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NONSKID CREPE RUBBER COMPOSITION

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This invention pertains to a moldable, self-adherent, non-skid crepe coating composition and to a method for preparing the same. For illustrative purposes only the invention will be particularly described as utilized in connection with the heeling and soling of shoes, although it is obviously not limited thereto.

Crepe shoe soles now available generally owe their name to their grainy, crinkled appearance and are usually applied as a thick sole to sport shoes. These crepe soles, however, are soft and often sticky and their wearing qualities are sacrificed for rugged appearance. Furthermore, such soles readily slip on painted, varnished or waxed wood, linoleum or marble floors, or polished brick or concrete floors when wet with water, grease, gasoline, etc.

Compositions used to make crepe soles usually contain rubber, rubber type polymers, or mixtures of these which are vulcanized, cured and sheeted at the plant. The resulting crepe type material, in sheet or board form, is cut to size by the skilled shoe worker or repairman when making or repairing shoes. These crepe soles can also be stamped or pressed in different shoe sole and heel sizes at the plant thus relieving the worker of some waste and the extended cutting step required to shape the crepe shoe sole material.

Before the crepe sole is placed on the shoe bottom, an adhesive or a binder is applied to the shoe bottom or sole to secure the two together and this step may require the application of heat and pressure to insure a good bond. Furthermore, where vulcanization or gluing is not suitable, it is customary to rivet, nail, sew, or by similar mechanical means secure the sole to the shoe bottom.

Thus, it is apparent that prior processes of producing crepe sole material have resulted in crepe materials which readily slip on wet surfaces or pavements and which have required the steps of vulcanizing, sheeting, cutting, or stamping, etc., the crepe sole material before it is placed on the shoe bottom necessitating skilled workmanship, with the further requirement of an additional material, and very possibly an additional step being needed to secure the crepe sole to the shoe bottom when soling or resoling shoes.

It is, therefore, an object of this invention to provide a moldable, non-skid, self-adherent crepe coating composition.

It is another object of this invention to provide a non-skid, crepe shoe sole composition that

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can be shaped without cutting or stamping and can be secured to shoe bottoms without the need of adhesives, heat, pressure, or mechanical attaching means.

It is still another object of this invention to provide a method for making a non-skid, crepe shoe sole composition characterized by the fact that it can be directly applied to shoe bottoms and shaped without cutting or stamping and without the necessity of employing adhesives, heat, pressure, or mechanical securing means.

It is yet another object of this invention to provide shoes with non-skid, self-adherent, crepe rubber heels and soles.

It is a further object of this invention to provide a method for applying a non-skid, crepe shoe sole composition to a shoe bottom without the necessity of cutting or stamping the material or of applying heat, pressure, adhesives, or mechanical securing means.

It has now been found that a moldable, non-skid, self-adherent, crepe shoe sole composition can be obtained by dispersing finely divided rubber and fibers in a neoprene solution. On applying the composition to a shoe bottom before the grated rubber has dissolved in the solution and with evaporation of the solvent, a non-skid, crepe sole results that is self-adherent and tough.

In general, when practicing the invention disclosed herein, it is first necessary to prepare a neoprene solution in which the ground rubber can be dispersed. This solution is conveniently prepared by first milling polymerized chloro-2-butadiene-1,3 of the "AC" type until it breaks down and will readily dissolve in a volatile hydrocarbon solvent. After the chlorobutadiene has dissolved in the solvent, a filler, part of which is fibrous, and a tackifier are added to the solution to form a base composition. The rubber is then grated and mixed with this base composition until a thorough mixture has been obtained. Before the rubber has dissolved in the base composition, the mixture is applied by means of a spatula to a roughened, dry, cleaned shoe bottom. On evaporation of the solvent from the mixture, a grainy and corrugated self-adherent, non-skid crepe shoe sole is obtained.

The polymerized chloro - 2 - butadiene - 1,3 of the "AC" type as manufactured by the Du Pont Company, hereinafter called "neoprene," should conform to ASTM requirements shown in their specification entitled "Tentative method of test for plasticity and recovery of rubber and rubber-like materials by the parallel plate plastometer,"

ASTM–D–926–47–T, with plasticity numbers (readings made in mm.) of 302 to 377 and recovery numbers of 38 to 53 at 80° C. and three minutes. The neoprene has a specific gravity of about 1.23, and it can contain traces of iron compounds, some soap or antioxidants such as are well known to those skilled in the art.

