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3,526,538

NONWOVEN FABRIC PRODUCT AND THE LIKE AND PREPARATION THEREOF

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No Drawing. Continuation of application Ser. No. 444,441, Mar. 31, 1965. This application Sept. 23, 1968, Ser. No. 768,954

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U.S. Cl. 117-140

3 Claims

ABSTRACT OF THE DISCLOSURE

A latex composition comprising an aqueous medium having colloiddally suspended in it an emulsion-polymerized interpolymer of vinyl acetate, ethylene and allyl glycidyl ether, the interpolymer containing 5 to 40% ethylene and at least about 0.5% allyl glycidyl ether based on the vinyl acetate, is used to make nonwoven fabrics from a loosely assembled mass of fibers, the interpolymer deposited from the latex being effective as a binder for the fibers.

This is a continuation of our application Ser. No. 444,441, filed Mar. 31, 1965 (now abandoned).

This invention relates to so-called "nonwoven" fabrics of the type composed of loosely assembled webs of fibers bound together with an adhesive binder, and is more particularly concerned with cellulosic nonwoven fabrics bound by a binder which is a modified copolymer of vinyl acetate.

Adequately bonded nonwoven fabrics have advantages over woven fabrics for a large variety of uses. Bonded nonwoven fabrics have heretofore been formed by impregnating, printing or otherwise depositing an adhesive bonding material on a base web predominantly comprising relatively long fibers, including those of textile length of from about 1/2 inch to about 2 1/2 inches, or more. The base web of nonwoven fibers, to which the binder is applied, can be produced inexpensively and with low capital investment by carding, garnetting, air-laying, papermaking procedures, or other known operations for which efficient automation is possible. The operation of bonding the fibers in place is much less expensive than conventional spinning and weaving. In comparison with woven fabric, the bonded nonwoven fabrics can be made in a much greater range of the thicknesses per unit weight, with more homogeneous structures, no unraveling tendency, and with greater water absorbency, porosity and resiliency, when required.

Generally speaking, it is desirable to produce a soft fabric with as much strength as possible. The choice of the bonding agent is very important in obtaining both softness and strength. Many bonding agents which are used in the art, such as polyvinyl acetate, have adequate bonding strength when dry, but lose a major proportion of this strength when the fabric is wetted with water. This wet strength may be defined as the ability of the fabric to retain part of its dry strength after substantially complete saturation with water. This property of wet strength is very important when the fabric is to be used in any manner where it will become moist or will come into contact with fluid, e.g., when it is used as a towel, as a disposable diaper, or the like.

In order to obtain fabrics which are textile-like in quality, having drape, flexibility, and softness, the polyvinyl acetate resins have been softened with "external" plasticizers such as dibutyl phthalate before application

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to the web, or the vinyl acetate has been copolymerized with internally plasticizing comonomers such as the alkyl acrylates, the alkyl methacrylates, vinyl stearate, or the dialkyl fumarates or maleates. The internally plasticized copolymers impart to the resulting fabric somewhat improved wet strength and wet abrasion resistance over the use of externally-plasticized homopolymers as binders, but still greater wet strength at high flexibility is desired for the widest use of these fabrics.

Considerable time and effort have also been expended on developing techniques for improving the wet strength of bonding agents for nonwoven fabrics consisting predominantly of cellulose fibers. One of the more important developments in this field has been the treatment of the fabrics with aminoaldehyde condensation products such as urea-formaldehyde and melamine-formaldehyde, along with heat and catalyst to set the resin and cross-link the cellulose fibers. Such treatments, however, although they increase wet strength, are relatively costly and complicated, reduce the softness of the fabric, and tend to have a detrimental odor due to the release of formaldehyde.

It is accordingly an object of this invention to provide new, improved polymeric binders for nonwoven fabrics.

Another object of the invention is to provide relatively low-cost, binder-containing nonwoven fabrics of enhanced wet strength and other desirable properties.

