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TEXTILE FINISHING MATERIAL

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This invention relates to an improved finishing composition for textile fabrics, to an improved textile fabric, and to methods of production thereof. More particularly, it relates to a finishing composition having as a binding agent a polymerized terpene resin.

Heretofore, textile fabrics have been treated with finishing compositions having a binding agent (stiffening agent or agglutinate) casein, gelatin, glue or gums, as, for example, tragacanth, tragasol, or mucilage, or, as more commonly used, starch in combination with suitable fillers, softeners, and/or deliquescents, with the object of improving the appearance and handle of the fabric. A common finishing treatment consists of back-filling a fabric with a composition comprising as a filler, china clay or talc; as a softener, oils, fats, waxes, soap, or sulfonated oils; and as a binding agent, starch. This treatment is generally applied to one side of the fabric, thereby closely filling in the open weave of the fabric to increase the weight and improve the handle of the material. This type of mixture is not entirely satisfactory for back-filling inasmuch as the starch does not satisfactorily bind the talc or china clay to the fabric, and on handling, the starch-back-filling composition dusts out, thereby altering the body, weight, and handle as well as the appearance of the fabric. Further, in starch-back-filling compositions, particularly where a colored fabric is being treated, the starch composition, which has a relatively high solids content, has a tendency to cloud or cover the filled side of the fabric resulting in a material having a weaker color on the filled side as compared to the unfilled side. Attempts have been made to overcome this difficulty by adding a dye to the back-filling composition but further difficulties result in exactly matching the color of the unfilled side of the fabric. Other disadvantages found in fabrics finished with a composition having starch as the sole binding agent are: Rapid fabric breakdown caused by aging due to exposure in normal atmosphere resulting in the material becoming limp in handle and raggy in appearance, and the boardiness characteristic of the fabric as contrasted to the smooth, full-bodied and flexible textile desired.

The highly desirable features of this invention are realized and the undesirable properties of the prior art binding agents when used alone are eliminated by treating a textile fabric with a dispersion comprising as an essential constituent a polymerized terpene resin, having a melting point (Hercules drop method) within the range of about 30° C. and about 120° C. and more preferably within the range of about 40° C. to about 60° C. By the term "dispersion" is meant an organic solvent solution, an aqueous emulsion, or an aqueous emulsion formed by dispersing an organic solvent solution of the polymerized terpene resin in water. In addition to the polymerized terpene resin, the dispersion used in providing the various advantageous features of this invention may contain any of the usual textile finishing materials such as starches, gums, talc, softeners, etc.

More particularly, in carrying out the method in accordance with this invention, a textile fabric is treated with a dilute dispersion containing as an essential ingredient a polymerized terpene resin. After treatment of the fabric with the dispersion, the excess dispersion is removed from the textile fabric and the fabric treated in any suitable manner.

The improved fabric composition and method of application in accordance with this invention are illustrated by the following specific examples, all parts and percentages being by weight unless otherwise specified.

Example 1

Textile finishing composition containing polymerized terpene resin as the sole binding agent

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

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

Step 1 (Formulation of emulsified compositions).—An emulsion of the above-prepared resin was made using the following ingredients:

	Parts
Resin (80% in xylene)	100
Water	139.2
Duponol ME (dry)	0.8

The emulsion was made by dissolving the Duponol ME (emulsifier) in the water. The 80% xylene solution of resin was added to the Duponol-water solution with stirring. The mixture was then passed through a suitable homogenizer to produce the desired emulsion.

Step 2 (Fabric impregnation treatments).—A sample of sheeting (2.58 yards) was treated with the above emulsion formed in Step 1, which emulsion had been diluted to 3% solids by dilut-

ing 3 parts of the 33⅓% solids emulsion with 30⅓ parts of water. The sheeting was impregnated in this dilute emulsion, squeezed, and dried over heated cans at about 250° F. The sheeting was characterized by an increased stiffness and fullness as compared to its untreated condition and further characterized by a stiffness factor which increased to 64.4 mg. (Gurley method) as compared to its untreated condition having a stiffening factor of 28 mg.

