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SIZE FOR TEXTILE FABRICS

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This invention relates to an improved method of sizing textile fabrics prior to the weaving operation and also relates to the sized fiber obtained.

It has been customary to apply a sizing composition to textile yarns and fibers before they are woven to provide a more efficient working of the yarn in the weaving operation. For this purpose, sizing compositions containing starches, gums, gelatin, sulfonated oils, tallows, and mineral oils have been used. Thus, for example, before weaving of cotton yarn, a sizing composition comprising starch, gums, and softeners is usually applied to improve the weaving properties. The starch serves to strengthen the fibers, the gum binds the fibers together and the softener provides lubrication.

In the sizing of such synthetic fabrics as rayon and Nylon yarns, a sizing composition containing gelatin together with sulfonated oils or mineral oils is used. After the weaving operation, the size is generally removed from the fabric by a de-sizing operation.

In the case of starch sizes, considerable difficulty is encountered in removing the size from the fabric. Starch enzymes such as diastase are usually used for this purpose. Such an operation is not readily applicable to these synthetic fabrics. For this reason, starch does not find much use in rayon sizes. Starch sizes also result in fibers which shed appreciably. Gelatin is not very satisfactory as a sizing material because of its non-uniform characteristics and because it requires long soaking treatments for removal from the woven fabric. The de-sizing of gums after weaving usually requires quite drastic treatment, such as caustic boiling under pressure. Use of sulfonated oils is not too practical because of the tendency of such materials to become rancid on ageing. Sulfonated oils also usually provide too much softening effect and too little lubrication. Mineral oil provides certain lubricating and softening characteristics but is subject to the disadvantage that its impregnation of the fibers is uneven and its removal from the fibers after weaving is non-uniform, usually resulting in oil spots on the fabric.

In carrying out the method in accordance with this invention, natural or synthetic fibers in the form of yarns, threads, filaments, etc., are treated with a composition containing a solution of a polymerized terpene resin in mineral oil. These compositions may be applied as an aqueous emulsion, as an organic solvent solution, as

an aqueous dispersion of an organic solvent solution, or as the mineral oil solution of the polymerized terpene resin itself. The portion of polymerized terpene resin included in the mineral oil composition may be varied depending on the type of sizing desired and the particular fiber being treated. Also the amount of the sizing composition employed will depend on the particular effect desired.

To illustrate the sizing of textile fibers in accordance with the invention, the following examples may be cited. In the examples all proportions are by weight unless otherwise indicated.

EXAMPLE 1

Textile size composition containing starch modified by a polymerized terpene resin

The polymerized terpene resin used in this example has the following physical properties:

Melting point	About 50-60° C.
Color	About 30 amber
Odor	Balsam-like
Appearance	Semi-hard resin

Step 1—Emulsification of polymerized terpene resin size.—A quantity of the above polymerized terpene resin was mixed with enough mineral oil to make a 50-50 mixture. This mixture was emulsified with triethanolamine oleate.

Step 2—Polymerized terpene resin-starch size mixture.—15 parts of the above emulsified polymerized terpene resin mixture was added to 81 parts of starch and 1404 parts of water and the resulting mixture heated and agitated until the starch was thoroughly cooked.

Step 3—Sizing of textile fibers.—Cotton yarn was sized with the above polymerized resin-starch sizing mix by the usual procedure. The sized yarn showed increased tensile strength and elongation over conventional sized yarns and the ease of removability of the size from the fabric showed that it could be done with the existing equipment now being used. Tensile strength determinations were made on the above sized cotton yarn and the results were as follows:

Av. break	High	Low	Diff.	Av. Elongation	High	Low	Diff.
Grams 372.1	Grams 368.0	Grams 318.0	Grams 127.0	Per cent 7.30	Per cent 9.70	Per cent 6.50	Per cent 2.20

EXAMPLE 2

Textile size composition containing starch modified by a polymerized terpene resin (Piccolyte S-40)

The polymerized terpene resin used in this example is known in the trade as "Piccolyte S-40" and has the following properties:

Melting point..... 40-42° C.
Color..... WW
Acid number..... Less than 4
Appearance..... Semi-hard

Step 1—Emulsification of polymerized terpene resin size.—A quantity of the above polymerized terpene resin was mixed with enough mineral oil to make a 50-50 mixture. The mixture was emulsified with triethanolamine oleate.

Step 2—Polymerized terpene resin-starch size mixture.—25.2 parts of the polymerized terpene resin-mineral oil emulsion was added to a water starch solution prepared by mixing 144 parts of 60 fluidity corn starch and 1330 parts of water at a temperature of from 70-75° C. with stirring. After the addition of the resin-mineral oil emulsion was made to the water-starch solution, the temperature of the resulting mixture was brought to about 90-95° C. at which temperature the mixture was cooked for one hour.