In accordance with this invention it has been found that these and related objects are achieved by a binder for nonwoven fabrics which is an interpolymer of vinyl acetate and ethylene copolymerized with allyl glycidyl ether which is effective to polymerize and to cross-link with the initial vinyl acetate and ethylene containing interpolymer under the action of heat. The above-described binder is applied to the fiber web to bind the fibers together into the desired nonwoven fabric in the form of an aqueous latex containing the interpolymer of vinyl acetate, ethylene and copolymerized allyl glycidyl ether in the dispersed phase. The vinyl acetate-ethylene-allyl glycidyl ether interpolymer is characterized by an ethylene content of 5 to 40%, preferably 16 to 40%, a particle size of 0.1 to 2μ, preferably 0.1 to 0.25μ, and an intrinsic viscosity of 1 to 2.5 dl./g. The amount of allyl glycidyl ether is 0.5 to 10% based on the vinyl acetate.

The binder is readily prepared by the interpolymerization of vinyl acetate, ethylene and allyl glycidyl ether in an aqueous dispersion system. The allyl glycidyl ether readily copolymerizes with the vinyl acetate and the ethylene to form an interpolymer or terpolymer but, as mentioned, is adapted to undergo further reaction after this initial polymerization upon the application of heat in the processing of the nonwoven fabric to further cross-link the interpolymer. Particularly suitable as the binder is a vinyl acetate-ethylene-allyl glycidyl ether interpolymer latex which is prepared by the following process.

Vinyl acetate and ethylene are copolymerized in the presence of the allyl glycidyl ether in an aqueous medium under pressure not exceeding 100 atmospheres in the presence of a catalyst and at least one emulsifying agent, the aqueous system being maintained, by a suitable buffering agent, at a pH of 2 to 6, the catalyst being added incrementally. The process is a batch process which involves first a homogenization period in which the vinyl acetate suspended in water is thoroughly agitated in the presence of ethylene under the working pressure to effect solution of the ethylene in the vinyl acetate up to the substantial limit of its solubility under the conditions existing in the reaction zone, while the vinyl acetate is gradually heated to polymerization temperature. The homogenization period is followed by a polymerization period during which the

catalyst, which consists of a main catalyst or initiator, and may include an activator, is added incrementally, and the allyl glycidyl ether is similarly added incrementally, the pressure in the system being maintained substantially constant by application of a constant ethylene pressure.

Various free-radical forming catalysts can be used in carrying out the polymerization of the monomers, such as peroxide compounds. Combination type catalysts employing both reducing agents and oxidizing agents can also be used. The use of this type of combined catalyst is generally referred to in the art as "redox polymerization" or "redox system." The reducing agent is also often referred to as an activator and the oxidizing agent as an initiator. Suitable reducing agents or activators include bisulfites, sulfoxylates, or other compounds having reducing properties such as ferrous salts, and tertiary aromatic amines, e.g., N,N-dimethyl aniline. The oxidizing agents or initiators include hydrogen peroxide, organic peroxides such as benzoyl peroxide, t-butyl hydroperoxide and the like, persulfates, such as ammonium or potassium persulfate, perborates, and the like. Specific combination type catalysts or redox systems which can be used include hydrogen peroxide and zinc formaldehyde sulfoxylate; hydrogen peroxide, ammonium persulfate, or potassium persulfate, with sodium metabisulfite, sodium bisulfite, ferrous sulfate, dimethyl aniline, zinc formaldehyde sulfoxylate or sodium formaldehyde sulfoxylate. Other types of catalysts that are well-known in the art can also be used to polymerize the monomers, such as the peroxide compounds, with or without the addition of reducing agents or other activating materials. It is advantageous to utilize more water-soluble peroxides, such as hydrogen peroxide, rather than the more oil-soluble peroxides such as t-butyl hydroperoxide, in the redox system, to catalyze the monomer polymerization. Redox catalyst systems are described, for example, in "Fundamental Principles of Polymerization," by G. F. D'Alelio (John Wiley and Sons, Inc., New York, 1952) pp. 333, et seq. Other types of catalysts that are well-known in the art can also be used to polymerize the monomers according to this invention, with or without the addition of reducing agents or other activating materials.

