United States Patent Office 2,949,386
Patented Aug. 16, 1960

2,949,386

NON-WOVEN FABRICS

Norman S. Cassel, Ridgewood, N.J., assignor to Interchemical Corporation, New York, N.Y., a corporation of Ohio

No Drawing. Filed Nov. 3, 1958, Ser. No. 771,204

9 Claims. (Cl. 117—140)

This invention relates to non-woven fabrics and aims to provide improved non-woven fabrics especially for outer wear.

Non-woven fabrics for use as outer wear garments must be strong, durable, resilient, flexible, remain intact after washings and, if colored, the color should be washfast and resistant to dry cleaning.

Thermosetting melamine-formaldehyde, urea-formaldehyde, phenol-formaldehyde and dicyandiamide-formaldehyde have been proposed as binders for non-woven fabrics but these resins, after being thermoset, leave the fabric stiff, or boardy. Also, various thermoplastic resins or polymers have been suggested, usually in the form of a latex. The thermoplastics alone do not give durable bonding of the fibers to each other. One of the best binders for making non-woven fabrics has been the synthetic latices made by emulsion copolymerization of acrylonitrile and butadiene. Preferably these copolymers are given a "sulfur cure" or vulcanization after they are applied to the web of non-woven fibers. Non-woven fabrics made in this conventional manner are strong, durable, flexible, and reasonably resistant to dry cleaning solvent, but they are not washfast. For optimum washfastness the art has used a combination of thermosetting urea or melamine-formaldehyde resin with a latex of a polymer containing reactive groups. Such binder systems require relatively high temperatures, i.e. on the order of 350° F., to produce a non-woven fabric having good washfastness.

It has now been discovered that non-woven fabrics having good washfastness and good hand can be obtained by using as a binder a latex of a polymer containing carboxyl groups and a particular type of "cross-linking agent," i.e. a multifunctional monomeric material that will react at relatively low temperatures with the polymer and thus "cross-link" the polymer chains with each other making the polymer even more insoluble. The binder system of the present invention cures to give good washfastness at temperatures as low as 210° F. Higher temperatures can be used however, if a shorter cure time is needed. The new binder system provides as good or better washfastness and hand, as any prior art process and at the same time permits greater speed and lower costs by providing a binder system that cures at a relatively low temperature or in shorter time at the more conventional higher temperature. The cross-linking agents that we have found to be very effective are those containing three α,β-alkylene imine groups especially trialkylene phosphoramides. These cross-linking agents are preferably added to the polymer latex prior to its application to the web of non-woven fibers. This formulation, containing cross-linker, cross-linkable polymer, and pigment if desired, is applied to the non-woven web in conventional manner as by impregnating or spraying.

The solids pick up by the fabric can be between 15 and 200% of its weight of cross-linker plus polymer.

It is recognized that cross-linking linear polymer systems employing polyfunctional imine cross-linking agents have been used heretofore for producing films, for insolubilizing resins and for preparing resins of various sorts. For instance, U.S. Patent No. 2,327,760 discloses treating a viscose fabric with an aqueous impregnating bath containing sodium polyacrylate and a difunctional imine compound. U.S. Patent No. 2,341,413 describes the after-treatment with imine compounds of fabrics that have been previously impregnated with water-soluble coating materials such as albuminous substances, cellulose compounds or water-soluble artificial resins. German Patent No. 896,790 describes the use of the compound obtained by reacting one mole of phosphorous oxychloride with three moles of ethylene imine, in conjunction with polymers containing carboxyl groups, for finishing textiles, or in conjunction with pigments, for printing or padding on textiles. German Patent No. 867,085 discloses cross-linking water-soluble salts of acidic polymers and the use of such a system for finishing and decorating textiles.

None of the prior art teaches or suggests the use of the polyfunctional imines as binders for non-woven fabrics, especially the advantageous results that the present applicants are able to obtain. For instance none of the prior art would suggest or make obvious the result that the present invention makes possible, viz. that non-woven fabrics having good washfastness can be obtained without impairing the hand of the fabric at cure temperatures as low as 210° F. The saving in time and expense that is made possible by the binder system is great.

Conventional non-woven fabric webs are employed for making the non-woven fabrics of the present invention. The webs may be made of cotton fibers, viscose rayon fibers or any of the blends with synthetic fibers, or all synthetics.