If about 1½ pounds of this "neoprene" are milled on a two-roll, water-cooled 12-inch rubber mill for less than about 10 minutes, however, or not at all, the neoprene swells in the hydrocarbon solvent and will not readily go into solution to form a base composition that can be used as an adhesive carrier for the ground rubber to make crepe soles and as an adhesive to secure the composition to the shoe bottom. On the other hand, if the neoprene is milled for over 30 minutes, it has been found that the resulting base composition has become too soft and sticky, and due to too much cold flow it lacks the quality of retaining its shape when applied to shoe bottoms after addition of the ground rubber and on evaporation of the solvent. A milling period of from about 10 to about 30 minutes has been found to give very satisfactory results. The crepe shoe sole composition made with this milled neoprene after being applied to the shoe bottom and on evaporation of the solvent does not exhibit any tendency to cold flow. Excellent crepe shoe sole compositions have been obtained when using neoprene milled for about twenty minutes. The plasticity numbers of the milled neoprene should be from about 280 to 355 at 80° C. Although it has been shown above that the plasticity numbers for the neoprene before milling are from about 302 to 377, this unmilled neoprene lacks desirable adhesive properties (although it will dissolve somewhat in about 50 per cent of the aromatic solvent), its molecular structure is such that it will swell or gel in the solvent, or will not properly go into solution until it has been milled for from about 10 to about 30 minutes. Milling this neoprene, thus, appears to change its molecular structure and prevents any tendency to form gels resulting in a polymer that will readily dissolve in the solvent to form a base composition having excellent adhesive properties.

Milling also changes the viscosity of the neoprene solution. For example, a solution of an "unmilled" neoprene having a plasticity number of 330 will have a viscosity of from 1 to 1½ minutes while a solution of "milled" neoprene of the same plasticity will have a viscosity of ½ to ¾ minute as shown by the modified Gardner-Holdt test. Since the neoprene has been reduced in molecular size, or changed in molecular structure, it readily penetrates the fibrous leather surface of the shoe bottom permitting great adherence of the neoprene with the shoe bottom. It is, furthermore, not necessary to use milled neoprene of one plasticity number. For example, one-half of the milled neoprene used in the solution can have a plasticity number of about 330 and the other half can have a plasticity number of about 280.

Other rubber milling machines can be used to mill the neoprene besides that disclosed above if the rollers are very closely positioned together and operated at different speeds so that the rubber is crushed and rubbed rather than rolled around. It is, of course, obvious to those skilled in the art that when milling on other machines, the time will vary somewhat from the above depending on the size of the rubber mill, the

quantity of neoprene, the speed of the rolls, the temperature, the spacing between the rolls, etc. For example, it will take from 40 minutes to 90 minutes to obtain sufficient milling on a 60 inch mill to result in a milled neoprene having the above desirable properties as represented by its plasticity numbers of from 280 to 355. From 20 to 30 minutes is required on a 30-inch mill. A "refiner," i. e., a mill where one roll is usually larger than the other and which is generally used in the rubber reclaiming industry, can likewise be employed to mill the neoprene.

After milling, the neoprene is dissolved in a suitable hydrocarbon solvent. This can be conveniently accomplished by adding the solvent to the milled neoprene in a cement churn, or dough or paddle-type mixer. The milled neoprene can be grated, chopped, ground or otherwise finely divided in order to facilitate dissolving it in the solution. This can be obtained by first chilling or freezing the neoprene followed by frictional disintegration, etc. The time required to mix and dissolve the neoprene will vary from about two to about five hours depending on the quantities of neoprene and solvent used. About three hours mixing time at room temperature is usually sufficient to dissolve the milled neoprene.

It has been found that about 100 parts by weight of milled neoprene can be dissolved in from about 250 to about 500 parts by weight of a hydrocarbon solvent. It is preferred to use from 100 parts by weight of the dry, milled neoprene to from 300 to 400 parts by weight of the hydrocarbon solvent. A solvent solution which provides excellent results is prepared by mixing from 40 to 60 parts by volume of toluene with from 60 to 40 parts by volume of gasoline. The primary function of the toluene is to dissolve the milled neoprene while the primary function of the gasoline is to promote evaporation. Benzene or xylene may be substituted for toluene, and in place of the gasoline, heptane, hexane, or pentane can be used. It is, of course, obvious to those skilled in the art that many other hydrocarbon solvents can be utilized in this composition besides those listed above.

