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(54) Flooring felt compositions and method for preparing the same.

(57) Flooring felt compositions having dimensional and thermal stability and resistance to water and high humidity as well as the necessary mechanical properties to provide satisfactory backing for the thermoplastic vinyl resins ordinarily used as floor coverings can be prepared by forming an anionic aqueous fiber dispersion of from about 4 to about 55% and preferably from about 8 to about 35% of water dispersible spurted polyolefin fibers, from about 2 to about 30% of wood pulp fibers, from about 2 to about 20% water dispersible glass fibers, from about 1 to about 10% of an anionic polyacrylamide resin, from about 0 to about 50% of an inorganic filler, from about 5 to about 25% of an anionic, elastomeric polymeric binder and from about 1 to about 10% of a cationic resin (all percentages of the above components are by weight, based on the dry weight of flooring felt composition) and then passing the resulting dispersion through a paper making machine to form a fibrous sheet material.

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Flooring Felt Compositions and Method for Preparing the Same

This invention relates to flooring felt compositions and to a process for preparing them. More particularly, the invention relates to flooring felt compositions having the dimensional and thermal stability and the resistance to water and high humidity of asbestos-based compositions, but which additionally have the overall mechanical properties necessary to provide a satisfactory backing for the thermoplastic vinyl resins ordinarily used as floor coverings and to a process for making the same.

As is well known, cellulosic fibers have traditionally been a major component of fibrous compositions used as backing materials for vinyl floor coverings and linoleum. However, these floor covering products have not always exhibited satisfactory dimensional stability, and it has additionally been known that this problem of dimensional stability could be overcome through utilization of asbestos, in whole or in part, in place of the cellulosic fibers. However, due to the health hazards associated with the use of asbestos, considerable effort has more recently been directed in the art to the development of asbestos-free formulations having the desirable properties of the asbestos-based compositions. For example, considerable work has been done with water dispersible glass fibers, which are thermally stable, strong and flexible and which provide dimensionally stable webs, but it is not apparent from the art that known compositions containing such fibers have the required balance of physical properties presently considered as necessary for a satisfactory backing material.

U.S. Patent 4,274,916 discloses a backing material comprised of polypropylene fibers, wood pulp fibers, glass fibers, filler, polymeric binder and a cationic quaternary modified acrylic polymer. Optionally, a wet strength resin, including cationic resins, can be added.

Now, in accordance with this invention, such a backing material has been found, said material being an asbestos-free flooring felt composition comprising from about 4 to about 55% and preferably from about 8 to about 35% of water dispersible spurted polyolefin fibers, from about 2 to about 30% of wood pulp fibers, from about 2 to about 20% of water dispersible glass fibers, from about 0 to about 50% of an inorganic filler, from about 5 to about 25% of an anionic, elastomeric polymer binder, from about 1 to about 10% of an anionic polyacrylamide resin containing from about 2 to about 15% by weight of acrylic acid units and from about 1 to about 10% of the cationic resin reaction product of epichlorohydrin and a poly(diallylamine) or an aminopolyamide derived from a dicarboxylic acid and a polyalkylene polyamine having two primary amine groups and at least one secondary or tertiary amine group. All percentages of the above components are by weight based on the dry weight of the flooring felt composition.

The foregoing compositions will have a density of from about 0.35 to about 0.80 gm/cc, a dry tensile strength greater than 1000 psi, a hot (177°C.) tensile strength greater than 300 psi, where the composition has a basis weight of from about 160 to about 340 pounds/3000 sq. ft. and a thickness of from about 0.01 to about 0.04 inch (10 to 40 mils). Moreover, these compositions are dimensionally stable, thermally stable, resistant to water, sufficiently flexible to permit easy installation of the floor covering product backed therewith, and resistant to discoloration upon exposure to heat and light.

The process of preparing the compositions of this invention comprises the steps of forming an anionic aqueous fiber dispersion comprising from about 4 to about 55% and preferably from about 8 to about 35% of water dispersible spurted polyolefin fibers, from about 2 to about 30% of wood pulp fibers, from about 2 to about 20% of water dispersible glass fibers and from about 1 to about 10% of an anionic polyacrylamide resin containing from about 2 to about 15% by weight of acrylic acid units, adding to said anionic aqueous fiber dispersion from 0 to about 50% of an inorganic filler, from about 5 to about 25% of an anionic elastomeric polymeric binder and from about 1 to about 10% of the cationic resin reaction product of epichlorohydrin and a poly(diallylamine) or an aminopolyamide derived from a dicarboxylic acid and a polyalkylene polyamine having two primary amine groups and at least one secondary or tertiary amine group (all percentages of the above components being by weight based on the dry weight of the flooring felt composition) and then passing the resulting dispersion through a papermaking machine to form a fibrous sheet material.

