

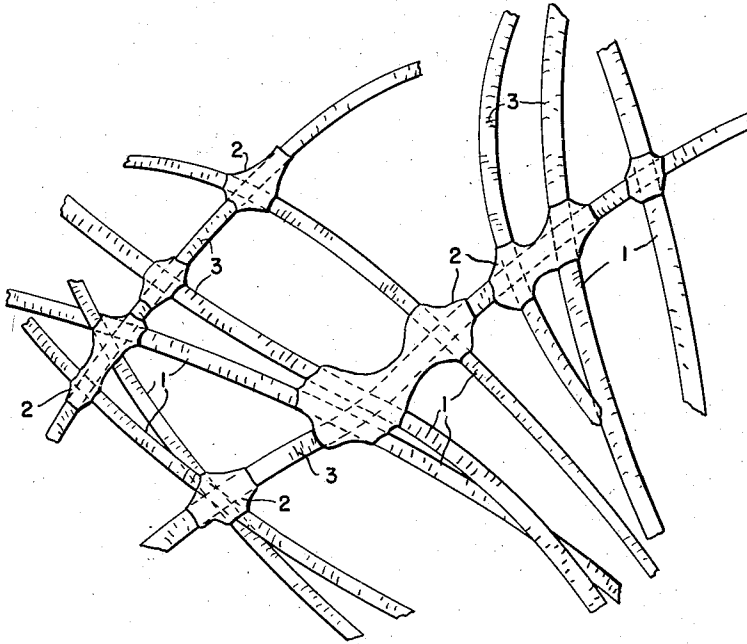
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PRODUCTION OF NON-WOVEN FABRICS

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PRODUCTION OF NON-WOVEN FABRICS

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A non-exclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

This invention concerns the production of fabrics of the non-woven category, that is, fabrics wherein the fibrous elements which compose the fabric are joined by bonding with an adhesive composition rather than by weaving or knitting. In particular, the invention concerns the production of non-woven fabrics wherein the individual fibrous elements are, for the most part, bonded only at points of contact between individual fibers and the greatest part of each fiber is essentially free from bonding agent. Because the fibers are bonded in such manner the fabrics of this invention exhibit many of the characteristics of a typical woven fabric. Thus the products of the invention combine good tensile strength with porosity, air-permeability, flexibility, softness and the appearance and handle of a woven fabric. Further objects and advantages of the invention will be obvious from the following description.

The attached drawing depicts the microscopic appearance of a non-woven fabric prepared in accordance with the invention.

In the single figure of the drawing there is illustrated an enlarged view of a typical non-woven fabric, produced according to this invention, in which overlapping fibers matted in a random pattern are cemented to one another by spots of adhesive only at the points of intersection but are free of adhesive along their lengths between such points.

Non-woven fabrics in various forms have been known and used since ancient times. Fur and wool felts, for example, have been used in clothing manufacture for centuries. In more recent times, various procedures have been developed for producing non-woven fabrics which have less of the boardiness and denseness of felts. At present large yardages of fabrics are prepared in the following manner: Selected fibers are formed into a web which is impregnated with a resin, such as an emulsion of polyvinyl acetate. The web is then squeezed to remove the excess resin and is subjected to heat to cure the resin. Instead of polyvinyl acetate, other adhesive materials may be employed such as solutions of cellulose xanthate, rubber cements or latices, etc. Fabrics produced by such procedures are useful for many special applications but are not truly comparable to woven fabrics. Thus much of binder composition is coated on the individual fibers so that the fabric lacks the softness, drape and other qualities of hand which are exhibited by a woven fabric. In essence, the non-woven fabrics of the prior art possess more of the characteristics of the material used as the binder than characteristics of the fibers themselves.