While ground rubber can now be added to this composition to provide a crepe shoe sole composition that is applied to shoes without the necessity of vulcanizing, heating, using pressure, adhesives or mechanical securing means and which, of itself, is strongly adherent to the shoe bottom, certain specific resinous materials may be added to the base composition, if found desirable, to improve somewhat the initial tack and bond strength of the resulting crepe sole composition. These materials include wood rosin, wood rosin derivatives (such as hydrogenated methyl abietate), rosin ester derivatives such as Staybelite ester (polyhydric alcohol ester of hydrogenated rosin) or ester gum (glycerol ester of rosin), phenol-formaldehyde resins, resorcinol - formaldehyde resins, coumarone - indene resins, etc. They are generally added to the neoprene base composition or solution prior to the addition of the filler in the proportion of from about 2 to about 20 parts by weight of the resin to about 100 parts by weight of the dry, milled neoprene although it has been found best to use from 5 to 10 parts of resin to 100 parts of neoprene.

When the milled neoprene has completely dissolved in the solvent, and after the resinous materials have been added thereto, a filler, of which

at least about 40 per cent by weight is fibrous, is then added and mixed in with the solution. The fibrous filler is essential in producing a non-skid type of crepe shoe sole. Although the outer surface of the crepe sole, on first being formed, is rough and thus has some non-skid properties, the fibrous filler materially increases the tack of the sole on slippery or wet surfaces. The non-skid property of the sole which is attributable to the fibrous filler is of great importance after the surface of the sole has worn smooth, because the fibrous filler prevents the sole from slipping or skidding. As each layer of material is worn away through use, a new surface layer is presented containing more fibrous material, which was dispersed throughout the sole material during preparation of the composition, so that the non-skid properties are maintained during the life of the sole. A strong milled neoprene-to-fiber bond has formed after evaporation of the solvent giving exceptionally good tensile strength to the sole as well as flexibility. Since the neoprene solution or base composition contains a dispersion of fibers therein and since the outer layers of the grated rubber particles are in a gel or soft state, it is believed the fibers also extend from the matrix of milled neoprene into the outer layers of the rubber particles, giving further strength to the mass on drying. The fibrous filler also aids in promoting evaporation of the solvent. The non-fibrous filler aids in strengthening the sole, filling pores, shaping the sole, and improving the wearability of the resulting sole. Instead of adding the filler to the neoprene solution, it can be added to the neoprene while it is being milled on the rubber mill, and, thus, the time of preparation of the shoe sole composition can be easily reduced.

The total amount of filler, fibrous and non-fibrous, added to the composition is from about 20 to about 125 parts of filler per 100 parts of dry milled neoprene. It is desirable to use from 40 to 80 parts of filler per 100 parts of the neoprene. If more than about 125 parts of filler are used, the resulting crepe-sole composition is too viscous to apply readily by hand and adherence is poor while if less than about 20 parts of filler are present, the composition is slow in drying, has poor wearability, and has lost its non-skid properties.

Examples of suitable fibrous fillers are cotton, or leather fibers, cellulose flock, wood fiber, and synthetic yarn fibers (nylon, viscose rayon or cellulose acetate). These fibers can be used singly or mixed in any proportions. It is preferred to use a mixture of leather fibers and cotton fibers where the cotton fibers have lengths of from 0.060 inch to 0.25 inch. From 10 to 75 parts by weight of the fibrous filler per 100 parts by weight of the dry milled neoprene are used in the composition although it has been found best to use from 20 to 50 parts of fiber per 100 parts of the neoprene. If the fibers are micropulverized, their quantity can be increased up to about 120 parts by weight.

Other organic and mineral fillers, having good abrasive and wear qualities can also be added in amounts up to about 60 per cent by weight of the total filler material in the neoprene solution. These additional fillers can be added singly or mixed together and include rock wool, clay, hydrated calcium silicate, carbon black, metallic particles, wood flour, ground cork, nutshell meal or flour, leather dust, leather blocks or particles, hard rubber dust, Vinylite or styrene and

other synthetic or resinous dusts. These organic and mineral fillers can be added to the composition in the ratio of from 10 to 50 parts of filler per 100 parts of the dry milled neoprene although it is preferred to add from 20 to 30 parts per 100 parts of the neoprene.