The water dispersible spurted polyolefin fibers which are used in accordance with this invention typically can be prepared by a process wherein the polyolefin is dispersed in a liquid which is not a solvent for the polypropylene at its normal boiling point, heating the resulting dispersion at superatmospheric pressure to dissolve the polymer and then discharging the resulting solution into a zone of reduced temperature and pressure to form the fibrous product. The liquid in which the polyolefin is dispersed may be a halogenated hydrocarbon such as methylene chloride, chloroform or carbon tetrachloride, an aromatic hydrocarbon such as benzene, toluene or xylene, an aliphatic hydrocarbon such as pentane or hexane, or an alicyclic hydrocarbon such as cyclohexane. Mixtures of these solvents may be used, and water may be present when it is desired to form an emulsion of the polyolefin. Moreover, the pressure generated by the solvent vapors may, and normally will, be augmented by a pressurized inert gas such as nitrogen or carbon dioxide.

The temperature to which the dispersion of the polyolefin in the solvent is heated to form a solution of the polyolefin will depend upon the particular solvent used but should be sufficiently high to effect dissolution of the polypropylene. Temperatures in the range of about 100° to about 225°C. ordinarily will be used, and the concentration of the polyolefin in the resulting solution normally will be from about 5 to about 40% by weight. The pressure on the polyolefin solution may be from about 600 to about 1500 p.s.i., preferably from about 900 to about 1200 p.s.i. The orifice through which the solution is discharged will have a diameter of from about one-half to about fifteen millimeters and a length to diameter ratio of from about 1.0 to 10:1.

The polyolefin fibers shown in the examples are spurted polypropylene and combinations of spurted polypropylene and polyethylene fibers. The invention is applicable not only to spurted polypropylene but to admixtures of from about 50 to about 100% polypropylene and from about 0 to about 50% polyethylene, based on the dry weight of the entire composition. Moreover, spurted fibers prepared from polyethylene and copolymers of ethylene or propylene and other 1-olefins such as

1-butene, 4-methyl-pentane-1 and 1-hexene, and mixtures of any of the aforementioned polymers may also be used in accordance with this invention.

The wood pulp fibers used in the flooring felt compositions of this invention are anionic and are preferably of the bleached softwood pulp variety, for example, the Rayonier bleached softwood kraft (RBK) pulp fibers shown in the examples. These fibers have lengths of from about 0.05 to about 0.2 inch, with length to width ratios of about 100:1 or more. Also useful are hardwood pulp fibers, such as Weyerhaeuser bleached hardwood kraft (WBK) pulp fibers. Ordinarily, these fibers are used in combination with RBK pulp fibers, a 50:50 by weight admixture being highly satisfactory.

The water dispersible glass fibers of the flooring felt compositions are characterized by very high tensile strengths and densities, excellent dimensional stability, thermal stability and resistance to water and moisture. They preferably average from about 0.1 to about 0.7 inch in length and from about 0.0002 to about 0.0006 inch in diameter.

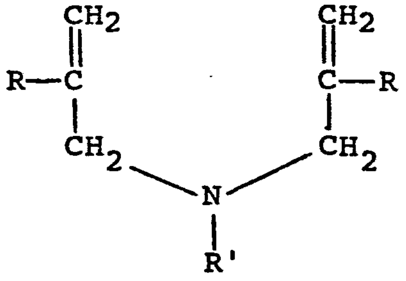
The inorganic fillers which may be used in the felt compositions of this invention are essentially water-insoluble and exemplified by the clay filler used in the examples. Alternative fillers are materials such as calcium carbonate, talc, magnesium silicate, calcium silicate, mica, aluminum silicate and diatomaceous earth. The particle size of the fillers is such that the major amount will be below 50 microns in diameter, with the average diameter generally being above 0.1 micron and preferably from about 0.1 to about 20 microns.

The anionic elastomeric polymer binder component of the felt compositions is illustrated in the examples by the 50:50 styrene-butadiene copolymer containing carboxylic functionality. The ratio of the styrene component to the diene monomer may be varied from about 40:60 to about 90:10. Also operable are ethylene-acrylic acid copolymers, the corresponding methacrylic acid copolymers, polychloroprenes containing carboxylic functionality, alkylacrylate-acrylonitrile-acrylic acid copolymers, the corresponding alkylmethacrylate copolymers, butadiene-acrylonitrile-methacrylic acid copolymers, the corresponding acrylic acid copolymers and vinyl acetate-acrylic acid copolymers. For ease of application and use, the elastomeric polymer binder will be in the form of a latex, that is, an aqueous colloidal dispersion, containing from about 35 to about 60% solids, usually about 50%. The latex will normally already contain, or have added thereto prior to use in accordance with this invention, a stabilizer to retard thermal discoloration of the polymer.