The catalyst is employed in the amount of 0.1 to 2%, preferably 0.25 to 0.75%, based on the weight of vinyl acetate introduced into the system. The activator is ordinarily added in aqueous solution and the amount of activator is generally 0.25 to 1 times the amount of catalyst.

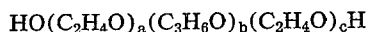
The emulsifying agents which are suitably used are nonionic. Suitable nonionic emulsifying agents include polyoxyethylene condensates. Polyoxyethylene condensates may be represented by the general formula:



where R is the residue of a fatty alcohol containing 10-18 carbon atoms, an alkyl phenol, a fatty acid containing 10-18 carbon atoms, an amide, an amine, or a mercaptan, and where *n* is an integer of 1 or above. Some specific examples of polyoxyethylene condensates which can be used include polyoxyethylene aliphatic ethers such as polyoxyethylene lauryl ether, polyoxyethylene oleyl ether, polyoxyethylene hydroabietyl ether and the like; polyoxyethylene alkaryl ethers such as polyoxyethylene nonylphenyl ether, polyoxyethylene octylphenyl ether and the like; polyoxyethylene esters of higher fatty acids such as polyoxyethylene laurate, polyoxyethylene oleate and the like as well as condensates of ethylene oxide with resin acids and tall oil acids; polyoxyethylene amide and amine condensates such as N-polyoxyethylene lauramide, and N-lauryl-N-polyoxyethylene amine and the like; and polyoxyethylene thio-ethers such as polyoxyethylene n-dodecyl thio-ether.

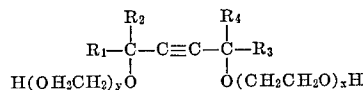
The nonionic emulsifying agents which can be used according to this invention also include a series of sur-

face active agents known as "Plurionics." The Plurionics have the general formula:



where *a*, *b*, and *c* are integers of 1 or above. As *b* increases, the compounds become less water soluble or more oil soluble and thus more hydrophobic when *a* and *c* remain substantially constant.

In addition, highly suitable are a series of ethylene oxide adducts of acetylenic glycols sold commercially under the name "Surfynals." This class of compounds can be represented by the formula



in which R₁ and R₄ are alkyl radicals containing from 3 to 10 carbon atoms, R₂ and R₃ are selected from the group consisting of methyl and ethyl, *x* and *y* have a sum in the range of 3 to 60, inclusive.

Some examples of nonionic emulsifying agents which can be used are as follows:

A polyoxyethylene nonylphenyl ether having a cloud point of between 126 and 133° F. is marketed under the trade name "Igepal CO-630" and a polyoxyethylene nonylphenyl ether having a cloud point above 212° F. is marketed under the trade name "Igepal CO-887." A similar polyoxyethylene nonylphenyl ether having a cloud point of about 86° F. is marketed under the trade name "Igepal CO-610." A polyoxyethylene octylphenyl ether having a cloud point of between 80° F. and 160° F. is marketed under the trade name "Triton X-100."

A polyoxyethylene oleyl ether having a cloud point of between 80° F. and 160° F. is marketed under the trade name "Atlas G-3915" and a polyoxyethylene lauryl ether having a cloud point above 190° F. is marketed under the trade name "Brij 35."

A polyoxypropylene having a cloud point of about 140° F. is marketed under the trade name "Pluronic L-64," and a polyoxypropylene having a cloud point of about 212° F. is marketed under the trade name "Pluronic F-68." "Pluronic L-64" is a polyoxyethylene-polyoxypropylene glycol conforming to the above general formula for "Plurionics" in which the polyoxypropylene chain has a molecular weight of 1500 to 1800 and the polyoxyethylene content is from 40 to 50 percent of the total weight of the molecule. "Pluronic F-68" is a polyoxyethylene-polyoxypropylene glycol conforming to the above general formula for "Plurionics" in which the polyoxypropylene chain has a molecular weight of 1500 to 1800 and the polyoxyethylene content is from 80 to 90 percent of the total weight of the molecule. The polyoxypropylene "Plurionics" are obtained by condensing ethylene oxide on the polyoxypropylene base and the hydrophobic-hydrophilic nature of the resulting compound is controlled by varying the molecular weight of either the hydrophobic base or the hydrophilic portion of the molecule.

