alcohol characterized by the generalized formula $H_2N\ (RO)_m(CH_2CHR''O)_nH$ wherein m is 0 or 1, n is an integer from 2 to 14, R is an alkylene group having from 3 to 5 carbon atoms, and each R'' is independently hydrogen or methyl. Illustrative examples of these amino alcohols include triethylene glycol monoamine, diethylene glycol monoamine, tripropylene glycol monoamine, diethylene glycol monoamine, dipropylene glycol monoamine, pentaoxyethylene monoamine, pentaoxypropylene monoamine, polyoxyethylene (14) monoamine, polyoxypropylene (5) monoamine, dipropylene glycol propyl amine, diethylene glycol propyl amine, and decaoxyethylene pentyl amine. As pointed out above, the mixture of diamine and amino alcohol may contain from 10 to 100 percent by weight of diamine and from 90 to 0 percent by weight of the amino alcohol. The mixture of diamine and amino alcohol used may be the crude reaction product resulting from the ammonolysis of a glycol or by mixing together separately prepared diamine and amino alcohol.

The following examples illustrate the preparation of methylolated polyurea polymers and methylolated polyureaurethane polymers which may be used as binders for the preparation of non-woven fabrics in accordance with the present invention. Additional polymers may be prepared by substituting other monoamines and diamines for those used in the illustrative examples.

EXAMPLE A

Into a 300 ml. flask equipped with stirrer, thermometer, and receiver system, are added 37.6 grams of urea and 200 grams of a crude aminated triethylene glycol containing substantially equal molar proportions of triethylene glycol diamine and triethylene glycol monoamine. The mixture is then heated between 97° – 159° C. for 3 hours, between 159°–181° C. for 1 hour, and then between 181°–190° C. for 8 hours. The reaction product is terminally vacuum stripped at 15–20 mm mercury absolute. The resulting polyureaurethane polymer is a dark, viscous liquid having a molecular weight of about 1,500 and a Gardner Holt viscosity in meta-cresol of N(25 percent solution in 25° C.). To a solution of 5 grams of the polyureaurethane polymer in 63.9 grams of water are added 7.1 grams of 36.7 percent aqueous formaldehyde. The pH of the solution is adjusted to 8.5, and the solution is heated at 51° C. for 15 hours. The concentration of the resulting solution is then adjusted to provide an aqueous solution containing 34.3 percent by weight of the methylolated polyureaurethane polymer.

EXAMPLE B

74.5 grams (1.24 mols) of urea, 142.9 grams (0.62 mols) of Jeffamine D-231 (bis-2-aminopropyl ether of diethylene glycol) and 65.3 grams (0.62 mols) (diethylene glycol monoamine) are charged to a 3-necked, round-bottom flask provided with stirrer, thermometer, and reflux condenser vented through a dry ice trap. The mixture is then heated for 6 hours between 104°–175° C., 16 hours at 175° C., 4 hours at 195° C. and then terminally vacuum stripped for 2 hours at 195° C. at 15–20 mm mercury absolute. The product is a yellow oil containing 11.2 percent nitrogen. To a solution of 212 grams of this product and 424 grams of water are added 172 ml of 37 percent aqueous formaldehyde. The pH of the solution is adjusted to 8.5 and the solution allowed to react at room temperature overnight. The product is a clear yellow solution which becomes turbid above 30° C. The concentration of the resulting solution is adjusted to provide a 37 percent solution of the methylolated polyureaurethane.

EXAMPLE C

A mixture of 357 grams of bis-2-aminopropyl ether of diethylene glycol, 163.2 grams of diethylene glycol monoamine and 186.3 grams of urea is heated at 117°–200° C. for several hours to form a polyureaurethane polymer. The reaction mixture is then vacuum stripped at 195° C. and 0.3 mm mercury absolute. The resulting polyureaurethane is a clear amber solid. 1,247 grams of a 33 percent aqueous solution of the polyureaurethane and 369 grams of a 37 percent aqueous formaldehyde solution are mixed together and the pH adjusted to 8.5. The solution is then heated at 50° C. for 15 hours to form the methylolated polyureaurethane polymer. The concentration of the resulting aqueous solution is adjusted to 34 percent solids content.

EXAMPLE D

250 grams of a 33 percent solution of the polyureaurethane prepared in Example C is contacted with 54.7 grams of 37 percent aqueous formaldehyde solution. The pH of the solution is adjusted to 8.5, and the solution is heated at 50° C. for 15 hours. The concentration of the resulting solution is adjusted to have 36.6 percent by weight of the methylolated polyureaurethane polymer.