The anionic polyacrylamide resin component used in accordance with this invention is a copolymer containing from about 85 to about 98% by weight of acrylamide units and from about 2 to about 15% by weight of acrylic acid units. The copolymer is normally obtained by copolymerization of acrylamide and acrylic acid, as illustrated in Example 4. Comparable products can be prepared, however, by partial hydrolysis of polyacrylamide or a poly(acrylamide-co-alkyl acrylate), such as a copolymer of acrylamide with ethyl acrylate. Any of these polyacrylamides are prepared by conventional methods for the polymerization of water-soluble monomers and preferably have molecular weights less than about 25,000, for example, in the range of from about 10,000 to about 20,000.

One of the cationic resins used in this invention is the reaction product of epichlorohydrin and a poly(diallylamine). The preparation of such a product is shown in Example 2.

Polymers having units of the above formula are obtained by polymerizing the hydrohalide salt of a diallylamine having the formula:



in which R and R' are as indicated above, in the presence of a free radical catalyst and then neutralizing the salt to give the polymer free base. In both of the above formulae, the R substitution can be the same or different, and, as stated, can be hydrogen or lower alkyl. The alkyl groups contain from 1 to 6 carbons and are preferably methyl, ethyl, isopropyl or n-butyl. R' of the formula represents hydrogen, alkyl or substituted alkyl groups. The R' alkyl groups will contain from 1 to 18 carbon atoms (preferably from 1 to 6 carbon atoms) such as methyl, ethyl, propyl, isopropyl, butyl, tert-butyl, hexyl, octyl, decyl, dodecyl, tetradecyl, and octadecyl. R' can also be a substituted alkyl group. Suitable substituents include, in general, any group which will not interfere with polymerization through a vinyl double bond. Typically, the substituents can be carboxylate, cyano, ether, amino (primary, secondary or tertiary), amide, hydrazide and hydroxyl.

Specific hydrohalide salts of the diallylamines which can be polymerized to provide the polymer units of the invention include diallylamine hydrochloride; N-methyldiallylamine hydrochloride; N-methyldiallylamine hydrobromide; 2,2'-dimethyl-N-methyldiallylamine hydrochloride; N-ethyldiallylamine hydrobromide; N-isopropyldiallylamine hydrochloride; N-tert-butyldiallylamine hydrochloride; and N-octadecyldiallylamine hydrochloride. The diallylamines and N-alkyldiallylamines are themselves prepared by the reaction of ammonia or a primary amine with an allyl halide. For example, N-methyldiallylamine can be prepared by reaction of two moles of an allyl halide, such as allyl chloride, with one mole of methylamine.

In the reaction of the poly(diallylamine) with epichlorohydrin, the latter is used in an amount ranging from about 0.5 mole to about 1.5 moles, preferably from about one mole to about 1.5 moles, per mole of secondary plus tertiary amine present in the polymer. The reaction is carried out at a temperature of from about 30° to about 80°C., preferably from about 40° to about 60°C., until the viscosity measured at 25°C. on a solution containing 20 to 30% solids is in the range of A to E, and preferably C to D, on the Gardner scale. The reaction preferably is carried out in aqueous solution to moderate the reaction, and at a pH of from about 7 to about 9.5. When the desired viscosity is reached, sufficient water is added to adjust the solids content of the resin solution to about 25% or less, and the solution is cooled to room temperature. The poly(diallylamine)-epichlorohydrin product can be stabilized against gelation by adjusting the pH of the solution to about 2 with, for example, sulfuric or hydrochloric acid.

The other cationic resin which may be used in accordance with this invention is the reaction product of epichlorohydrin and an aminopolyamide derived from a dicarboxylic acid and a polyalkylene polyamine having two primary amine groups and at least one secondary or tertiary amine group. A representative product of this type is described in Example 3. Particularly suitable dicarboxylic acids are the saturated aliphatic dicarboxylic acids containing from 3 through 10 carbon atoms such as malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, and sebacic acid. Other suitable dicarboxylic acids include terephthalic acid, isophthalic acid, phthalic acid, maleic acid, fumaric acid, itaconic acid, glutaconic acid, citraconic acid, and mesaconic acid. A number of polyalkylene polyamines can be employed. Polyalkylene polyamines can be represented as polyamines in which the nitrogen atoms are linked together by groups of the formula $--C_nH_{2n}--$ where n is a small integer greater than unity and the number of such groups in the molecule ranges from two up to about eight. Polyamines such as diethylenetriamine, triethylenetetramine, tetraethylenepentamine, and dipropylenetriamine, which can be obtained in reasonably pure form, are suitable for preparing water-soluble aminopolyamides. Other polyalkylene polyamines that can be used include methyl bis-(3-aminopropyl)amine; methyl bis-(2-aminoethyl)amine; and 4,7-dimethyltriethylenetetramine. Mixtures of polyalkylene polyamines can be used, if desired.