Step 3—Sizing of textile fibers.—The resin-mineral oil emulsion-starch mixture was applied to 18/1 cotton yarn squeezed and dried. The resulting fibers showed good stiffness, smoothness, and tensile strength. The ease of removability of this composition from the fibers was such that it would be readily removed with conventional equipment. The results of the tensile strength determinations made on the 18/1 cotton yarn were as follows:

Av. break	High	Low	Diff.	Av. elongation	High	Low	Diff.
Grams 539.6	Grams 601.0	Grams 470.0	Grams 221.0	Per cent 5.18	Per cent 6.70	Per cent 4.10	Per cent 2.60

EXAMPLE 3

Textile size composition containing starch modified by a polymerized terpene resin (Piccolyte S-55)

The polymerized terpene resin used in this example is known in the trade as Piccolyte S-55 and has the following properties:

Melting point..... 55-58° C.
Color..... WW
Acid number..... Less than 4
Appearance..... Semi-hard

Step 1—Emulsification of polymerized terpene resin size.—A quantity of the above polymerized terpene resin was mixed with enough mineral oil to make a 50-50 mixture. The mixture was emulsified with triethanolamine oleate.

Step 2—Polymerized terpene resin-starch size mixture.—25.2 parts of polymerized terpene resin-mineral oil emulsion was added to a water-starch solution prepared by mixing 1440 parts of 60-fluidity color starch with 1330 parts of water at a temperature of from 70-75° C. with stirring. After the resin-mineral oil emulsion was added to the water-starch solution, the resulting mixture was brought to a temperature of about 90-95° C. and the mixture cooked for one hour.

Step 3—Sizing of textile fibers.—The resin-mineral oil emulsion-starch mixture was applied to 18/1 cotton yarn squeezed and dried. The resulting fibers showed good stiffness, smoothness and tensile strength. The ease of removability of this material from the fibers was such that it would be readily removed with conventional equipment. Results of tensile strength determinations on the 18/1 treated cotton yarn are as follows:

Av. break	High	Low	Diff.	Av. elongation	High	Low	Diff.
Grams 426.6	Grams 550.0	Grams 316.0	Grams 164.0	Per cent 5.43	Per cent 7.20	Per cent 4.30	Per cent 2.90

EXAMPLE 4

20 Polymerized terpene resin textile size composition

Any one of the above resins described in the foregoing examples may be emulsified according to the following formula:

	Parts
25 Polymerized terpene resin.....	85
Fractol A (mineral oil).....	85
Cobalt naphthenate (6%).....	1
Oleic acid.....	19.8
30 Triethanolamine.....	10.2
Water.....	199

and used as a textile size independently of starch or other similar material.

Step 1—Emulsification operation.—The polymerized terpene resin was melted and the Fractol A stirred in. To 50 g. of this solution, oleic acid was added and stirred in. The triethanolamine was then added and stirred until the reaction was completed. The balance of the resin-oil solution was then added to this mixture and stirred until homogeneous and water was added slowly with high speed agitation. The stability of the emulsion was improved by passing the emulsion through a suitable emulsifier colloid mill.

Step 2—Sizing of textile fibers.—The resin-mineral oil emulsion mixture was applied to 18/1 cotton yarn, squeezed and dried. The resulting fibers showed good stiffness, smoothness and tensile strength. The ease of removability of this material from the fibers was such that it would be readily removed with conventional equipment.

In carrying out the method in accordance with this invention, the natural or synthetic fibers such as yarns, threads, filaments, etc., are treated with the improved polymerized terpene resin containing size. The size may be applied as an aqueous emulsion polymerized terpene resin in mineral oil, as an organic solvent solution of polymerized terpene resin in mineral oil, as an aqueous dispersion of an organic solvent solution of polymerized terpene resin modified starch emulsion.

The polymerized terpenes as, for example, the terpene polymers and copolymers which have been found to provide the desirable emulsions for textile treatment include the resinous materials obtained by polymerization and copolymerization of terpenes, for example, the alicyclic, monocyclic and bicyclic terpenes and their mixtures. Specific terpenes suitable for this invention are alpha pinene, beta pinene, dipentene, limonene, terpinene, terpinolene, allo-ocimene, myrcene, camphene, fenchene, carene, etc. in the presence of catalytic materials such as aluminum chloride, complexes of aluminum chloride with inorganic salts such as $AlCl_3 \cdot NaCl$, zinc chloride,

boron trifluoride, fuller's earth, activated clay, sulfuric acid, phenol diozonium fluoborate, phosphoric acid, tetraphosphoric acid, stannous chloride, fluoboric acid, etc.