At the time the filler is added to the neoprene base composition or solution in the mixer, a small amount of a dispersing agent can also be added thereto to reduce the overall mixing time. From about ½ to about 5 parts by weight of lignin per 100 parts by weight of the dry neoprene is an excellent material for this purpose.

The milled neoprene in the base composition can liberate small amounts of hydrochloric acid during storage or while in service with some deterioration of cellulosic and other materials. It is, therefore, desirable in some cases to add acid acceptors to the composition. For this purpose about four parts of calcined magnesia and about five parts of zinc oxide per 100 parts of the dry neoprene can be used.

Small amounts of organic and inorganic dyes such as are well known to those skilled in the art can likewise be added to the composition to change its color if desired.

The grated, ground, or finely divided rubber can be readily prepared by freezing and subsequent frictional disintegration as well known to those skilled in the art. A rubber mill or grinding machine can likewise be employed for vulcanized rubbers. The rubber should be of sufficient fineness to pass through an 8 mesh size screen. Sizes substantially larger than this do not result in a strong crepe sole. The term "rubber" for the purposes of this invention, is to be construed to mean vulcanized or unvulcanized natural rubber, "S" neoprene, "GRS" (a government synthetic rubber-type S (for styrene) polymer produced from butadiene and styrene, a polybutadiene-styrene copolymer) or "GN" a government produced tough elastic product obtained by heating a plastic benzene soluble polymerizable chloro-2-butadiene-1, 3 polymer neoprene, "AC" neoprene, scrap rubber, e. g., red rubber tubes, natural rubber tubes, or butyl tubes; other synthetic rubbers like nitrile rubbers are also applicable. The "rubber" does not have to be milled as it is used for its wearing qualities and crepe effect and any adhesive properties that it may have are only adventitious. It has, of course, certain non-skid properties since it renders the surface of the sole rough and corrugated. In the case of the grated unvulcanized S, AC, GRS or GN, or natural rubbers, it is necessary to apply the shoe sole composition within a short time after being mixed with the base composition as these particular rubbers tend not only to gel but to dissolve in the solvent of the composition. Thus, on evaporation of the solvent the rough, crepe effect is lost. However, if the shoe sole composition is used shortly after mixing, only a slight softening or gelling of the outer layers of these rubber particles occurs permitting the grated particles to substantially retain their shape and to allow a bond to form between these rubber particles and the continuous phase of the milled neoprene on evaporation of the solvent thereby resulting in a strong crepe, non-skid sole. On the other hand, grated, vulcanized or cured rubbers do not apparently dissolve in the solvent over extended periods of time although they may tend to gel somewhat on their surfaces. Thus, the shoe sole composition containing vulcanized

or cured S, AC, GRS or GN, and natural rubbers, can stand for some time before being applied to a shoe bottom and, on evaporation of the solvent, a crepe, non-skid sole results.

These grated rubber materials can be used singly or mixed together and then added to the base composition (milled neoprene, solvent, filler). Of course, when the grated rubber mixture contains some unvulcanized natural GRS or GN, AC, or S neoprene, it is necessary to apply the composition to the sole before these particular rubbers have dissolved in the solvent. From about 50 to about 200 parts by weight of grated rubber to 100 parts by weight of the dry, milled neoprene are used in the composition. It is preferred, however, to use from about 50 to about 100 parts of grated rubber per 100 parts of the dry, milled neoprene. If less than 50 parts of the grated rubber are used, the composition on drying has no crepe appearance. On the other hand, over 200 parts of the grated rubber results in a crepe sole composition that lacks the requisite strength.

After the rubber has been grated, it is thoroughly mixed with, or dispersed in, the neoprene base composition. The mixing time for small batches, i. e., enough for a pair of shoes, will take from about 2 to about 5 minutes. Larger batches will take obviously longer times. The mixing equipment is not critical and any suitable type can be utilized.