The temperatures employed for carrying out the reaction between the dicarboxylic acid and the polyalkylenepolyamine may vary from about 110°C. to about 250°C. or higher at atmospheric pressure. For most purposes, however, temperatures between about 160°C. and 210°C. have been found satisfactory and are preferred. In carrying out the reaction, it is preferred to use an amount of dicarboxylic acid sufficient to react substantially completely with the primary amine groups of the polyalkylenepolyamine but insufficient to react with the secondary and/or tertiary amine groups to any substantial extent. This will usually require a mole ratio of polyalkylenepolyamine to dicarboxylic acid of from about 0.9:1 to about 1.2:1. However, mole ratios of from about 0.8:1 to about 1.4:1 may be used with satisfactory results.

In converting the aminopolyamide to a cationic resin, it is reacted with epichlorohydrin at a temperature from about 45°C. to about 100°C. and preferably between about 45°C. and 70°C. until the viscosity of a 20% solids solution at 25°C. has reached about C or higher on the Gardner scale. This reaction is preferably carried out in aqueous solution to moderate the reaction. Adjustment of pH is usually not necessary. When the desired viscosity is reached, sufficient water is then added to adjust the solids content of the resin solution to the desired amount, i.e., about 10% more or less, the product cooled to about 25°C. and then stabilized by adding sufficient acid to reduce the pH at least to about 6 and preferably to about 5. Sulfuric acid is preferred. In the aminopolyamide-epichlorohydrin reaction, it is preferred to use sufficient epichlorohydrin to convert all secondary amine groups to tertiary amine groups. In general, satisfactory results may be obtained utilizing from about 0.5 mole to about 1.8 moles of epichlorohydrin for each secondary or tertiary amine group of the aminopolyamide. It is preferred to utilize from about 1.0 mole to about 1.5 moles for each secondary amine group of the aminopolyamide.

All of the individual components of the flooring felt composition of this invention, with the exception of the inorganic filler, are necessary in order to obtain the desired properties in the finished composition. The spurted polyolefin fibers are needed to provide the required dimensional stability, resistance to water and moisture and tensile strength. Less than the lower limit of 4% by weight based on the dry weight of the composition gives a product inadequate in tensile strength. On the other hand, the upper limit of about 55% merely represents a practical amount, above which no outstanding advantages are obtained. The preferred amount of water dispersible spurted polypropylene fibers is from about 8 to about 35% by weight.

The wood pulp fibers are needed for a number of reasons, one of which is to act as the dispersant for the polypropylene fibers in the aqueous medium in which the components of the flooring felt composition are brought together. In this capacity, about two parts of the wood pulp fibers per 100 parts of the polypropylene fibers is desirable. In addition to acting as the dispersant for polypropylene fibers, the wood pulp fibers provide uniformity and smoothness during sheet formation, with no formation of clumps or floc. Other dispersants are in no way comparable to wood pulp fibers in this respect. Moreover, the presence of wood pulp fibers in the flooring felt composition improves appreciably its tensile strength. At least about two percent of the wood pulp fibers is required to insure proper sheet formation, and increasing amounts up to about 30% by weight provide the desired improvement in tensile strength. The preferred range is from about 5 to about 10%.

The water dispersible glass fibers are also required to contribute to tensile strength and dimensional stability, and they also provide thermal stability to the flooring felt composition, all of which are important properties. The amount of water dispersible glass fibers to provide these properties is in the range of from about 2 to about 20% by weight based on the dry weight of the flooring felt composition. A preferred range is from about 3 to about 10% by weight.

As previously indicated, the inorganic filler is not a necessary component of the flooring felt composition. However, its presence will not only decrease the overall cost of the composition but will also increase the density and decrease the stiffness of the composition when such changes in these properties of the composition are desired. The amount of filler will ordinarily be in the range of from about 10 to about 20% by weight but should not exceed about 50%, since at higher levels it detracts from the overall balance of properties desired in the composition.

The anionic elastomeric polymer binder component is essential to the cohesion of the fibrous components and the filler, if present, and also to the water and moisture resistance of the flooring felt composition. For example, a composition containing no binder may absorb as much as twice the amount of water as a composition containing the binder. The amount of the binder should be at least about 5% up to about 25% by weight based on the dry weight of the flooring felt composition, preferably from about 10 to about 15% by weight. It is noteworthy that the effectiveness of the binder is dependent upon the presence of both the anionic and cationic resin components of the composition. In the absence of both resin components, the tensile strength of the composition is inadequate, and the same is true when either one of the resin components is not present.

Further to the anionic and cationic resin components, each is necessary, as just mentioned, and the amount required is from about 1 to about 10% by weight, preferably from about 2 to about 5%. In addition, the anionic resin functions to maintain the anionic character of the aqueous fiber dispersion up to and during the addition of the anionic, elastomeric polymer binder, and the cationic resin functions to precipitate the polymer binder on the fiber and filler, if present, components of the composition and to react with the anionic resin to form a crosslinked composition.