It has now been found that non-woven fabrics which combine the desirable properties of high tensile strength with porosity, air-permeability, softness, flexibility and good handle as are characteristic of woven fabrics, can be produced in a simple and effective manner. Briefly

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described, the process of the invention involves the following steps: A batch of fibers is coated with a first bonding composition and another batch of fibers is coated with a second bonding composition, capable of reacting with the first composition. The two batches of fibers are then intimately blended and the mixture of the different fibers is formed into a web. The web is then subjected to heat to cause a reaction between the two bonding compositions at the places where fibers coated with the respective compositions come into contact. Thus the individual fibers of the web are spot-bonded to one another. After the curing step, the web is washed to remove unreacted bonding material from the fibers so that the fibers then are essentially free from any bonding composition except for the deposits of inter-reacted material at points of contact between individual fibers. Also, the interstices between fibers are completely free from binder material. Since the major part of each fiber is free from any bonding composition, the fabric exhibits typical textile characteristics, that is, characteristics of the fibers rather than the bonding composition.

The bonding action that takes place in the process is further explained as follows, having reference to specific materials which can be used in the treatment. One batch of fibers is coated with a polyepoxide, another batch of fibers is coated with a polyamide. The coated fibers are intimately blended and formed into a web. At this point one has a mat of interlaced fibers some coated with polyepoxide, others coated with the polyamide. When the web is heated to effect the bonding, a co-polymerization takes place at the points where the polyepoxide-coated fibers and the polyamide-coated fibers come into contact. At such points the polyepoxide and polyamide contact one another and co-polymerize forming a high molecular weight resin which effectively cements the fibers together at such points of contact. At those parts of the individual fibers where there is no contact with a differently coated fiber essentially no polymerization takes place. Thus the portions of the polyepoxide-coated fibers and the portions of the polyamide-coated fibers which are not in contact with one another remain in essentially their original state. When the web is subsequently treated to remove excess bonding agent, these portions of the polyepoxide and polyamide are removed. As a net result, there is produced a fabric wherein individual fibers are united with one another at points of individual fiber contact whereas the major portion of each fiber is essentially free from bonding agent. Moreover, interstices between fibers are completely free from bonding material. Microscopic studies of the fabrics of the invention have shown that this result actually takes place. Under the microscope, the wool fibers can be seen as joined to one another at points of contact by minute drops of co-polymer whereas the parts of the fibers not in contact with one another are free from resin and the original scales of the wool fibers can be discerned.

The significance of the process of this invention is that the fibers are effectively bonded together so that the fabric has good mechanical properties such as tensile strength, resistance to abrasion, and other properties dependent on adequate interlocking of individual fibers. Moreover, since the fibers are cemented together only at points of contact and the major part of each fiber is free from bonding agent, the fabric has a porous, air-permeable structure and displays the softness, flexibility and drape qualities of a textile. In essence, the properties of the fabric represent in the aggregate the properties of the fibers, not the properties of the bonding agent.

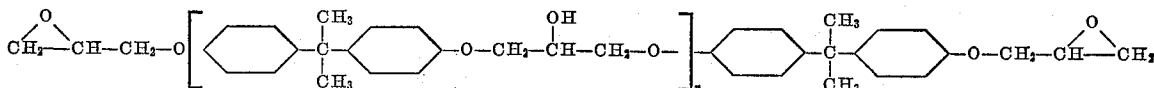
In the preferred modification of the invention, the separate batches of fiber are coated with a polyepoxide and a polyamide, respectively. These substances have the characteristics that when the web of blended, coated fiber

is subsequently subjected to heat, the individual resins will copolymerize at points where they contact one another but neither of the substances will polymerize further (by itself) to any significant degree. This of course means that the coatings on the non-contacting portions of the fibers can be readily removed after completion of the heat curing step. Also these substances will not permanently combine with most fibers to any significant extent. Where wool fibers are used, the polyepoxide will combine to some extent with the wool so that on subsequent extraction all of the polyepoxide cannot be removed. However, the larger part is removable and the portion that remains will not cause any undesirable characteristics in the end product.