Typical latex polymers that are operable in the invention include the water-insoluble emulsion copolymers of acrylic acid or methacrylic acid, maleic acid, fumaric acid, and itaconic acid, with at least one copolymerizable monomeric material. Such copolymerizable monomers include, for instance, acrylonitrile, methacrylonitrile, butadiene, styrene, lower alkyl esters of acrylic or methacrylic acid, etc. These latices are prepared in conventional manner by emulsion polymerization using a free radical forming catalyst. We prefer that the monomer containing the free acid group comprise no more than about 7% by weight of the total weight of monomers being polymerized. Also, we prefer that a major portion of the remaining portion of the polymer be comprised of a lower alkyl ester of acrylic acid, i.e. methyl, ethyl, propyl or butyl acrylate. Such copolymers containing up to about 7% acrylic acid give non-woven fabrics of exceptionally soft hand and good washfastness. Best resistance to alternate light wash is obtained by including 25 to 35 percent acrylonitrile as a monomer. Quite desirable results are also obtained using 1,3-butadiene:acrylonitrile:methacrylic acid copolymers such as Hycar 1571. This latter material is typical of the latex polymers that are used in the trade as binders for non-woven fabrics. Conventional vulcanizing agents or zinc oxide are recommended in the trade as curing agents for this binder. The present invention provides an improvement in this prior practice in that the use of trialkylene phosphoramide as the cross-linking agent for the Hycar 1571 latex type of non-woven fabric binder gives a non-woven fabric having improved washfastness.

Example 1.—A non-woven cotton fabric (85 grams/sq. yd.) made of randomly distributed intersecting fibers impregnated by immersing it in an aqueous composition of 12% solids, the solids consisting of a mixture of 100 parts of a 53.5:43.5:3 butadiene:acrylonitrile:methacrylic acid emulsion copolymer, 5 parts tripropylene phosphoramide and 1.12 parts of sodium alginate (used as a migration control agent). The fabric was removed from the composition and the excess impregnant removed by running it between rubber-squeeze rolls adjusted to give a dry weight pick up on the fabric of 25–30%. The fabric was dried and cured by placing in a circulating hot air oven at 280° F. for 5 minutes. A sample of the material, when subjected to an Accelerated Wash Fastness Test (Number 3A, under Standard Test Method 61–54 as outlined in the Technical Manual and Year Book of the American Association of Textile Chemists and Colorists) showed excellent washfastness compared to a similar fabric which had been given a similar treatment with an impregnating composition containing conventional rubber curing agents, i.e. 0.5 part ammonium caseinate, 5.0 parts zinc oxide, 2.0 parts sulfur and 2.0 parts zinc diethyldithiocarbamate, instead of the tripropylene phosphoramide. Curing for 20 minutes instead of 5 minutes at 280° F. gave the same results.

Example 2.—The above example was repeated except that 1 part of tripropylene phosphoramide was used instead of 5 parts. There was only a slight loss of color when the fabric was subjected to a No. 3A wash test. A fabric similarly treated except that no cross-linking agent was present in the impregnating bath resulted in a fabric that completely disintegrated when subjected to the No. 3A wash test.

Example 3.—Example 1 was repeated using 3 parts of tripropylene phosphoramide instead of 5 parts and this resulted in a fabric having excellent washfastness when subjected to a No. 3A washfastness test.

Example 4.—Example 1 was repeated using 5 parts of tripropylene thiophosphoramide instead of 5 parts of tripropylene phosphoramide. The resulting fabric showed only a slight loss in color when subjected to a No. 3A wash test.

Example 5.—Example 1 was repeated using a 60:35:5 emulsion copolymer of butyl acrylate:acrylonitrile:methacrylic acid instead of the butadiene:acrylonitrile:methacrylic acid copolymer. The non-woven fabric obtained had excellent washfastness when subjected to a No. 3A wash test and the fabric had excellent hand and was also non-yellowing.

Example 6.—Example 1 was repeated using the impregnating bath at 35% solids instead of 12%. This resulted in a 100% by weight pick-up by the fabric. The fabric had excellent washfastness but was slightly stiffer than the fabric of Example 1.

Example 7.—A non-woven viscose fabric made up of oriented overlapping intersecting viscose fibers and weighing 400 grains per square yard was impregnated with a composition consisting of (1) 63.5 parts of a 37% solids latex of copolymer containing 90% ethyl acrylate, 5% acrylonitrile and 5% methacrylic acid, (2) 2.5 parts of tripropylene phosphoramide and (3) 34 parts of water. The impregnated fabric was passed between squeeze rollers adjusted to give a pick-up of impregnant of 25% of the weight of the web (dry basis). After can drying for one minute at 210° F. the fabric was aged at room temperature for 24 hours. The fabric had excellent washfastness for its weight (e.g. withstands AATCC #2 Wash) and tensile strength. Its tensile strength is equal to the tensile strength of a similar non-woven fabric made with 50% dry pick-up of polyvinyl acetate (a conventional non-woven fabric binder). The fabric made with polyvinyl acetate as binder was also stiffer than the product of this example and had no detectable washfastness. The non-woven fabric of this example was non-yellowing when exposed to heat or light (Fadeometer).