The particular order of addition of the various components is readily determinable by one skilled in the paper making art. A major factor is the arrangement of the available paper making equipment. A second factor is whether the water dispersible spurted polyolefin fibers and the wood pulp fibers are to be refined. Where refining is desired, these fibers may be refined either separately or together. The inorganic filler, the anionic polyacrylamide resin and anionic elastomeric polymeric binder may be added in any order, before or after, refining of the fibers. In contrast, the water dispersible glass fibers and the cationic resins are preferably not added until after any refining has been completed. Otherwise the water dispersible glass fibers may be added at any time. Regardless of whether refining has occurred, it has been found preferable to add at least some of the cationic resin after all of the anionic elastomeric polymer binder has been incorporated into the composition.

Other components may be present in minor amounts in the flooring felt compositions of this invention, being present in the aggregate in an amount no more than about 10% and usually no more than about 3 to about 5%. They are minor also in the sense that they do not directly contribute functionally to the physical properties of the compositions. Exemplary of such components are stabilizers for the polypropylene fibers and the polymer binder, antioxidants, pigments, dyes and preservatives such as mildewicides. Also present may be small amounts of defoamers, retention aids and drainage aids, all to improve the efficiency of the operation of the papermaking machine used in forming the felt compositions.

In using the flooring felts of this invention in the preparation of floor coverings, one or more layers of resinous polymeric compositions are applied to the flooring felt backing material. These resinous polymeric compositions are generally based on vinyl chloride homopolymers or copolymers, but also may be based on polyurethanes and other suitable thermoplastic polymers. Methods of preparing such layers of the resinous polymeric compositions are disclosed, for example, in U.S. 3,293,094 and U.S. 3,293,108, both to Nairn et al. The compositions of this invention may also be used to form other useful products, such as gaskets, filters and acoustical board, wherein the physical properties of the compositions are desired.

Having generally described the embodiments of this invention, the following specific examples thereof are presented. In the examples, all amounts are based on parts by weight unless otherwise noted.

Example 1

This example illustrates the preparation of a typical spurted polypropylene fiber used in accordance with this invention. One hundred eighty parts of isotactic polypropylene having an intrinsic viscosity of 2.7 dl/g in decahydronaphthalene at 135°C. and 1020 parts of pentane were charged to a closed autoclave. The contents of the autoclave were stirred and heated to 160°C., at which point the vapor pressure in the autoclave was raised to 850 p.s.i. by the introduction of nitrogen. The resulting solution was spurted from the autoclave into the atmosphere through an orifice having a diameter of one millimeter and a length of one millimeter, resulting in evaporation of the pentane solvent and formation of the polypropylene fiber product. Typically, this product will be composed of very fine filaments, of a thickness of the order of a micron, connected to one another to form a three-dimensional network. The general shape of the fibers, which have a flocculent appearance, is oblong. Their length varies from about one-half millimeter to about five centimeters and their diameter varies from about one-hundreth of a millimeter to about five millimeters. The specific surface area of these products is greater than one square meter per gram and in some cases may be greater than ten square meters per gram.

Example 2

This example shows the preparation of an epichlorohydrin-poly(diallylamine) reaction product usable as the cationic resin component in accordance with this invention. To 333 parts of methyldiallylamine was slowly added 290-295 parts of concentrated hydrochloric acid to provide a solution having a pH of 3 to 4. The solution then was sparged with nitrogen for 20 minutes and the temperature was adjusted to 50° to 60°C. An aqueous 10.7% solution of sodium bisulfite and an aqueous 10.1% solution of t-butyl hydroperoxide were simultaneously added to the reaction mixture over a period of four to five hours until the resulting polymer, poly(methyldiallylamine hydrochloride), had a reduced specific viscosity of 0.2 dl/g as measured on a one percent solution in aqueous one molar sodium chloride at 25°C. The amount of each of the sodium bisulfite and the 5-butyl hydroperoxide used was two mole percent based on the polymer repeat units.

To the above polymer solution there then was added 600 parts of aqueous four percent sodium hydroxide, and the temperature of the resulting solution was adjusted to 35°C. After addition of sufficient water to bring the solids content of the polymer solution to 22%, there was added 416.3 parts of epichlorohydrin. The temperature of the reaction mixture was maintained at about 45°C. while the Gardner viscosity of the mixture increased from less than A to B+. After the addition of about 304 parts of 36% hydrochloric acid, the reaction mixture was heated to 80°C. and maintained at this temperature with continual addition of further amounts of hydrochloric acid until the pH of the reaction mixture had stabilized at 2 for one hour. The reaction mixture then was cooled to 40°C., adjusted to a pH of 3.5-4.0 with aqueous four percent sodium hydroxide and diluted to 20% solids.

The resin product from the above process, prior to use in accordance with this invention, is preferably base activated. This is accomplished by adding 18 parts of water and 12 parts of one molar sodium hydroxide solution to each 10 parts of the 20% solids solution of the resin. The resulting five percent solids solution, after aging for 15 minutes, should have a pH of 10 or higher. Additional sodium hydroxide should be added, if necessary, to obtain this level of pH.