Instead of using a polyepoxide and a polyamide, other pairs of bonding agents may be used which are characterized by an ability to copolymerize under the mild heating conditions used in the curing stage and which are also characterized by not polymerizing to any substantial extent by themselves under the same conditions of temperature. Also the agents should preferably be of the type which do not react to any substantial extent with the fibers themselves. Illustrations of materials which meet these qualifications are set forth below:

Where the agent applied to one batch of fibers is polyepoxide, the other agent may be any polymer of that type which has units which contain active hydrogen atoms, for example in the form of hydroxyl, amino, imino, or amide radicals. Typical polymers in this category are polyamides, polyalkyleneimines, polyvinyl alcohol, polyacrylamide, esters of cellulose, especially partially esterified celluloses, etc.

Instead of applying a polyepoxide to a batch of the fibers, one can use other agents for example, a polyurethane or a polyalkyleneimine. Where a polyurethane is used with one batch of fibers, the other batch is coated



with a polymer containing active hydrogen atoms, for example a polyamide, polyalkyleneimine, polyvinyl alcohol, polyacrylamide, or cellulose ester. Where a polyalkyleneimine is used to coat one batch of fibers, the other batch may be coated with other polymers containing active hydrogen atoms such as a polyamide, polyvinyl alcohol, polyacrylamide, ester of cellulose, etc.

As noted above, it is preferred to use a polyepoxide for coating one batch of the fibers. The polyepoxides are organic compounds having at least two epoxy groups per molecule and may be saturated or unsaturated, aliphatic, cycloaliphatic, aromatic or heterocyclic and may be substituted with non-interfering substituents such as hydroxyl groups, ether radicals, and the like. The polyepoxides may be monomeric or polymeric. It is generally preferred to employ polyepoxide polyethers. These compounds may be prepared, as well known in the art, by reacting a polyol, for example, a polyhydric alcohol or phenol, with at least 2 moles of halogen-containing epoxide per mole of polyol. A preferred example of the halogen-containing epoxide is epichlorohydrin; others are 3-chloro-1,2-epoxybutane, 3-bromo-1,2-epoxyhexane, and 3-chloro-1,2-epoxyoctane. Thus by way of example epichlorohydrin is reacted with a polyol such as glycerol, diglycerol, propylene glycol, ethylene glycol, diethylene glycol, butylene glycol, hexanetriol, sorbitol, mannitol, pentanetriol, pentaerythritol, dipentaerythritol, tripentaerythritol, polyglycerol, dulcitol, inositol, carbohydrates, methyltrimethylol propane, 2,6-octanediol, tetrahydroxycyclohexane, 2-ethyl hexanetriol-1,2,6-glycerol methyl ether, glycerol allyl ether, polyvinyl alcohol, polyallyl alcohol, resorcinol, catechol, hydroquinone, 4,4'-dihydroxy diphenyl ether, methyl resorcinol, 2,2-bis (parahydroxyphenyl) propane, 2,2-bis (parahydroxyphenyl) butane, 4,4'-dihydroxybenzophenone, bis (parahydroxyphenyl) ethane, 1,5-dihydroxy-

naphthalene, 1,4-dihydroxycyclohexane, bis (2,2'-dihydroxy-dinaphthyl) methane, etc. Illustrative examples of polyepoxides are as follows: 1,4-bis (2,3-epoxypropoxy) benzene, 1,3-bis (2,3-epoxypropoxy) benzene, 4,4'-bis (2,3-epoxypropoxy) diphenyl ether, 1,8-bis (2,3-epoxypropoxy) octane, 1,4-bis (2,3-epoxypropoxy) cyclohexane, 4,4'-bis (2-hydroxy-3,4-epoxybutoxy) diphenyl dimethylmethane, 1,3-bis (4,5-epoxypentoxy)-5-chlorobenzene, 1,4-bis (3,4-epoxybutoxy)-2-chlorocyclohexane, diglycidyl thioether, diglycidyl ether, ethylene glycol diglycidyl ether, propylene glycol diglycidyl ether, diethylene glycol diglycidyl ether, resorcinol diglycidyl ether, 1,2,5,6-diepoxycyclohexane, 1,2,3,4-tetrakis (2-hydroxy-3,4-epoxybutoxy) butane, 2,2-bis (2,3-epoxypropoxyphenyl) propane, glycerol triglycidyl ether, mannitol tetraglycidyl ether, pentaerythritol tetraglycidyl ether, sorbitol tetraglycidyl ether, etc. It is evident that the polyepoxide polyethers may or may not contain hydroxy groups, depending primarily on the proportions of halogen-containing epoxide and polyol employed. Polyepoxide polyethers containing polyhydroxyl groups may also be prepared by reacting, in known manner, a polyhydric alcohol or phenol with a polyepoxide. Illustrative examples are the reaction product of glycerol and diglycidyl ether, the reaction product of sorbitol and bis (2,3-epoxy-2-methylpropyl) ether, the reaction product of pentaerythritol and 1,2,4,5-diepoxypentane, the reaction product of 2,2-bis (parahydroxyphenyl) propane and bis (2,3-epoxy-2-methylpropyl) ether, the reaction product of resorcinol and diglycidyl ether, the reaction product of catechol and diglycidyl ether, and the reaction product of 1,4-dihydroxycyclohexane and diglycidyl ether. A particularly preferred type of polyepoxide polyether is that prepared by reacting epichlorohydrin with 2,2-bis (parahydroxyphenyl) propane. The structure of these compounds is illustrated by the formula