The thoroughly mixed crepe shoe sole composition can now be readily applied by unskilled persons at home to a shoe bottom which has been roughened, cleaned, and dried. It is preferred to first rub some of the crepe composition over the entire surface of the prepared shoe bottom. This thin layer does not have to thoroughly dry. A spatula can be used to apply a thick coating of the composition to the shoe bottom. In place of a spatula it is obvious that a knife or other simple tool can be employed. As soon as a skim coat has formed on the surface of the thick coating, the coating can be shaped to conform to the configuration of the shoe bottom by means of the spatula dipped in water to prevent the adherence of the crepe composition to it. Alternatively, the coating can be shaped before the skim coat forms by means of the spatula. After applying the composition sole, the shoes are placed with their soles up in order to dry. Thick coats or soles take about 48 hours to dry. Thin coats take about 24 hours. The time for drying will, of course, depend on the thickness of the composition. Since the thick coating applied all at once will contract on thorough drying and also tend to be less pleasing to the appearance and have less wearability, it is desirable to apply the shoe sole composition in successive layers which are molded with the spatula and allowed to partially dry or to form a skim coat until the desired thickness has been achieved. For example, a coating of the composition ¼'' thick will on drying be about $\frac{1}{16}$'' to ⅛'' thick. By applying successive coats an unskilled person can obtain a waterproof sole of the desired shape and dimensions which is similar in appearance to machine-made crepe soles.

The base composition containing a volatile solvent and a filler has excellent keeping qualities if suitably packaged so that the solvent will not readily evaporate. The material does not need to be used immediately after compounding as no polymerization or other adverse reactions set in. Packaged in commercially available screw cap containers or other sealable containers having metal surfaces of tin, aluminum or glass which do not cause deterioration of the milled neoprene, the base composition will maintain its semiplastic condition for extended periods of time, and can thus be used as required. Where iron containers are used, the base composition may discolor over a period of time, and the introduction of certain ingredients like zinc oxide or calcium silicate in an amount of about 5 parts per 100 parts of dry milled neoprene will serve to prevent this discoloration. Magnesium oxide in an amount up to four parts per 100 parts of dry neoprene will also serve to improve the stability of the composition. The grated rubber material can likewise be stored for extended periods of time without deterioration if suitably packaged.

The following example will serve to illustrate the invention with more particularity to those skilled in the art:

25 parts by weight (about 1.5 pounds) of neoprene were milled for 20 minutes on a 6 x 12 inch 2-roll rubber mill having a clearance between rollers of approximately 0.030''. The speed of the front roll was 18 R. P. M. while the speed of the rear roll was 26 R. P. M. The gear ratio was thus about 1.4. The rolls were cooled by circulating water through them. After milling, the neoprene was removed from the mill and dissolved in 75 parts by weight of a hydrocarbon solvent composed of 40 per cent toluene and 60 per cent gasoline. To this solution was then added 10 parts by weight of leather fibers and 1.25 parts by weight of coumarone-indene resin, both based on the dry weight of the neoprene, which was then thoroughly stirred. To the resulting base composition there was then added 25 parts of grated rubber from vulcanized natural rubber tubes. The mixture was thoroughly stirred for about 5 minutes until the natural rubber particles were completely dispersed throughout the base composition or solution. The resulting, semi-solid, or plastic mass was immediately applied by means of a spatula to a clean, dry, roughened oak tan shoe bottom to form a sole about ⅜'' thick without the use of any heat, pressure, adhesives, etc. After an air drying period of about 48 hours, the non-skid crepe composition shoe sole was tested and its adherence and wearability were found to be excellent.

This crepe, non-skid shoe sole composition can be applied to heels as well as to shoe bottoms. It adheres readily to previous soles of this crepe composition. It will adhere also to the soles of shoes where the sole is firmly attached to the shoe bottom and is in good condition, therefore eliminating the need of removing the old shoe sole. It may likewise be used to cover the sides or uppers of the shoe to render them waterproof. Likewise, the crepe, non-skid soling composition can be used to coat other composition soles or rubber soles as found in slippers, boots, rubbers, galoshes, etc. By roughening, cleaning, and drying the bottom of the footwear, the crepe soling composition, when added thereto, adheres as satisfactorily as when placed on leather bottoms. In the case of rubber articles, it is believed that the solvent in the crepe soling composition partially dissolves the rubber surface layers of the galoshes or rubber forming a strong bond at the interface between the old rubber surface and the new composition sole.