EXAMPLE E

200 grams of Jeffamine D-230 (bis-2-aminopropyl ether of diethylene glycol), 200 grams of diethylene glycol monoamine, and 166.5 grams of urea are charged to a 3-necked, round-bottom flask provided with a stirrer, thermometer, and reflux condenser vented through a dry ice trap. The mixture is then heated for 1 hour at 108°–150° C., 8 hours at 155°–200° C., and then terminally vacuum stripped for 2 hours at 195° C. and 15 mm mercury absolute. The resulting polymer is then reacted with formaldehyde overnight to yield a completely methylolated polyureaurethane polymer.

EXAMPLE F

A mixture of 200 grams of Jeffamine D-400, 200 grams of diethylene glycol monoamine, and 144.5 grams of urea are mixed together and heated at 83°–198°C. for ½ hours and then at 198°–211° C. for 8 hours. 1118.2 grams of a 33⅓ percent aqueous solution of the polyureaurethane polymer is blended with 257 grams of 37.1 percent aqueous formaldehyde while stirring with a magnetic stirrer. The pH of the solution is adjusted to 7.2 to 8.5 by adding 6.3 ml of 10 percent sodium hydroxide solution. The solution is then digested in an oven overnight at 50° C. while stirring. The resulting methylolated polyureaurethane polymer is adjusted with water to provide an aqueous solution containing 33.2 percent solids.

EXAMPLE G

90 grams (1.5 mols) of urea and 222 grams (1.5 mols) of triethylene glycol diamine are charged to a three-necked, round-bottom flask provided with a stirrer, thermometer, and reflux condenser vented through a dry ice trap. The reactants are heated, under an atmosphere of nitrogen, for 18 hours at 120°–165° C. The temperature is then slowly raised so as to allow an orderly collection of ammonia. The reaction is terminated when the calculated amount of ammonia has been formed. The product is then vacuum stripped for a short period of time to a final temperature of 185° C. The resulting polyurea polymer upon cooling is an opaque, light brown solid. A molecular weight determination using an osmometer gave a number average molecular weight of about 7,000. The polyurea polymer is soluble in water and contains 15.9 percent nitrogen and 0.09 percent amino nitrogen. A 25 percent solution of the polyurea at 22° C. in meta-cresol has a Gardner Holt viscosity of W. The polyurea polymer is then methylolated by heating a mixture of 5 grams of the polyurea polymer, 6 grams of 36.7 percent aqueous formaldehyde, and 69 grams of water at 51° C. for 15 hours.

EXAMPLE H

12 grams of urea and 200 grams of polyoxypropylene diamine (polyether L1000 from Union Carbide) are charged to the reaction flask described in Example G. The reactants are heated for 20 hours at 130°–140° C. and then vacuum stripped for a short period of time. The resulting polyurea polymer upon cooling is a dark amber viscous liquid. The polymer contains 2.76 percent nitrogen and 0.02 percent amino nitrogen. The polyurea is then methylolated by reacting 25 grams of the product with 25 grams of formaldehyde (55 percent solution of formaldehyde and methanol), adjusting the pH to 8.5 and refluxing the solution for 3½ hours.

EXAMPLE J

81.4 grams of triethylene glycol diamine, 5.8 grams hexamethylene diamine and 36 grams of urea are heated for 27 hours at 112°–180° C. The product is then vacuum stripped for a short period of time. The resulting polyurea upon cooling is a yellow solid. The polyurea is then methylolated by heating 2.5 grams of the polyurea with 3.53 grams 36.7 percent aqueous formaldehyde and 32 ml of water at 50° C. overnight.

The nonwoven fabrics of the present invention comprise random or oriented fibers held together by one or more of the methylolated polyurea polymers or methylolated polyureaurethane polymers described above. The nonwoven fabrics of this invention may be prepared by a process which comprises impregnating a non-woven textile material with a liquid application medium having dipersed therein one or more of the above-described methylolated polyurea polymers or methylolated polyureaurethane polymers.

The polymers used in the process of the present invention may be applied from any available form of liquid application medium such as from an aqueous emulsion or a solution in a suitable organic solvent. The binder polymer may be applied in any manner suitable for impregnating textiles with liquid textile treating medium, such as by dipping, padding, spraying, printing or immersing the textile in or with the treating medium. When emulsions are to be formed an emulsifying agent should be included. Emulsifying agents are well known in this art and any suitable agent can be employed. Examples of such suitable emulsifying agents include condensation products of octyl and nonyl phenol with from 8 to 17 mols of ethylene oxide.