wherein n varies between zero and about 10, corresponding to a molecular weight about from 400 to 8,000.

As explained above, when one batch of fibers is coated with a polyepoxide, the other batch is preferably coated with a polyamide. Polyamides are prepared in known manner by (a) condensation of a polybasic acid and a polyamine or (b) by self-polymerization of an amino acid. Regarding the procedure of category (a), there follows illustrative examples of acids and amines which may be condensed to form polyamides:

Acids—sebacic, octadecanedioic, adipic, suberic, azelaic, undecanedioic, malonic, glutaric, pimelic, brassylic, tetradecanedioic, oxalic, beta-methyl adipic, diglycolic, p-phenylenediacetic, diphenic, 1,2-cyclohexane dicarboxylic, etc.

Amines—ethylene diamine, propylene diamine, tetramethylene diamine, pentamethylenediamine, hexamethylenediamine, octamethylenediamine, decamethylenediamine, 3-methylhexamethylene diamine, triglycol diamine, 3,3'-diaminodipropyl ether, p-xylylene diamine, piperazine, diethylene triamine, etc.

Regarding category (b), referred to above, polyamides may be prepared by self-polymerization of such compounds as 6-aminohexanoic acid, 9-aminononanoic acid, 11-aminoundecanoic acid, 17-aminoheptadecanoic acid, and the like.

Preferred types of polyamides for use in the invention are those prepared by condensation of a diamine, such as ethylene diamine, with polymerized high molecular weight fatty acids. Products of this type are disclosed by Cowan et al., "Oil & Soap," vol. 21, No. 4, pp. 101-107 (April 1944); Falkenburg et al., "Oil & Soap," vol. 22, No. 6, pp. 143-148 (June 1945). These authors disclose, for instance, the production of polyamides by heating a mixture of ethylene diamine with heat-polymer-

lized fatty acids derived from drying or semi-drying oils such as soybean oil, cottonseed oil, corn oil, linseed oil, etc. The polyamides may also be produced by heating the diamine with methyl esters of the polymerized fatty acids, e.g., methyl esters of dimerized soybean fatty acids.

The amount of binding agent applied to the fibers can varied widely. Usually it is desired that after curing and removal of unreacted agents, the fabric contain on the order of 1 to 10% of copolymer. Depending on the use to be made of the fabric higher amounts of copolymer can be employed although the fabric will tend to be stiffer and less like a woven fabric. When the agents are initially placed on the fibers, they are usually employed in excess of the proportion desired in the finished product, the excess being removed during the described washing or extracting process. It is obvious that the proportion of each agent applied to the separate batches of fiber need not be the same. The respective proportions of the individual agents may be adjusted to provide the desired properties in the resulting deposits of co-polymer which form the spot bonds between individual fibers. For example where the agents used are a polyepoxide and a polyamide, it is generally preferred to employ a higher proportion of polyamide than polyepoxide so that the spot bonds will provide more flexible joints. Thus the amounts of these agents coated on the fibers may be on the order of 1.5 to 9 parts of polyamide for each part of polyepoxide.