Example 3

The present example illustrates the preparation of a typical epichlorohydrin-aminopolyamide reaction product also utilizable as the cationic resin in accordance with this invention. Diethylenetriamine in the amount of 0.97 mole was added to a reaction vessel equipped with a mechanical stirrer, a thermometer and a reflux condenser. There then was gradually added to the reaction vessel one mole of adipic acid with stirring. After the acid had dissolved in the amine, the reaction mixture was heated to 170°-175°C. and held at that temperature for one and one-half hours, at which time the reaction mixture had become very viscous. The reaction mixture then was cooled to 140°C., and sufficient water was added to provide the resulting polyamide solution with a solids content of about 50%. A sample of the polyamide isolated from this solution was found to have a reduced specific viscosity of 0.155 dl/g when measured at a concentration of two percent in a one molar aqueous solution of ammonium chloride. The polyamide solution was diluted to 13.5% solids and heated to 40°C., and epichlorohydrin was slowly added in an amount corresponding to 1.32 moles per mole of secondary amine in the polyamide. The reaction mixture then was heated at a temperature between 70° and 75°C. until it attained a Gardner viscosity of E-F. Sufficient water next was added to provide a solids content of about 12.5%, and the solution was cooled to 25°C. The pH of the solution then was adjusted to 4.7 with concentrated sulfuric acid. The final product contained 12.5% solids and had a Gardner viscosity of B-C.

Example 4

This example describes the preparation of a representative polyacrylamide which is used as the anionic resin in accordance with this invention. To a reaction vessel equipped with a mechanical stirrer, a thermometer, a reflux condenser and a nitrogen adapter was added 890 parts of water. There then was dissolved in the water 98 parts of acrylamide, two parts of acrylic acid and one and one-half parts of aqueous 10% cupric sulfate. The resulting solution was sparged with nitrogen and heated to 76°C., at which point two parts of ammonium persulfate dissolved in six and one-half parts of water was added. The temperature of the reaction mixture increased 21.5°C. over a period of three minutes following addition of the persulfate. When the temperature returned to 76°C., it was maintained there for two hours, after which the reaction mixture was cooled to room temperature. The resulting solution had a Brookfield viscosity of 54 centipoises at 21°C. and contained less than 0.2% acrylamide based on the polymer content.

Example 5

This example describes the preparation of one of the flooring felt compositions of this invention. A one-quart Waring blender was charged with 500 milliliters of water. To the water was added with stirring an admixture of 3.46 grams of water dispersible spurted polypropylene fibers, 0.43 gram of Rayonier bleached softwood kraft (RBK) fibers, 0.97 gram of water dispersible spurted polyethylene fibers and then an additional 1.30 grams of RBK fibers. The dispersion was transferred to a two-liter container equipped with a high-speed stirrer. Water was added to provide a total volume of one liter of the dispersion, after which the following were added with stirring: 10.15 grams of clay presuspended in approximately 80 milliliters of water; 2.83 grams of a manufacturer-stabilized carboxylated styrene-butadiene copolymer latex, commercially available as Dow XD 30608 (added as a 50% solids latex additionally diluted with about 20 milliliters of water); 0.03 gram of a cationic polyacrylamide retention aid (commercially available as Betz 1260), previously dissolved in 6 milliliters of water; 0.97 gram of water dispersible glass fibers having a length of about 3 milliliters and a diameter of about 6 microns; 8.15 grams of the anionic polyacrylamide resin solution (containing 0.81 gram of polymer) prepared according to Example 4; and 13.0 grams of the base-activated cationic resin solution (containing 0.65 gram of resin) prepared according to Example 2.

Using a Noble and Wood handsheet machine, the fibrous dispersion resulting from the above process was formed into a sheet on a 100-mesh screen. The formed sheet was wet pressed and then dried by six passes over a 240-250°F drum dryer, the side of the sheet in contact with the drum being alternated on each pass. Based on the dry weight of the resulting flooring felt composition, the individual components thereof were present in the following amounts: spurted polyethylene fibers, 4.5%; spurted polypropylene fibers, 16.0%; wood pulp fibers, 8.0%; glass fibers, 4.5%; clay, 47.0%; stabilized butadiene-styrene copolymer, 13.1%; anionic resin, 3.75%; cationic resin, 3.0%; and cationic retention aid, 0.14%. Upon analysis, the felt composition was found to have a basis weight of 302 pounds/3000 square feet, a caliper of 30 mils, a density of 0.710 gram/cubic centimeter, a dry tensile strength of 1353 p.s.i. and a hot (177°C) tensile strength of 522 p.s.i.