The concentration of the binder polymer in the application medium is not a critical feature of the present invention. Typically the application medium applied to the textile contains from about 1 to about 25 percent of polymer.

The amount of binder polymer solids (dry basis) applied to the nonwoven textile may vary over a wide range and will depend among other things on the particular textile material and on the type and use intended for the final fabric. It is, essential, of course, that the amount be sufficient to bind the textile fibers. Although other amounts of binder polymer may be used if desired, the amount of polymeric binder present on the nonwoven fabric is typically from about 2 percent to about 80 percent by weight, and preferably from about 5 percent to about 50 percent by weight, based on the total weight of the nonwoven fabric.

Following application, the treated textile is dried and then cured at elevated temperatures. The temperature used to effect curing of the treated fabric will vary and will depend on such factors as the specific textile material being treated and the particular equipment employed. Of course, curing temperatures which cause discoloration or burning of the textile materials should be avoided. Typically, a temperature in the range of from about 200° F. to about 350° F. is employed. The time used for curing may also vary, generally in inverse proportions to the temperature used. For example, when a temperature of 200° F. is employed, longer curing times are required then when a temperature of 300° F. is employed.

The process of the present invention is applicable to nonwoven textile materials of all types made from natural and synthetic fibers and blends thereof. Illustrative examples of such materials include cotton fibers, blends of cotton and rayon, rayon, blends of cotton or rayon with synthetic fibers, wool, silk, polyesters, nylon, viscose rayon, acetate rayon, polyolefins, and other natural and synthetic fibers. Although any fiber or textile material may be used for the preparation of nonwoven fabrics of this invention, cellulosic fibers are preferred because of their low cost.

The textile materials used to prepare the nonwoven fabrics of the present invention may be fibers or fibrous structures, including webs containing a random arrangement of fibers, webs containing oriented fibers, laminated structures consisting of two or more webs having any desired degree of fiber orientation, webs reinforced with yarn, filaments, or scrims, and the like.

Although the fundamental ingredients used in the preparation of the nonwoven fabrics of this invention is the binder polymer described above, numerous other materials may be added to the application medium. The application medium may contain such additives as thermosetting agents, curing catalysts, softening agents, shrink-proofing agents, flame-proofing agents, and so forth. Materials in these groups are well known to those skilled in the art and would be applied to obtain the special effect indicated by the function of the agent. Alternatively, such material may be applied to the textile material either before or after application of the binder composition of the present invention.

The binder resins may be applied to the textile material in accordance with this invention along with an acid catalyst. It is believed that the acid catalyst promotes reaction of the binder polymer with the active hydrogens present in the textile material. The acid catalyst and the binder polymer may be applied to the textile material simultaneously or in separate steps as is conventional in the art. Illustrative examples of curing catalysts include zinc nitrate, ammonium chloride, ammonium dihydrogen phosphate, ammonium thiocyanate, aluminum sulfate, magnesium sulfate, magnesium chloride, triethanolamine hydrochloride, and triethyleneamine hydrochloride. The amount of acid catalyst used is the amount conventionally used in the art for curing methylolated polymers onto textile materials. In general, satisfactory reuslts are obtained by using from about 1 percent to about 35 percent of catalyst based on the weight of methylolated polymer used.

In order that those skilled in the art may better understand how the present invention may be practiced, the following illustrative examples are given. These examples are set forth solely for the purpose of illustration and any specific enumeration of details contained therein should not be interpreted as expressing limitations of this invention. All parts and percentages are by weight, unless otherwise stated.

EXAMPLE 1

A solution was prepared containing 20 percent of the product of Example B (a 35 percent active water solution of methylolated polyureaurethane polymer), 2 percent zinc nitrate, and 78 percent water. Strands of drawn sliver, prepared from 2 inch, 3 dpf, rayon staple, placed between two layers of cheese cloth for support, are immersed in the solution and then passed between the squeezed rolls of a laboratory padder. A wet pick up of 100 percent is obtained, resulting in the deposition of approximately 7 percent resin solids based on the weight of the fiber. The treated sliver is dried for 5 minutes at 225° F. and cured for 5 minutes at 325° F. Tests showed the tensile strength of the treated sliver to be 9.6 kilograms per strand and to have good softness. The tensile strength decreased to 6.9 kilograms after a 15 minute wash in 0.1 percent TIDE solution at 140° F. There was no change in the strength after a 15 minute wash in perchloroethylene at room temperature. The ensile strength before washing is approximately 3 times that obtained with an equal amount (solid basis) of a commercially available self-crosslinking acrylic binder (Rhoplex HA-8), and the softness is approximately equal. After washing the tensile strength is still about 1.8 times greater than that of the washed sample containing Rhoplex HA-8 acrylic binder. The results are shown in Table I.