Representative of the "Surfynols" are "Surfynol 465" which is an ethylene oxide adduct of 2,4,7,9-tetramethyl decnediol containing an average of 10 moles of ethylene oxide per mole, and "Surfynol 485" which corresponds to "Surfynol 465" but contains an average of 30 moles of ethylene oxide per mole. "Surfynol 465" has a cloud point of about 145° F. and "Surfynol 485" has a cloud point above 212° F.

In the foregoing, cloud points recited are based on 1% aqueous solutions. A single emulsifying agent can be used, or the emulsifying agents can be used in combination. When combinations of emulsifying agents are used, it is advantageous to use a relatively hydrophobic emulsifying agent in combination with a relatively hydrophilic agent. A relatively hydrophobic agent is one having a cloud point in 1% aqueous solution below 190° F. and a relatively

hydrophilic agent is one having a cloud point in 1% aqueous solution of 190° F. or above.

The concentration range of the total amount of emulsifying agents useful is from 0.5 to 5% based on the aqueous phase of the latex regardless of the solids content. Latex stabilizers are also advantageously used. The stabilizers employed are, in part, governed by the use to which the copolymer latex is to be put, and/or the particle size of the copolymer. For example, the vinyl acetate ethylene copolymer latices prepared by the method described can have various average particle size ranges. When the latices are to have a small average particle size, e.g., below 0.25 μ as preferred in the present invention, an ethylenically-unsaturated acid having up to 6 carbon atoms, is advantageously used as the stabilizer. Typical acids of this character are acrylic acid, methacrylic acid, itaconic acid, maleic acid, vinyl sulfonic acid and the like. These unsaturated acids impart increased stability to the latices. They tend to copolymerize with the monomers in the system. The amount of unsaturated acid is suitably 0.1 to 3% based on vinyl acetate, preferably 0.2 to 1%.

On the other hand, when the latex has an average particle size above 0.25 μ , a protective colloid can be used in the polymerization mixture as the stabilizing agent, although an unsaturated acid can be used if desired. Various amounts of colloids can be incorporated into the latices as desired, but it is preferred to maintain the colloid concentration at the lowest level possible. The amount of colloid used will also depend upon the particular colloid employed. Colloids of higher molecular weight tend to produce a latex of a higher viscosity than like amounts of a lower molecular weight colloid. Other properties of the colloids aside from their molecular weight also affect the viscosity of the latices and also the properties of the films formed therefrom. It is advantageous to maintain the colloid content of the latices between about 0.05 and 2% by weight based on the total latex, and hydroxyethyl cellulose is a particularly advantageous colloid when used in the latices.

Various other colloids can also be used, including polyvinyl alcohol, partially-acetylated polyvinyl alcohol, e.g., up to 50% acetylated, casein, hydroxyethyl starch, carboxymethyl cellulose, gum arabic, and the like, as known in the art of synthetic polymer latex technology.

In order to maintain the pH of the system at the desired value, there is suitably added an alkaline buffering agent of any convenient type. Any alkaline material which is compatible with the stabilizing agent can be used as the buffer. The amount of buffer is that sufficient to adjust the pH of the system within the desired range. Ammonium and sodium bicarbonate are preferred buffers because of their compatibility with the system and their low cost. The amount of buffer is generally about 0.1 to 0.5% by weight, based on the monomers. Other buffers such as disodium phosphate, sodium acetate, and the like, can, however, also be used.

One of the features of the method described above is that latices of relatively high solids contents can be directly produced and thus the products generally have, as produced, solids contents of 45 to 60%. They can, of course, be easily thinned by the addition of water to lower solids contents of any desired value.