EXAMPLE 2

Textile finishing composition containing polymerized resin (Piccolyte S–40) as the sole binding agent

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 (Formulation of emulsified composition).—An emulsion of the above-prepared resin was made using the following ingredients:

	Parts
Piccolyte S–40 (80% in xylene)	100
Water	139.2
Duponol ME (dry)	0.8

The emulsion was made by dissolving the Duponol ME in water. The 80% xylene solution was added to the Duponol-water solution with stirring. The mixture was then passed through a suitable homogenizer to produce the desired emulsion.

Step 2 (Fabric impregnation treatment).—A sample of sheeting (2.58 yards) was treated with the above emulsion which had been diluted to 3% solids by diluting 3 parts of the 33⅓% solids emulsion with 30⅓ parts of water. The sheeting was impregnated in this dilute emulsion, squeezed, and dried over heated cans at about 250° F. The sheeting was characterized by an increased stiffness and fullness as compared to its untreated condition and further characterized by a stiffness factor which increased to 44 mg. (Gurley method) as compared to its untreated condition having a stiffness factor of 28 mg.

EXAMPLE 3

Textile finishing composition containing polymerized resin (Piccolyte S–55) as the sole binding agent

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 (Formation of emulsified composition).—An emulsion of the above-prepared resin was made using the following ingredients:

	Parts
Piccolyte S–55 (80% in xylene)	100.0
Water	139.2
Duponol ME (dry)	0.8

The emulsion was made by dissolving the Duponol ME in the water. The 80% xylene solution of resin was added to the Duponol-water solution with stirring. The mixture was then passed through a suitable homogenizer to produce the desired emulsion.

Step 2 (Fabric impregnation treatment).—A sample of sheeting (2.58 yards) was treated with the above emulsion formed in Step 1, which emulsion had been diluted to 3% solids by diluting 3 parts of the 33⅓% solids emulsion with 30⅓ parts of water. The sheeting was impregnated in this dilute emulsion, squeezed, and dried over heated cans at about 250° F. The sheeting was characterized by an increased stiffness and fullness as compared to its untreated condition and further characterized by an increase in stiffness factor (Gurley method) from 28 mg. (untreated fabric) to 64.4 mg.

EXAMPLE 4

Textile finishing composition containing starch modified by a polymerized terpene resin

(Formulation of composition).—In this example the polymerized terpene resin having the properties indicated in Example 1 was used in combination with cornstarch and tapioca flour mixed in the following proportions:

	Parts
Cornstarch	16
Tapioca flour	16
Talc	50
Sulfated tallow	8
Polymerized terpene resin emulsion	37½
Water	320

The above mixture was made by cooking together the starch, talc, flour, softener, and water with constant stirring. The resin emulsion was added after the starch mixture had reached the boiling point. Boiling was continued for 10 minutes after the resin emulsion was added.

Test 1 (Fabric impregnation treatment).—A sample of print cloth (80" by 80") was back-filled with the above resin-starch mixture, squeezed, and dried over heated cans. The cloth back-filled with this starch-resin formulation was fuller and more firm than a sample print cloth treated in the same manner with a straight starch mixture made in accordance with the following formulation:

	Parts
Cornstarch	20
Tapioca flour	20
Talc	50
Sulfated tallow	15
Water	320

The talc dusted out of the print cloth back-filled with the straight starch mixture very badly, especially when the print cloth was ruffled or torn. However, when the print cloth back-filled with the resin mixture was shaken and torn, very little dusting out was noticed.

Test 2 (Dye shade test).—In order to illustrate the improvement in the dyed shade in the back-filled side of a dyed fabric, the resin-starch composition and the starch composition prepared in accordance with Test 1 were each back-filled on separate samples of the blue-dyed Indianhead broad cloth and dried. The appearance of the back-filled side of the blue fabric finished with the starch composition was quite cloudy and about 15% weaker than the unfinished side, while the sample finished with the resin-starch composition showed very little effect of cloudiness and the strength of the color had not been reduced to any appreciable extent on the back-filled side as compared to the unfilled side.

Test 3 (Transparency test).—To illustrate the transparency of the finish and the strength of the film of the resin-starch finishing composition, the resin-starch emulsion was cast in a film on a plate glass and dried. The starch composition made in accordance with Test 1 was cast in a film of the same thickness as the starch-resin film on a glass plate and dried. The film obtained from the starch-resin composition was considerably more transparent and remarkably more resistant to scratching or dusting than was the starch film.