EXAMPLE 2

A solution containing 20 percent of the aqueous solution of Example B, 2 percent zinc nitrate, 4 percent Mykon SF (a 25 percent active polyethylene emulsion), and 74 percent water. This solution was used to treat strands of drawn sliver as described in Example 1. Properties of the treated sliver are shown in Table I.

EXAMPLE 3

The methylolated polyureaurethane of Example A is applied to rayon sliver as in Example 1 except that the treated sliver is dried for 8 minutes at 225° F. and cured for 5 minutes at 325° F. The properties of the treated sliver are shown in Table I)

EXAMPLE 4

A rayon sliver, prepared from 1.5 denier, rayon staple is padded with a solution containing 20 percent of the aqueous solution of Example A, 2 percent zinc nitrate, and 78 percent water as provided in Example 1. The treated sliver is dried for 8 minutes at 225° F. and then cured for 5 minutes at 325° F. The properties of the treated sliver are shown in Table I.

EXAMPLE 5

Example 4 is repeated except that the binder resin used is the methylolated polyureaurethane polymer of Example E. Tests results on the treated sliver are shown in Table I.

EXAMPLE 6

Example 4 is repeated using the methylolated polyureaurethane polymer binder of Example F.

EXAMPLE 7

Example 4 is repeated except that the binder resin used is the methylolated polyureaurethane polymer of Example C. The test results are shown in Table I.

EXAMPLE 8

Example 4 is repeated except that the binder resin used is the methylolated polyureaurethane polymer prepared in Example D. The test results are shown in Table I.

TABLE I

Example Number	Dis-color-ation (1= least yellow)	Softness (1> softest)	Tensile Strength[1] Kg. before washing	washed[2]	dry cleaned[3]
1	2	3	9.64	6.90	9.63
2	2	2.5	6.10	5.60	7.40
3	1	1.5	7.00	7.07	7.07
4	4.0	2.2	7.81	9.56	9.18
5	3.8	3.2	5.75	5.22	5.56
6	3.3	1.0	2.64	2.73	2.85
7	6.2	4.5	16.64	13.94	18.30
8	6.5	5.7	12.00	16.20	16.16

[1]Average value of ten single-end breaks
[2]Washed 15 minutes in 0.1 detergent solution at 140°F.
[3]Drycleaned 15 minutes in perchloroethylene at room temperature

EXAMPLE 9

A solution is prepared containing 28.6 percent of the product of Example A (a 34.3 percent active water solution of methylolated polyureaurethane polymer), 2.86 percent zinc nitrate hexahydrate, and 68.54 percent water. The solution is used to pad sliver prepared from 1½ inch and 1.5 denier nylon fiber and to pad sliver prepared from 1½ inch and 1.5 denier polyester fiber. A wet pick up of 100 percent is obtained, resulting in the deposition of approximately 10 percent resin solids based on the weight of the slivers. The treated slivers are dried for 8 minutes at 225° F. and cured for 5 minutes at 325° F. The cured products are evaluated for softness, discoloration, and tensile strength. The results are shown in Table II.

TABLE II

Product	Softness	Discoloration	Tensile Strength[1] - Kg before washing	washed[2]	Drycleaned[3]
Nylon	excellent	none	10.7	14.6	11.2
Polyester	do.	do.	9.0	11.8	9.8

[1]Average of ten single-end breaks
[2]Washed by shaking in 0.1% aqueous solution of TIDE detergent for 15 minutes at 140°F.
[3]Drycleaned 15 minutes in perchloroethylene at room temperature

EXAMPLE 10

A solution is prepared containing 20 percent of the methylotated polyurea of Example G, 2 percent zinc nitrate, and 78 percent water. The solution is used to pad a nonwoven fleece prepared of cotton fibers at 100 percent wet pickup. The padded products are dried for 10 minutes at 200° F. and cured for 5 minutes at 300° F. The cured product exhibited excellent tensile strength and softness and negligible discoloration.