Example 6

A handsheet was prepared according to the procedure of Example 5 except that the base-activated cationic resin solution of Example 2 was omitted. Upon analysis the felt composition was found to have a basis weight of 285 pounds/ 3000 square feet, an average caliper of 32.4 mils, a density of 0.563 gram/cubic centimeter, a dry tensile strength of only 315 p.s.i., and a hot (177°C) tensile strength of only 68 p.s.i.

Example 7

A handsheet was prepared according to the procedure of Example 5 except the anionic polyacrylamide resin solution of Example 4 was omitted. Upon analysis, the felt composition was found to have a basis weight of 283 pounds/3000 square feet, an average caliper of 30.9 mils, a density of 0.586 grams/cubic centimeter, a dry tensile strength of only 709 pounds/ square inch, and a hot (177°C) tensile strength of only 152 p.s.i.

Example 8

A handsheet was prepared according to the procedure of Example 5 except that the 0.03 grams of cationic polyacrylamide retention aid was replaced by 0.17 gram of alum (aluminum sulfate octadecahydrate). Upon analysis, the felt composition was found to have a basis weight of 302 pounds/ 3000 square feet, a caliper of 27.2 mils, a density of 0.719 gram/cubic centimeter, a dry tensile strength of 1513 pounds/ square inch, and a hot (177°C) tensile strength of 595 p.s.i.

Examples 9-15

Following the procedure of Example 5, additional flooring felt compositions were prepared and evaluated. The compostions are described in Table 1, and their physical properties are set forth in Table 2.

TABLE I

Example (No.)	Spurted Polypropylene Fiber (%)	Wood Pulp Fiber (%)	Glass Fiber (%)	Clay Filler (%)	Styrene-Butadiene Copolymer (%)	Anionic Resin (%)	Cationic Resin (Ex)	Cationic Resin (%)	Ingred. Dry Wt. (g.)
9	3.6	15.4	13.0	40.0	17.2	6.0	(2)	4.8	22.5
10	7.2	13.8	11.0	40.0	17.2	6.0	(2)	4.8	22.5
11	18.0	12.0	8.0	34.0	17.2	6.0	(2)	4.8	22.5
12	31.0	24.0	6.0	19.0	10.0	5.6	(2)	4.4	22.5
13	31.0	29.0	4.0	-	25.0	6.0	(2)	5.0	12.1
14	50.0	24.0	7.0	5.0	5.0	5.0	(2)	4.0	22.0
15	41.0	31.0	7.0	5.0	5.0	5.0	(2)	4.0	22.0

TABLE II

Example (No.)	Basis Weight (lb./3000 sq. ft.)	Caliper (mils)	Density (g/cm.^{3})	Dry Tensile (lb./sq. in.)	Hot (177°C.) Tensile (lb./sq. in.)
9	310	26.0	0.764	3704	2308
10	306	29.9	0.654	2923	1615
11	316	33.7	0.599	1932	890
12	332	34.3	0.620	1627	539
13	175	23.4	0.480	1906	662
14	321	36.0	0.571	1839	411
15	294	33.8	0.557	1988	630

Example 16

The procedure of Example 5 was duplicated except to replace the cationic resin component of that example with an equal amount of the cationic resin prepared in accordance with Example 3. The flooring felt product had a basis weight of 322 pounds/3000 square feet, a caliper of 27.4 mils, a specific gravity of 0.751 gram/cubic centimeter, a dry tensile strength of 1773 p.s.i. and a hot (177°C) tensile strength of 478 p.s.i.

Example 17

35.5 lb of spurted polypropylene fibers, 8.9 lb of spurted polyethylene fibers 8.9 lb RBK fibers, and 100 lb of clay were dispersed in soft water at 2-2.5% consistency in a beater. The pulp blend was refined to a Canadian Standard Freeness (CSF) of 500 in a double disc refiner. The refined pulp was then pumped to a mixing chest in which 39.4 lb (3.94 lb on the dry basis) of the anionic polyacrylamide of Example 4, was added to the pulp blend with good agitation. With continued agitation, 10.8 lb of glass fibers such as those described in Example 5 was slowly added to the pulp blend, and mixing of the fiber dispersion was continued for five minutes. There were then added to the agitated dispersion 1.2 lb of a defoamer, followed by 25.5 pounds of a 50:50 styrene-butadiene copolymer containing carboxylic functionality, which was added as a 50% solids latex containing 0.5 lb of a latex stabilizer. Finally, there were added 98.4 lb (4.92 pounds on the solids basis) of the base-activated cationic resin of Example 2, and 0.8 lb of a nigrosine dye.

The dilute pulp slurry existing at this point in the process was pumped to a conventional Fourdrinier paper machine and formed into a sheet, the rate of addition of the slurry to the wire, the wet pressing, the drying and the calendering being balanced so as to produce a sheet having a thickness in the range of 20 to 30 mils and a specific gravity in the range of 0.600 to 0.700. The felt composition product was found to have a basis weight of 220 pounds/3000 square feet, a caliper of 21 mils, a specific gravity of 0.670, a dry tensile strength of 1620 p.s.i. and a hot (177°C) tensile strength of 470 p.s.i.