Lower reaction temperatures for polymerizing vinyl acetate than have heretofore been feasible economically can also be used. The use of lower reaction temperatures has been found to result in higher molecular weight vinyl controlled by the rate of catalyst addition and by the rate of the heat dissipation therefrom. Generally, we have found that it is advantageous to maintain a mean temperature of about 50° C. during the polymerization of the monomers and to avoid temperatures much in excess of 80° C. While temperatures as low as 0° can be used, economically the lower temperature limit is about 30° C.

The reaction time will also vary depending upon other variable such as the temperature, the catalyst, and the desired extent of the polymerization. It is generally desirable to continue the reaction until less than 0.5% of the vinyl acetate and allyl glycidyl ether remains unreacted. Under these circumstances, a reaction time of about 6 hours has been found to be generally sufficient for complete polymerization, but reaction times ranging from 3 to 10 hours have been used, and other reaction times can be employed, if desired.

In carrying out the polymerization, a substantial amount of the vinyl acetate is initially charged to the polymerization vessel and saturated with ethylene in the manner discussed above. Most advantageously, at least about 75% of the total vinyl acetate to be polymerized is initially charged, preferably at least about 85%, and the remainder of the vinyl acetate is incrementally added during the course of the polymerization. The charging of all of the vinyl acetate initially is also contemplated, with no additional incremental supply. When reference is made to incremental addition, whether of vinyl acetate, allyl glycidyl ether, catalyst, or activator, substantially uniform additions, both with respect to quantity and time, are contemplated.

The quantity of ethylene entering into the copolymer is influenced by the pressure, the agitation, and the viscosity of the polymerization medium. Thus, to increase the ethylene content of the copolymer, higher pressures are employed, but even to introduce 40% or more of ethylene into the copolymer, pressures in excess of 100 atms. are not required. However, a pressure of at least about 10 atms. is most suitably employed. Similarly, when high ethylene contents are desired, a high degree of agitation should be employed, and high viscosities should be avoided, a low viscosity being preferred. When referring to viscosities, a viscosity of 30 to 150 centipoises is considered a low viscosity, a viscosity of 151 to 800 centipoises is considered a medium viscosity, and a viscosity of 801 to 3000 centipoises is considered a high viscosity.

The process of forming the vinyl acetate-ethylene-allyl glycidyl ether interpolymer latices generally comprises the preparation of an aqueous solution containing at least some of the emulsifying agent and stabilizer, and the pH buffering system. This aqueous solution and the initial charge of vinyl acetate are added to the polymerization vessel and ethylene pressure is applied to the desired value. As previously mentioned, the mixture is thoroughly agitated to dissolve ethylene in the vinyl acetate and in the water phase, agitation being continued until substantial equilibrium is achieved. This generally requires about 15 minutes. However, less time may be required depending upon the vessel, the efficiency of agitation, the specific system, and the like. In any case, by measuring the pressure drop of the ethylene in conventional manner, the realization of substantial equilibrium can be easily determined. Conveniently the charge is brought to polymerization temperature during this agitation period. Agitation can be effected by shaking, by means of an agitator, or other known mechanism. The polymerization is then initiated by introducing initial amounts of the catalyst, and of the activator when used. After polymerization has started, the catalyst and the activator are incrementally added as required to continue polymerization, and the allyl glycidyl ether and the remaining vinyl acetate, if any is similarly added.

As mentioned, the reaction is generally continued until the residual vinyl acetate and allyl glycidyl ether content is below 0.5%. The completed reaction product is then allowed to cool to about room temperature, while sealed from the atmosphere. The pH is then suitably adjusted to a value in the range of 4.5 to 7, preferably 6 to 6.5, to insure maximum stability.

The particle size of the latex can be regulated by the quantity of non-ionic emulsifying agent or agents em-

ployed and by the use or nonuse of a colloidal stabilizing agent. Thus, to obtain smaller particle sizes, greater amounts of emulsifying agent are used and colloidal stabilizing agents are not employed. For example, to provide average particle sizes below about 0.25, the total amount of nonionic emulsifying agent should be at least about 2%, based on the aqueous phase of the latex, and no colloidal stabilizing agent should be used, or if a colloidal stabilizing agent is used, only very small amounts should be employed.