Terpene copolymers of acyclic, monocyclic and bicyclic terpenes with other unsaturates such as styrene, coumarone, indene, cyclopentadiene, butadiene, isoprene, and isobutene are also found suitable for the purposes of this invention. Copolymers of terpenes with halogenated unsaturates as, for example, vinyl chloride are found operable.

Hydrogenated products of each of these terpene polymers and copolymers are found desirable for the purposes of this invention.

The polymerized terpenes which are effective in providing the improvements in accordance with this invention are those which are either viscose liquids at ordinary temperatures or soft solids melting up to about 150° C. by the drop method. Preferably, the polymerized terpenes melting at about 40° C. to about 80° C. will be used.

Terpene polymers as, for example, the "Piccolyte" resins herein disclosed may be prepared by procedures well known in the art. The polymerized terpene resin disclosed in Example 1 may be prepared as follows:

To 188 g. of anhydrous aluminum chloride, add 1250 g. of ethylene dichloride. Cool the mixture to 34° F. and add 750 g. of alpha-pinene during 2 3/4 hours with vigorous agitation, maintaining the temperature at about 65 to 70° F., remove the cooling and agitate the homogeneous solution for 1/2 hour at 60 to 70° F. Cool the solution to 40° F. and add 100 g. of ice with stirring during about 10 minutes, during which time the temperature will increase to about 120° F. Add to this mixture about 500 cc. of water with agitation for 15 minutes, thoroughly wash the ethylene dichloride solution and remove the ethylene dichloride by steam distillation. The yield will be about 650 g. of resin. The product is then heated to about 260° F. for about 1/2 hour under a blanket of carbon dioxide as a means of bleaching the color. The physical characteristics of this resin will be as follows:

Melting point	50-60° C.
Color	About 30 amber
Odor	Balsam-like
Appearance	Semi-hard resin

The above described resins may be made into a solution with the mineral oil by heating the resin to its melting point and subsequently adding the desired amount of mineral oil to the melted resin. This solution or an emulsion of it may be used as a substitution in full for gum and softeners used in typical sizing formulations and in full or in part for the starch or gelatin used in sizing formulations. Mineral oil solutions of the polymerized terpenes may be used in the range of about 10 to 85% of polymerized terpene, but preferably in the range of about 40 to 70% of the polymerized terpene.

In the preparation of emulsions of the polymerized terpene resin with mineral oil, the following emulsifying agents may be used as, for example, the alkali metal salts of the higher aliphatic ester sulfates, as sodium lauryl sulfate, sodium stearyl sulfate, sulfonated oils as sulfonated castor oil, sulfonated olive oil, soaps as sodium stearate, sodium oleate, potassium oleate, alkali metal salts and alkali naphthenic sulfuric

acids as sodium butyl naphthenate sulfonate, sodium isopropyl naphthenate sulfonate, triethanolamine, etc.

It is found that yarn sized with sizing compositions containing the above polymerized terpene resin-mineral oil solutions or emulsions of this solution have the following advantages over the conventional prior art starch sized yarns in that the polymerized resin sized yarn has a greater tensile strength, has less solids deposited on the yarn, has a greater smoothness and pliability characteristic, and has a greater effect on decreasing the amount of yarn shredding during weaving operations.

What I claim and desire to protect by Letters Patent is:

1. A textile fiber size comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin.
2. A textile fiber size comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized as having a melting point between about 40° C. and about 80° C.
3. A textile fiber size comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized as having a melting point between about 40° C. and about 60° C.
4. A textile fiber size comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized as having a melting point up to about 60° C.
5. A textile fiber starch size comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized as having a melting point up to about 60° C.
6. A textile fiber starch size comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized as having a melting point between about 40° C. and about 42° C.
7. A textile fiber starch size comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized as having a melting point between about 55° C. and 58° C.
8. The method of sizing textile fibers prior to weaving which comprises treating the fibers with a composition comprising a solution of a polymerized terpene hydrocarbon resin in mineral oil.
9. The method of sizing textile fibers prior to weaving which comprises treating the fibers with a composition comprising a solution of between about 10% to about 85% of polymerized terpene hydrocarbon resin and mineral oil.
10. The method of sizing textile fibers prior to weaving which comprises treating the fibers with a composition comprising a solution of between about 40% to about 70% of polymerized terpene hydrocarbon resin in mineral oil.
11. A textile fiber sized with a composition comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized by having a melting point between about 40° C. and about 60° C.
12. A textile fiber sized with a composition comprising a mineral oil aqueous emulsion of a polymerized terpene hydrocarbon resin, the resin being characterized by having a melting point between about 40° C. and about 80° C.

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