In summary, it will be apparent that there has been disclosed herein a novel method of preparing a new and novel non-skid, crepe shoe sole

composition. After the rubber particles and fibers have been dispersed throughout a neoprene solution, and on evaporation of the solvent before the rubber particles have dissolved therein, the rubber particles will retain substantiallly their former appearance and be bonded with the fibers in a continuous phase of milled neoprene resulting in a crepe, non-skid sole. It is thought that by thoroughly mixing the finely divided rubber particles with the neoprene base composition, each rubber particle is coated with a solution containing a material having adhesive properties. The rubber particles appear to be firmly bonded with the fibrous filler in the continuous phase or matrix of milled neoprene for no cracking, separation, etc., of the coating occurs on drying. This is probably due to the fact that the outer layers of each rubber particle soften or gel somewhat, permitting a bond or weld to exist between the rubber particles, the filler and the milled neoprene, making the sole also resilient and flexible as well as adherent. It is also believed that the increased density of the mass resulting from the intimate association of fine particles, i. e., fibrous and non-fibrous fillers, milled neoprene and grated rubber, renders the composition tough and long wearing. Such porosity as may exist in the dried coating is in the form of microscopic air pockets which are probably unconnected, for the dried coating is actually not only water resistant but waterproof and will not take up oil. This invention thus provides a novel, easy and inexpensive process to enable skilled and non-skilled persons to make or repair shoe soles, utilizing a moldable, self-adherent, non-skid, crepe composition, eliminating the former steps of sheeting, vulcanizing, curing, cutting or prestamping crepe materials before applying them to shoe bottoms as well as the need for binding materials or operations.

It is to be understood that while the invention has been described with particular emphasis on the heeling and soling of shoes, it is quite obvious that the composition and its method of preparation and application, as described herein, have a great many other uses. This novel composition will be useful wherever a crepe, non-skid, moldable, and self-adherent coating material is required.

What is claimed is:

1. A crepe, non-skid composition consisting essentially of 100 parts by weight of polymerized chloro-2-butadiene-1,3 having plasticity numbers in millimeters of 302 to 377 and recovery numbers of 38 to 53 at 80° C. and three minutes, a specific gravity of about 1.23 and which gels but does not dissolve in hydrocarbon solvents prior to milling which has been milled until it has ASTM plasticity numbers of from 355 to 280 at 80° C. and is soluble in hydrocarbon solvents, from 250–500 parts by weight of a volatile hydrocarbon solvent for said milled polymerized chloro-2-butadiene-1,3, from 2 to 20 parts by weight of a tack-improving organic resin from the group consisting of wood rosin, hydrogenated methyl abietate, polyhydric alcohol ester of hydrogenated rosin, ester gum, phenol-formaldehyde resins, resorcinol-formaldehyde resins, and coumarone-indene resins, from 20 to 125 parts by weight of a filler of which at least 40 per cent is fibrous and organic and the balance nonfibrous, and from 50 to 200 parts by weight of ground rubber of a particle size sufficient to provide a crepe effect on solidifying and not substantially greater than 8 mesh, and which has not dissolved in said solvent.

2. A crepe, non-skid composition consisting essentially of 100 parts by weight of polymerized chloro-2-butadiene-1,3 having plasticity numbers in millimeters of 302 to 377 and recovery numbers of 38 to 53 at 80° C. and three minutes, a specific gravity of about 1.23 and which gels but does not dissolve in hydrocarbon solvents prior to milling which has been milled until it has ASTM plasticity numbers of from 355 to 280 at 80° C. and is soluble in hydrocarbon solvents, from 300–400 parts by weight of a volatile hydrocarbon solvent for said milled polymerized chloro-2-butadiene-1,3, from 5 to 10 parts by weight of a tack-improving organic resin from the group consisting of wood rosin, hydrogenated methyl abietate, polyhydric alcohol ester of hydrogenated rosin, ester gum, phenol-formaldehyde resins, resorcinol-formaldehyde resins, and coumarone-indene resins, from 40 to 80 parts by weight of a filler of which at least 40 per cent is fibrous and organic and the balance nonfibrous, and from 50 to 100 parts by weight of ground rubber of a particle size sufficient to provide a crepe effect on solidifying and not substantially greater than 8 mesh, and which has not dissolved in said solvent.

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