On the other hand, when particle sizes of 0.25 and above are desired, at most about 2% of total emulsifying agent based on the aqueous phase of the latex should be used, and a colloidal stabilizing agent should be included in the amounts previously indicated. As a general rule, the smaller the amount of emulsifying agent employed and the greater the amount of colloidal stabilizing agent included in the latex system, the greater the average particle size. Conversely, the greater the amount of the emulsifying agent employed and the smaller the amount of colloidal stabilizing agent use, including the total absence of the latter, the smaller the average particle size. It will be understood that in each case, the quantity and size values referred to above are all within the ranges of values previously specified.

By following the procedure described above, particularly the initial saturation of the polymerization mixture with ethylene before polymerization is initiated, there can be produced the stable vinyl acetate-ethylene-allyl glycidyl ether interpolymers latex characterized above, with the copolymer having an ethylene content of 5 to 40%, an intrinsic viscosity of 1 to 2.5 dl./g., and an average particle size of 0.1 to 2, and the latex having a high solids content of up to 60% or more.

The ethylene content can be determined by means of the saponification number.

Intrinsic viscosity is suitably determined by conventional techniques, e.g., in accordance with the procedure described on pages 309-314 of "Principles of Polymer Chemistry," by Paul J. Flory (Cornell University Press—1963); using an Ubbelohde (suspended level) Viscometer at 30° C.

The vinyl acetate-ethylene allyl glycidyl ether binder described above is suitably used to prepare nonwoven fabrics by a variety of methods known to the art in which, in general, involve the impregnation of a loosely-assembled mass of fibers with the binder latex, followed by moderate heating to dry the mass. In the case of the present invention this moderate heating also serves to cure the binder by forming a cross-linked interpolymers. Before the binder is applied it is, of course, mixed with a suitable catalyst for the allyl glycidyl ether. Thus, basic catalysts such as organic amines, e.g., ethylenediamine and piperidine, or metal salts of weak acids, such as sodium acetate or zinc fluoborate are suitably used, as known in the art. The amount of catalyst is generally about 0.5 to 2% of the total resin.

The starting layer or mass can be formed by any one of the conventional techniques for depositing or arranging fibers in a web or layer. These techniques include carding, ginning, air-laying, and the like. Individual webs or thin layers formed by one or more of these techniques can also be laminated to provide a thicker layer for conversion into a fabric. In general, the fibers extend in a plurality of diverse directions in general alignment with the major plane of the fabric, overlapping, intersecting and supporting one another to form an open, porous structure. When reference is made to "cellulose" fibers, those fibers containing predominantly C₆H₁₀O₅ groupings are meant. Thus, examples of the fibers to be used in the starting layer are the natural cellulose fibers such as cotton and hemp and the synthetic cellulose fibers, such as rayon, and regenerated cellulose. Often the fibrous starting layer contains at least 50% cellulose fibers, whether they be natural or syn-

thetic, or a combination thereof. Other fibers in the starting layer may comprise natural fibers such as wool, or jute; artificial fibers such as cellulose acetate; synthetic fibers such as polyamides, i.e. nylon, polyesters, i.e. "Dacron," acrylics, i.e. "Dynel," "Acrilan," "Orlon," polyolefins, i.e. polyethylene, polyvinyl chloride, polyurethane, etc., alone or in combination with one another.

The fibrous starting layer or web suitably weighs from about 100 grains to about 2,000 grains per square yard and preferably weighs about 200 grains to about 800 grains per square yard. This fibrous starting layer, regardless of its method of preparation, is then subjected to at least one of the several types of bonding operations to anchor the individual fibers together to form a self-sustaining web. Some of the better-known methods of bonding are overall impregnation, or printing the web with intermittent or continuous straight or wavy lines or areas of binder extending generally transversely or diagonally across the web and additionally, if desired, along the web.