The process of this invention may be used to prepare fabrics from any type of fiber, for example wool, silk, linen, cotton, nylon, regenerated cellulose, cellulose acetate, synthetic fibers from proteins such as corn, soybeans, keratins, etc., hair, fur, mohair, jute, coir, sisal, asbestos, glass fibers, wood fibers, bark fibers, and so forth. It is obvious that the fibers need not be the same and blends of different fibers can be used as desired. Since the curing of the coated and blended fibers is accomplished by application of heat at a temperature of about 50 to 100° C. it is obvious that the fibers selected for the process should be of the kind which are not damaged by such temperatures.

In coating the batches of fibers with the different binding agents, it is preferable to apply these materials in the form of liquid preparations such as solutions or emulsions. Aqueous solutions may be used, for example, where the agents are soluble in this medium. In many cases other volatile solvents may be required such as acetone, benzene, alcohol, ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, dioxane, etc., depending on the properties of the agents in question. It is often convenient to apply the agents in the form of aqueous emulsions prepared by application of any of the known emulsification techniques. A preferred procedure is to dissolve the binding agent in a suitable quantity of alcohol or other volatile organic solvent and add this solution to water with vigorous agitation. In this way the contact of the solution with the water will precipitate the agent in minute particles which will be relatively easy to emulsify. To assist in forming and maintaining the emulsion, one may add a small proportion of a conventional emulsifying agent. For such purpose one may employ agents such as soaps, long chain alkyl sodium sulphates or sulfonates, long chain alkyl benzene sodium sulphonates, esters of sulphosuccinic acid, etc., typical examples being sodium lauryl sulphate, sodium dodecane sulphonate, sodium alkyl (C₁₀-C₂₀) benzene sulphonate, sodium dioctylsulphosuccinate, etc. Preferably, emulsifying agents of the non-ionic type are used, for example, the reaction products of ethylene oxide with fatty acids, polyhydric alcohols, alkyl phenols, and so forth. Typical examples of such agents are a polyoxyethylene ether of sorbitan monolaurate containing 16 oxyethylene groups per mol, a polyoxyethylene ether of sorbitan monostearate containing 20 oxyethylene groups per mol, a distearate of poly-

oxyethylene ether of sorbitol containing 40 oxyethylene groups per mol, iso-octyl phenyl ether of polyethylene glycol, and so forth. Cationic emulsifying agents may also be used for example long chain alkyl trimethyl ammonium chlorides, bromides, or methosulphates. Other suspending agents such as gums, gelatin, pectin, soluble starch, dextrans, etc. can of course be employed to keep the bonding agents in suspension.

The coating of the fibers with the solutions or emulsions can be effectuated in any conventional manner such as by spraying, immersing, brushing or the like. The treated fibers are then preferably dried to remove water or other medium used in the solution or emulsion. The coated fibers are then blended to form a uniform composite mat of fibers. This blending can be carried out by carding or other methods well known in the textile arts. The blended fiber is then formed into a web which is then subjected to a heat curing operation to effectuate the spot bonding of individual fibers. During the curing operation, the web may be subjected to compression between platens, rolls or the like to obtain a fabric of the desired thickness and density. The temperature required in the curing step will depend on the chemical nature of the pair of bonding agents used and especially their reactivity. In many cases a temperature about from 50 to 100° C. will be effective. The web is subjected to heat until an effective bond is attained between individual fibers. Such conditions as time and temperature of cure will vary depending on such factors as the chemical nature of the agents used and the degree of copolymerization desired to be attained. In any particular case the proper conditions can be readily determined by conducting pilot trials at varying times and temperatures and noting the conditions which give the desired results.

The heat curing is generally carried out by placing the web in an oven where it is subjected to hot air at the desired temperature. In many cases curing can be accomplished more rapidly by subjecting the web to direct contact with steam or hot water.