EXAMPLE 5

Textile finishing composition containing starch modified by a polymerized terpene resin

(Formulation of composition).—In this example the polymerized terpene resin having the properties indicated in Example 2 was used in combination with cornstarch and tapioca flour mixed in the following proportions:

	Parts
Cornstarch	16
Tapioca flour	16
Talc	50
Sulfated tallow	8
Piccolyte S–40	37½
Water	320

The above mixture was made by cooking together the starch, talc, flour, softener, and water with constant stirring. The resin emulsion was added after the starch mixture had reached the boiling point. Boiling was continued for 10 minutes after the resin emulsion was added.

Test 1 (Fabric impregnation treatment).—A sample of print cloth (80" by 80") was back-filled with the above resin-starch mixture, squeezed, and dried over heated cans. The cloth back-filled with this starch-resin formulation was fuller and more firm than a sample print cloth treated in the same manner with a straight starch mixture made in accordance with the following formulation:

	Parts
Cornstarch	20
Tapioca flour	20
Talc	50
Sulfated tallow	15
Water	320

The talc dusted out of the print cloth back-filled with the starch composition very badly, especially when the print cloth was ruffled or torn. However, when the print cloth back-filled with the resin-starch mixture was shaken and torn, very little dusting out was noticed.

Test 2 (Dye shade test).—In order to illustrate the improvement in the dyed shade in the back-filled side of a dyed fabric the resin-starch composition and the starch composition prepared in accordance with Test 1 were each back-filled on separate samples of blue-dyed Indianhead broad cloth and dried. The appearance of the back-filled side of the blue fabric finished with the starch composition was quite cloudy and about 15% weaker than the unfinished side, while the sample finished with the resin-starch composition showed very little effect of cloudiness and the strength of the color had not been reduced to any appreciable extent on the back-filled side or compared to the unfilled side.

Test 3 (Transparency test).—To illustrate the transparency of the finish and the strength of the film of the resin-starch finishing composition, the resin-starch emulsion was cast in a film on a plate glass and dried. The starch composition made in accordance with Test 1 was cast in a film of the same thickness as the starch-resin film on a glass plate and dried. The film obtained from the starch-resin composition was considerably more transparent and remarkably more resistant to scratching or dusting out than was the starch film.

EXAMPLE 6

Textile finishing composition containing starch modified by a polymerized terpene resin

(Formulation of composition).—In this example, the polymerized terpene resin having the properties indicated in Example 3 was used in combination with cornstarch and tapioca flour mixed in the following proportions:

	Parts
Cornstarch	16
Tapioca flour	16
Talc	50
Sulfated tallow	8
Piccolyte S–55	37½
Water	320

The above mixture was made by cooking together the starch, talc, flour, softener, and water with constant stirring. The resin emulsion was added after the starch mixture had reached the boiling point. Boiling was continued for 10 minutes after the resin emulsion was added.

Test 1 (Fabric impregnation treatment).—A sample of print cloth (80" by 80") was back-filled with the above resin-starch mixture, squeezed, and dried over heated cans. The cloth back-filled with this resin-starch formulation was fuller and more firm than a sample print cloth treated in the same manner with a straight starch mixture made in accordance with the following formulation:

	Parts
Cornstarch	20
Tapioca flour	20
Talc	50
Sulfated tallow	15
Water	320

The talc dusted out of the print cloth back-filled with the starch composition very badly, especially when the print cloth was ruffled or torn. However, when the print cloth back-filled with the resin mixture was shaken and torn, very little dusting out was noticed.

Test 2 (Dye shade test).—In order to illustrate the improvement in the dyed shade in the back-filled side of a dyed fabric the resin-starch composition and the starch composition prepared in accordance with Test 1 were each back-filled on separate samples of blue-dyed Indianhead broad cloth and dried. The appearance of the back-filled side of the blue fabric finished with the starch composition was quite cloudy and about 15% weaker than the unfinished side, while the sample finished with the resin-starch composition showed very little effect of cloudiness and the strength of the color had not been reduced to any appreciable extent on the back-filled side or compared to the unfilled side.

Test 3 (Transparency test).—To illustrate the transparency of the finish and the strength of the film of the resin-starch finishing composition, the resin-starch emulsion was cast in a film on a plate glass and dried. The starch composition made in accordance with Test 1 was cast in a film of the same thickness as the starch-resin film on a glass plate and dried. The film ob-

tained from the starch-resin composition was considerably more transparent and remarkably more resistant to scratching or dusting than was the starch film.