CLAIMS

1. An asbestos-free flooring felt composition which includes polypropylene fibers, wood pulp fibers, glass fibers, filler and polymeric binder characterized in that it is comprised of from 4 to about 55% of water dispersible spurted polyolefin fibers, from about 2 to about 30% of wood pulp fibers, from about 2 to about 20% of water dispersible glass fibers, from 0 to about 50% of an inorganic filler, from about 5 to about 25% of an anionic elastomeric polymer binder, from about 1 to about 10% of an anionic polyacrylamide resin containing from about 2 to about 15% by weight of acrylic acid units, and from about 1 to about 10% of a cationic resin reaction product of epichlorohydrin and a poly(diallylamine) or an aminopolyamide derived from a dicarboxylic acid and a polyalkylene polyamine having two primary amine groups and at least one secondary or tertiary amine group, all percentages of the above components being by weight based on the dry weight of the flooring felt composition.

2. The composition of Claim 1 further characterized in that the water dispersible spurted polyolefin fibers are spurted polypropylene fibers.

3. The composition of Claim 1 further characterized in that the water dispersible spurted polyolefin fibers are an admixture of from about 50 to 100% spurted polypropylene and from about 0 to about 50% spurted polyethylene composition based on weight.

4. The composition of Claim 1 further characterized in that clay as the inorganic filler is present in an amount of from about 10 to about 20%.

5. The composition of Claim 1 further characterized in that the anionic elastomeric polymer binder is a 50:50 styrene-butadiene copolymer containing carboxylic functionality.

6. The composition of Claim 5 further characterized in that the anionic polyacrylamide resin is a copolymer of acrylamide and acrylic acid.

7. The composition of Claim 6 further characterized in that the cationic resin is the reaction product of epichlorohydrin and a poly(diallylamine).

8. The composition of Claim 7 further characterized in that the poly(diallylamine) is poly(N-methyl-diallylamine).

9. The process of preparing an asbestos-free flooring felt composition which includes the step of forming an aqueous dispersion containing polypropylene fibers, wood pulp fibers, glass fibers, filler and polymeric binder characterized in that it comprises the steps of forming an anionic aqueous fiber dispersion comprising from about 4 to about 55% of water dispersible spurted polyolefin fibers, from about 2 to about 30% of wood pulp fibers, from about 2 to about 20% of water dispersible glass fibers, and from about 1 to about 10% of an anionic polyacrylamide resin containing from about 2 to about 15% by weight of acrylic acid units, from 0 to about 50% of an inorganic filler, from about 5 to about 25% of an anionic elastomeric polymer binder, and from about 1 to about 10% of a cationic resin reaction product of epichlorohydrin and a poly(diallylamine) or an aminopolyamide derived from a dicarboxylic acid and a polyalkylene polyamine having two primary amine groups and at least one secondary or tertiary amine group, all percentages of the above components being by weight based on the dry weight of the flooring felt composition, and passing the resulting dispersion through a papermaking machine to form a dimensionally stable fibrous sheet material.

10. The process of Claim 9 further characterized in that the water disperisble spurted polyolefin fibers are spurted polypropylene fibers, the anionic elastomeric polymer binder is a 50:50 styrene-butadiene copolymer containing carboxylic functionality, the anionic polyacrylamide resin is a copolymer

of acrylamide and acrylic acid and the cationic resin is the reaction product of epichlorohydrin and poly(N-methyldiallylamine).

European Patent
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EUROPEAN SEARCH REPORT

0097974
Application number
EP 83 10 6392

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ^{3})
X,Y	GB-A-2 051 170 (ARMSTRONG WORLD INDUSTRIES) * Whole document * ---	1-5,9	D 21 H 5/00 D 21 D 3/00 D 06 N 7/00
D,Y	US-A-4 274 916 (R.E. GROSE) * Whole document * ---	1,4,5	
Y	GB-A-2 028 887 (W.R. GRACE) * Whole document * ---	1,6,9	
Y	US-A-3 049 469 (R.W. DAVISON) * Whole document * ---	6,9	
Y	DE-B-1 209 867 (ZELLSTOFFFABRIK WALDHOF) * Whole document * ---	9	TECHNICAL FIELDS SEARCHED (Int. Cl. ^{3})
Y	US-A-4 156 628 (T.W. RAVE) * Claims 1-4,7-9; column 1, last paragraph; column 2; column 3, lines 1-4, 37-68; column 4, lines 1-15; examples 1,8,11,19; columns 12-16; column 17, lines 1-19 * ---	1-3,6-10	D 06 N D 21 D D 21 H
A	GB-A-2 021 173 (GIORGIA-BONDED FIBRES) -----		

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	09-09-1983	NESTBY K.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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