The amount of binder, calculated on a dry basis, applied to the fibrous starting web, suitably ranges from about 20 to about 100% or more by weight of the starting web, and preferably from about 35 to about 50% by weight of the starting web. The impregnated web is then dried and cured. Thus, the fabrics are suitably dried by passing them through an air oven or the like and then through a curing oven. Ordinarily, drying is effected at 150°-200° F. for 4-6 min., followed by curing at 300°-310° F. for 3-5 min. or more. However, other temperature relationships can be employed, as is well known in the art, shorter times at higher temperatures or longer times at lower temperatures being used. For example, the curing step can be carried out at 280° F. for about 15 min. or more. However, economic considerations make the use of excessively long times undesirable, and the upper temperature limit is governed by the nature of the fibers. Temperatures which degrade the fibers are, of course, avoided. However, if the fibers are heat resistant, temperatures even as high as 350° F. or higher can be used with times of 5-10 min. or more. In some cases, if desired, the drying and curing can be effected in a single exposure or step, e.g. at 300° F. for 5-10 min.

Nonwoven fabrics prepared in accordance with this invention have wet strength values as great as the usual woven cotton fabrics. In addition, these nonwoven fabrics have the outstanding advantage of low cost, both in comparison with woven fabrics and with nonwoven fabrics prepared with binders previously available.

External plasticizers are not needed with the binders of this invention, which greatly simplifies the preparation of nonwoven fabrics. However, they can be used to modify the properties of the fabrics when desired. Thus some external plasticizers can be added when an extremely soft fabric is desired.

Thus, it has been observed that the flexibility of the fabric can be increased by the addition of a hydrophobic external plasticizer to the binder composition without loss of desirable properties. Examples of external plasticizers which are suitably used include dibutoxyethylphthalate, dibutyl phthalate, tricresyl phosphate, and low molecular weight polyesters.

These external components may be added just before application, if their stability in the dispersion or solution is low, or they may be formulated into the aqueous dispersion of the binder and stored if the stability in aqueous dispersion is high.

The following examples are given to illustrate the present invention, but it will be understood that they are intended to be illustrative only and not limitative of the invention. In the examples, all parts are by weight unless otherwise indicated.

EXAMPLE 1

The following was charged to a 25-gal. stainless steel pressure reactor equipped with temperature controls and an agitator:

Vinyl acetate -----	G.	5
Water -----	36,000	
Igepal 887 -----	33,000	
Igepal 630 -----	1,020	
Ferrous ammonium sulfate (1% solution) -----	510	10
Sodium lauryl sulfate -----	5	5

After purging with nitrogen and ethylene, 105 g. potassium persulfate was added to the mixture. The agitator was set at 300 r.p.m. and the kettle pressurized with ethylene to 40 atm. After reaching equilibrium and after heating to 50° C. the agitation was reduced to 195 r.p.m. and polymerization was started by adding 20 cc. of a 0.5% solution of Formopon. During the polymerization 139 g. of maleic acid and 1800 g. of allyl glycidyl ether were added incrementally, in addition to 136 g. of potassium persulfate which was also added incrementally as needed. The polymerization was complete after 4½ hrs. The latex was cooled and neutralized with ammonia to a pH of 5.2. The latex had the following properties:

Solids—47.3

Ethylene in copolymer—21%

T₁₃₅ = -5° C.

T₄ = +4° C.

Intrinsic viscosity = 0.29 (100 ml./g., benzene, 30° C.)

Particle size = less than 0.18μ

The above-described latex was diluted to 10% solids, 2% sodium acetate (based on the weight of solids) was added and the latex applied to a non-woven web of 50% viscose rayon-50% cellulose acetate fibers, using a Butterworth three-roll padder. The latex was applied at the rate of about 18% (solids) based on the weight of the web. The web was then dried and cured on a pin frame at 300° F. for 6 min., and then 5% zinc fluoborate was added and curing continued at 350° F. for 5 min.

The cured web was then subjected to a 1-hr. accelerated washing test at a temperature of 160° F. employing an AATCC Launder Ometer, in accordance with Standard Test Method 61-1962 as set forth on pages B-76 and B-77 of the 1962 Technical Manual of the American Association of Textile Chemists and Colorists, with the sample being tumbled in a stainless steel cylinder containing 100 stainless steel balls and the wash solution. The web was found to be completely intact after the washing operation.