In greater detail, the highly desirable features of this invention as herein set forth and compared are realized by treating in a suitable manner the textile fabric as, for example, the sheeting and print cloth of the examples with a dilute dispersion containing as an essential ingredient a polymerized resin having a melting point within the range of about 30° C. to about 120° C. and preferably within the range of about 40° C. to about 60° C., removing excess dispersion from the textile fabric, evaporating the volatile ingredients from the treated textile fabric, and drying in any suitable manner, such as on hot rolls, in a blast of heated air, with a hot iron at a temperature within the range of about 160° F. to about 400° F., and preferably within the range of about 200° F. to about 300° F. The exact temperature used for drying will depend on the particular composition containing a polymerized terpene resin and on the particular cloth.

In following the method in accordance with this invention, the excess impregnating composition remaining in the textile fabric after the impregnation treatment may be desirably removed by passing the fabric between padder rolls. The amount of impregnating composition remaining in the fabric, after the excess has been removed, will desirably be such that the amount of dry composition remaining in the fabric will, at the end of the procedure, be insufficient fully to impregnate the individual threads or to form a continuous coating over the surfaces of the textile fabric.

The temperature at which the finishing compositions are applied to textile fabrics in accordance with this invention is not critical. The usual procedures used in the industry are entirely satisfactory and the use of the usual drying equipment consisting of dry cans and tenter frames is satisfactory.

The improved product produced in accordance with this invention comprises a textile fabric which has been treated with a dispersion containing as an essential constituent a polymerized terpene resin. The finished textile fabric is characterized broadly as having an improved, flexible finish. Depending on the type of fabric treated and the auxiliary finishing agents such as starches, gums, talc, softeners, etc., used with the polymerized terpene resin, the finished textile fabric will have various other advantageous properties. Thus, a spun rayon treated with a dispersion of the polymerized terpene resin alone will possess a very satisfactory handle and body. The back-filled fabric produced with use of starch, talc, softeners, etc., as auxiliary agents with the polymerized terpene resin in the finishing composition will not be subject to "dusting out" of the starch, will be more flexible than the usual starch filled fabric and will show improved resistance to breaking of the fabric. If the back-filled fabric is colored, the shade will be cleaner and brighter both on the back and the face than obtainable without the inclusion of the specific polymerized terpene resin in the finishing compositions. If the fabric has been dyed after the finishing treatment, a greater depth and brightness of shade is obtainable than with comparable finished fabrics not containing the polymerized terpene resin. If the textile fabric has been dyed before the finishing treatment, it will also exhibit increased color brightness. A hosiery fabric treated in accordance with the invention will have improved run-proof properties.

In making an emulsion of these polymerized terpene resins, it is preferable to first make a solvent solution of the resins using any of the well-known solvents, such as, for example, xylene, benzene, or naphthalene. It has been found that an 80% solution of the polymerized terpene resin in a solvent as, for example, xylene, is satisfactory for the purpose of this invention.

In the preparation of the emulsion for use in the method of this invention, any of the well-known emulsifying agents may be employed, such as, for example, sodium oleate, triethanolamine stearate, "Duponol ME" (sodium lauryl sulfonate), "Nekal 3A" (sodium alkyl naphthalene), as well as other emulsifiers or combinations of emulsifiers. It is desirable in some instances to stabilize the emulsions by passage through a colloid mill or homogenizer. Stabilizing agents may be included in the emulsions as desired as, for example, sulfated castor oil, methyl cellulose, gelatin, gum arabic, etc.

In preparing the compositions containing starch modified by a polymerized terpene resin, as exemplified in Examples 4, 5, and 6, the starch, talc, and softener mixture is preferably mixed thoroughly with agitation provided by mechanical means or by steam and heated to boiling. When the mix reaches the boiling point, the emulsion of the polymerized terpene resin is added and the mixture boiled until the starch gelatinizes. On gelatination of the starch, the polymerized terpene resin becomes an intimate part of the starch gel structure, forming a smooth, creamy mixture. The cooking time of the starch mixture is preferably reduced with use of the polymerized terpene resin emulsion, about one-half the normal time usually satisfactory.