Example 8.—A non-woven web made up of randomly distributed cotton fibers was impregnated (35% dry pick-up) in a bath consisting of

	Parts
Phthalocyanine blue pigment (added as a 16% aqueous dispersion)	3.0
37% latex (95:5 ethylacrylate:methacrylic acid copolymer)	75.00
Tripropylene phosphoramide	2.0
Water	20.00

After can drying as in Example 7 the fabric was heated for three minutes at 300° F. The fabric had good washfastness (#3A AATCC Wash Test) and dry cleaning fastness even though it had a very soft hand. It also had good light fastness (40 hours in Fadeometer with no change) and good alternate light-wash fastness.

Example 9.—A non-woven web made up of 70% rayon and 30% nylon fibers was impregnated (40% dry pick-up) with a bath consisting of

	Parts
Phthalocyanine blue pigment (as a 16% aqueous dispersion)	3.00
37% latex (60:35:5 copolymer butyl acrylate: acrylonitrile:methacrylic acid)	80.00
Tripropylene phosphoramide	1.50
Water	15.50

After can drying one minute at 210° F. the fabric was given an additional cure of three minutes at 300° F. The resulting fabric had fastness properties similar to those for the fabric produced in Example 8.

The main purpose for adding the blue pigment to the impregnating baths in the above examples was to make it possible to observe the tendency of the binder to migrate on drying, but from these examples it is obvious that pigments and dyes could be used with the binder so that coloring and binding can be achieved in a single operation. If migration of the binder becomes a problem, so that even distribution of binder over the web is not obtained, it can be largely overcome by the addition of a small amount of sodium alginate to the bath.

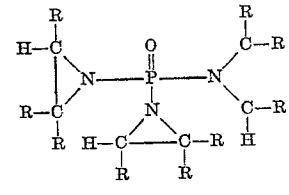
I claim:

1. A non-woven fabric comprising a web of overlapping intersecting fibers uniformly bonded with a binder formed in situ by cross-linking a linear polymer containing carboxyl groups with a polyfunctional imine cross-linking agent.

2. A non-woven fabric comprising a web of overlapping intersecting fibers uniformly bonded with 5 to 200% of its weight of a cross-linked polymer formed by cross-linking a linear polymer containing carboxyl groups with an imine cross-linking agent.

3. A non-woven fabric as in claim 2 wherein the binder is formed by cross-linking a linear copolymer containing 1 to 7% of copolymerized monomer of the group consisting of acrylic acid and methacrylic acid.

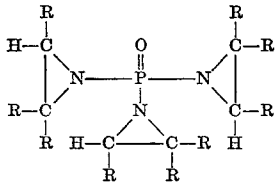
4. A non-woven fabric as in claim 2 wherein the imine cross-linking agent is a trialkylene phosphoramide having the structure



where R is a member of the group consisting of hydrogen, methyl and ethyl.

5. A non-woven fabric comprising a web of overlapping intersecting fibers bonded together with 5 to 200% of its weight of a binder formed by cross-linking a linear addition polymer of 1 to 7% by weight of methacrylic acid, 60 to 75% by weight of a monomer selected from the group consisting of butadiene and butyl acrylate, and 0

to 35% acrylonitrile with 1 to 5% of its weight of a polyfunctional imine of the formula:



where R is selected from the group consisting of hydrogen, methyl and ethyl.

6. A non-woven fabric as in claim 5 wherein the polyfunctional amine is tripropylene phosphoramide.

7. A non-woven fabric comprising a web of overlapping intersecting fibers bonded together with 5 to 200% of its weight of a binder formed by cross-linking a linear addition polymer containing 1 to 7% by weight of methacrylic acid, 0 to 35% by weight of acrylonitrile and 60 to 95 parts of butyl acrylate, with 1 to 5% of its weight of tripropylene phosphoramide.

8. The process for making non-woven fabric which consists of (1) uniformly applying to a web of overlapping intersecting fibers 5 to 200% by weight of cross-linkable binder consisting of 95 to 99 parts of a cross-linkable linear addition polymer and 1 to 5 parts of an imine cross-linking agent and (2) heating the web and binder to cause cross-linking of the polymer and cross-linking agent.

9. A process as claimed in claim 8 wherein the cross-linking agent is tripropylene phosphoramide.

References Cited in the file of this patent

UNITED STATES PATENTS

2,676,128	Piccard	Apr. 20, 1954
2,698,574	Dougherty et al.	Jan. 4, 1955
2,719,806	Nottebohm	Oct. 4, 1955
2,774,687	Nottebohm	Dec. 18, 1956
2,823,142	Sumner et al.	Feb. 11, 1958