EXAMPLE 2

The above-described procedures with respect to the

preparation of a bonded non-woven fiber web were repeated, except that the binder used was a vinyl acetate homopolymer latex initially having a solids content of 48.7 and a pH of 6-6.5, and no zinc fluoborate was added. At the end of the washing test, the web was no longer intact and had failed the test.

In the characterization of the interpolymer of Example 1, T₁₃₅ is the temperature at which the torsional modulus is 135,000 lbs./in.², and T₄ the temperature at which the torsional modulus is 10,000 lbs./in.² determined according to ASTM-D1043-61T.

It will be apparent that various changes and modifications may be made in the embodiments of the invention described above, without departing from the scope of the invention, as defined in the appended claims, and it is intended, therefore, that all matter contained in the foregoing description shall be interpreted as illustrative only and not as limitative of the invention.

What is claimed is:

1. A nonwoven fabric formed from a loosely assembled web of fibers bonded together with a binder comprising an emulsion polymerized interpolymer of vinyl acetate-ethylene-allyl glycidyl ether, the ethylene in said interpolymer being, before polymerization, unsubstituted monomeric ethylene, said interpolymer containing 5 to 40% by weight ethylene and a minor amount of allyl glycidyl ether of at least about 0.5% up to about 10% by weight based on the vinyl acetate.

2. A nonwoven fabric formed from a loosely assembled web of fibers bonded together by a binder deposited from a vinyl acetate-ethylene-allyl glycidyl ether interpolymer latex comprising an aqueous medium having colloiddally suspended therein a vinyl acetate-ethylene-allyl glycidyl ether interpolymer, the ethylene in said interpolymer being, before polymerization, unsubstituted monomeric ethylene, said interpolymer containing 5 to 40% by weight ethylene and a minor amount of allyl glycidyl ether of at least about 0.5% up to about 10% by weight based on the vinyl acetate.

3. A nonwoven fabric as defined in claim 2 wherein said vinyl acetate-ethylene allyl glycidyl ether interpolymer in said aqueous medium has a particle size of 0.1μ to 2μ.

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SAMUEL H. BLECH, Primary Examiner

U.S. Cl. X.R.

117-138.8, 161; 260-8, 17.4, 27, 29.6, 30.6, 31.4, 318, 78.5, 79.3, 80.72, 873

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,526,538 Dated September 1, 1970

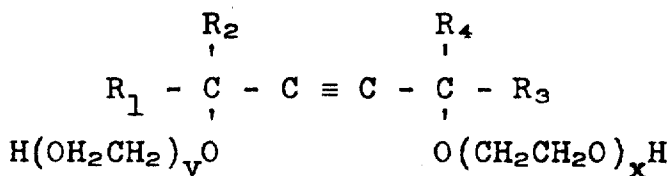
Inventor(s) M. K. Lindemann and R. P. Volpe

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

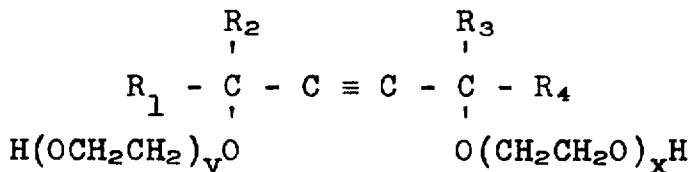
Col. 2, line 17, after "and" should be inserted -- a --;
line 45, "base" should read -- based --;
line 49, "vxinyl" should read -- vinyl --;
line 59, "pressure" should read -- pressures --.

Col. 3, line 71, "poyloxyethylene" should read -- polyoxyethylene --

Col. 4, in the formula



should read



Col. 5, line 21, after "acid" insert -- used --;
line 67, after "vinyl" insert -- acetate copolymers. The reaction temperature can be --.

Col. 6, line 2, "variable" should read -- variables - .

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Attest:

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Attesting Officer

WILLIAM E. SCHUYLER, JR.
Commissioner of Patents