The polymerized terpene resin may be used in various proportions in the finishing compositions depending on the weight of fabric desired, the temperature of application, and the particular fabric being treated. The amount of polymerized terpene resin used with the other finishing agents, such as starches, talc, gums, softeners, etc., will depend entirely on the particular effect desired.

The polymerized terpenes as, for example, the terpene polymers and copolymers which may be utilized in accomplishing the improvements of the textile fabrics described herein are resins characterized by permanent thermoplasticity and high flexibility. They are essentially water-insoluble and, therefore, remain to a substantial extent in the fabric after cleaning operations.

The terpenes used in the production of a polymerized resin may be any of the known alicyclic, monocyclic and bicyclic unsaturated terpene hydrocarbons and their mixtures, which result in the formation of a resin with desirable melting points. These unsaturated terpene hydrocarbons, being, for example, as follows: Alpha-pinene, beta-pinene, terpinene, limonene, terpinolene, dipentene, sylvestrene, phellandrene, carvene, camphene, sabinene, allo-ocimene, myrcene, fenchene, carene, etc. It is preferred to perform the polymerization operation in such a manner as to provide a polymer having a melting point characteristic within the range hereinbefore set forth.

Terpene polymers and copolymers may be prepared by procedures well known in the art. The

polymerized terpene resin used 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 2¾ hours with vigorous agitation, maintaining the temperature at about 65 to 70° F., remove the cooling and agitate the homogeneous solution for ½ 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 ½ 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 polymerization operation of the terpenes may be carried out by means of catalysts other than aluminum chloride, as, for example, fuller's earth, activated clay, boron trifluoride, sulfuric acid, phosphoric acid, zinc chloride, diazodinitrophenol borate, complexes of aluminum chloride with inorganic salts such as $AlCl_3 \cdot NaCl$, 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.

In accordance with this invention, it is found that such polymerized terpene resins possess very unique properties highly advantageous in textile finishing compositions. Being of the thermoplastic type and having resinous properties, such polymerized resins provide adhesive qualities to other binding agents such as starches, and modifies these binding agents in such a way that the materials no longer retain their undesirable characteristics of the polymerized resin to produce the smooth, full-bodied and flexible textile desired.

The invention is applicable to either pure or back-filled finishes. Thus, in the finishing of spun rayon, a very satisfactory handle and body are developed. In the back-filling of lining cloth with the usual starches, talc and softeners, this improved method and finishing composition provide a finished fabric which is free from "dusting out" of starch and talc and which has improved flexibility and resistance to breaking of the fabric. In the finishing of colored fabrics with starch, this improved finishing composition and method provide a finish which possesses increased color brightness over that obtainable with the usual starch treatment. In addition, the starch particles are more firmly adherent to the fabric. A similar increased color brightness may be obtained by dyeing the fabric after it has been finished according to this invention. In the finishing of hosiery, this improved method binds the cross threads at their point of contact and thereby produces runproof properties.

The many advantages of the treatment of textile fabrics with finishing compositions containing as an essential ingredient a polymerized terpene resin have been described in the above with particular reference to the type of fabric treated and to the effect of the specific polymerized terpene resin on the usual finishing materials such as starches, gums, talc, softeners, etc., when used in conjunction with such materials. In addition to these advantages, it will be readily apparent that the finishes are easy to apply. A greater general uniformity of finishes is obtainable and at a cost which presents a reduction in finishing cost. This advantage results from the fact that greater yardage is obtained, for example, from a starch composition containing the specific polymerized terpene resin than is possible without it, the viscosity being lower. The greater uniformity of finish is a result of a more even spread and better penetration of the solids into the fabric because of better dispersion of the particles of fine dimensions. Due to the resinous character of the polymerized terpene resin used, the treated textile fabric has a greater tensile strength than obtained with straight starch finishes.

Where, in the specification and claims, the term "textile fabric" has been used, the term is intended to cover yarns as well as knitted or woven fabrics.

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

1. A textile fabric finishing composition comprising as a binding agent starch modified with a polymerized terpene hydrocarbon resin.

2. A textile fabric finishing composition comprising as a binding agent cornstarch modified with a polymerized terpene hydrocarbon resin.

3. A textile fabric containing starch modified with a polymerized terpene hydrocarbon resin.

4. A textile fabric containing a finishing agent comprising starch modified with a polymerized pinene hydrocarbon resin, and talc.

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