When the web has been heat cured as above described it is subjected to a washing treatment to remove from the fibers the unreacted bonding agents. This washing or extraction as it may be termed involves treating the web with a liquid which will dissolve the unreacted materials. In many cases a washing with water and soap or synthetic detergent will remove the resins. For this purpose one may employ any of the surface active agents above enumerated as emulsifiers for forming the emulsions of the bonding agents. In most cases more efficient results are attained by washing the cured web with a volatile organic solvent in which the unreacted materials are soluble, for example, methyl alcohol, ethyl alcohol, propyl alcohol, isopropyl alcohol, the various isomeric butyl alcohols, acetone, methyl ethyl ketone, benzene, toluene, cyclohexane, hexane, gasoline or other petroleum hydrocarbons, carbon tetrachloride, chloroform, ethylene dichloride, trichlorethylene, dioxane, ethyl butyl ether, isopropyl ether, ethyl acetate, methyl propionate, ethyl acetal, ethyl formal, formamide, dimethyl formamide, furfural, monoethers of ethylene glycol such as the methyl, ethyl, propyl, butyl, and phenyl ethers, monoethers of diethylene glycol such as the methyl, ethyl, and butyl ethers, etc.

The invention is further demonstrated by the following example.

Example

A batch of wool fibers was soaked in an aqueous solution containing about 1 to 2% of polyepoxide polyether, being a reaction product of epichlorhydrin and 2,2 (bis-parahydroxyphenyl) propane. The material had a melting point of 8 to 12° C., a viscosity of Z_5-Z_6+ on the Gardner-Holdt scale at 25° C., and an epoxide equivalent of 190-210. The wool was then removed from the solution and dried.

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Another batch of the same wool was soaked in an aqueous solution containing about 1 to 2% of a polyamide being a condensation product of ethylene diamine and heat-polymerized high molecular weight fatty acids. The polyamide was a viscous liquid at ordinary temperatures with a viscosity of A_3-A_1 on the Gardner-Holdt scale at 25° C. and a specific gravity of 0.99 at 25° C. The wool was then removed from the solution and dried.

The two batches of dried, impregnated wool were then carded to form an intimate blend and formed into a thin web. The web was then pressed between glass plates while heated in an oven at 100° C. for one hour. The resulting fabric was washed thoroughly with soap and water to remove unreacted materials.

The fiber was then examined under a microscope to determine its structure. The microscopic appearance (enlarged 150 times) of the fabric is depicted in the figure in the annexed drawing. Referring to this figure, it can be seen that the individual fibers 1 are bonded to one another at points of contact by spot bonds 2 consisting of polyepoxide-polyamide copolymer. The portions of the fibers except for intersecting points are essentially free from polyepoxide and polyamide as the typical scales 3 can be seen in many of the fibers.

Having thus described the invention, I claim:

1. A process for producing a fiber-like, porous, air-permeable flexible sheet material which comprises coating a first batch of fibers with a first bonding composition, coating a second batch of fibers with a second bonding composition of different chemical constitution from said first bonding composition, blending said first and second batches of fibers to form a uniform mass of fibers, compacting said mass into a thin web of intermingled fibers, subjecting the web to heat to cause spot bonding at points

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where fibers of the first batch contact fibers of the second batch, and washing the web to remove the bonding compositions from non-contacting portions of the fibers, the said bonding compositions having the ability of retaining their original state with essentially no polymerization when subjected to heat in the absence of one another but having the ability to react to form a resinous copolymer when subjected to heat in presence of one another.

2. The process of claim 1 wherein one of the bonding compositions contains a polyepoxide and the other contains a polyamide.

3. A process for producing a fabric-like, porous, air-permeable flexible sheet material which comprises coating a first batch of fibers with a polyglycidyl ether of a polyhydric alcohol, coating a second batch of fibers with a polyamide of an aliphatic diamine and a polybasic aliphatic acid, blending said first and second batches of fibers to form a uniform mass of fibers, compacting said mass into a thin web of intermingled fibers, subjecting the web to heat to cause spot bonding at points where fibers of the first batch contact fibers of the second batch by formation of a polyglycidyl ether-polyamide copolymer, and washing the web to remove the polyglycidyl ether and polyamide from non-contacting portions of the fibers.

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