EXAMPLE 11

A nonwoven polyester fleece is sprayed with a solution containing 15 percent of the methylolated polyurea of Example H, 3 percent zinc nitrate, and 82 percent water. The treated polyester is dried for 8 minutes at 225° F. and then cured for 5 minutes at 330° F. The resulting nonwoven fabric has high tensile strength, a high degree of softness, and negligible discoloration.

EXAMPLE 12

An aqueous textile bath was prepared by mixing 10 parts of the methylolated polyurea of Example J with 2 parts of zinc nitrate and 88 parts of water. Nonwoven webs of rayon fleece are impregnated with this textile bath at 100 percent wet pickup, dried in air at 250° F. for 10 minutes, and then heated an additional 5 minutes at 300° F. The nonwoven fabrics exhibit excellent softness and drapeability, high tensile strength, and good color fastness.

EXAMPLE 13

Example 12 is repeated except that the polymer used is the methylolated polyureaurethane polymer of Example A. The treated nonwoven fabric has excellent tensile strength, color fastness, softness, and drapeability.

The nonwoven fabrics of the present invention are particularly useful as interfacings, linings, and shaping materials in the garment, shoe and luggage trade and in disposable items of wearing apparel such as hospital garments, underwear, dresses, shirts and so forth which may be worn once or several times before being discarded, and in sheets and needle-punched blankets.

Although this invention has been described with reference to specific methylolated polyureas, methylolated polyureaurethane polymers, and textile materials as well as specific processes and method steps, it will be apparent that other equivalent material may be substituted for those described and the method steps and types of processes may be altered, reversed, and in some cases eliminated, all within the spirit and scope of this invention as defined in the appended claims.

Having described the invention, what is desired to be secured by Letters Patent is:

1. A nonwoven fabric comprising fibers held together by from 2 percent to 80 percent of a binder resin, based on the total weight of fabric, wherein the binder resin comprises a methylolated polyurea characterized by the generalized formula

$$\left[-\overset{R}{\overset{|}{N}}-\overset{O}{\overset{\|}{C}}-\overset{R}{\overset{|}{N}}-(R'O)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-\right]_{p'}$$

wherein m is 0 or 1, q is an integer from 1 to 13 p' is an integer from 3 to 100, R' is an alkylene group containing from 3 to 5 carbon atoms, each R'' is independently hydrogen or methyl, and each R is independently selected from the group consisting of hydrogen and CH_2OH with the proviso that at least one R is CH_2OH or a methylolated polyureaurethane characterized by the generalized formula $(A)_x$ wherein x is an integer from 2 to 100 and each A is independently selected from the group consisting of

$$-\overset{R}{\overset{|}{N}}-\overset{O}{\overset{\|}{C}}-\overset{R}{\overset{|}{N}}-(R'O)_m\left(CH_2CHR''O\right)_q-CH_2CHR''-$$

and

$$-\overset{O}{\overset{\|}{C}}-\overset{R}{\overset{|}{N}}-(R'O)_m\left(CH_2CHR''O\right)_n-$$

wherein m is 0 or 1, n is an integer from 2 to 14, q is an integer from 1 to 13, R' is an alkylene group having from 3 to 5 carbon atoms, each R'' is independently hydrogen or methyl and each R is independently selected from the group consisting of hydrogen and CH_2OH, with the proviso that at least one A is

$$-\overset{R}{\overset{|}{N}}-\overset{O}{\overset{\|}{C}}-\overset{R}{\overset{|}{N}}-(R'O)_m-\left(CH_2CHR''O\right)_q-CH_2CHR''-$$

and at least one A is

$$-\overset{O}{\overset{\|}{C}}-\overset{R}{\overset{|}{N}}-(R'O)_m\left(CH_2CHR''O\right)_n-$$

with the proviso that at least on R is CH_2OH.

2. A nonwoven fabric of claim 1 wherein the binder resin is a methylolated polyurea.

3. A nonwoven fabric of claim 1 wherein the binder resin is a methylolated polyureaurethane.

4. A nonwoven fabric of claim 1 wherein the fiber comprises cellulosic fibers.

5. A nonwoven fabric of claim 1 comprising cellulose fibers held together by a methylolated polyureaurethane.

6. A nonwoven fabric of claim 1 wherein the fiber comprises nylon fibers.

7. A nonwoven fabric of claim 1 wherein the fiber comprises polyester fibers.

* * * * *