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PROCESS OF SIZING TEXTILES, THE SIZE, THE PREPARATION OF THE SAME, AND THE SIZED ARTICLE

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The invention relates, in general, to a process of sizing textile materials and, in particular, to a process of sizing synthetic yarns in the production of crepe fabrics therefrom and to correlated improvements in sizing materials employed in such process.

Synthetic fibers in the form of filaments, threads, yarns, etc., of the type employed in the manufacture of woven fabrics, particularly the crepe-type fabrics, are characterized by being somewhat plastic in nature and elongate or stretch readily when subjected to tensile forces of a degree equal to that normally employed in the processing thereof or in the fabrication of goods therefrom. Synthetic yarns, filaments, threads, etc., are not inherently capable of retaining or storing the energy imparted thereto during the twisting step in the manufacture of crepe fabrics with the result that the desired kink or waviness called crepe is not obtained. Moreover, these textile materials show a loss in denier when subjected to the aforementioned elongation or twisting, and also exhibit as a result of such deformation a decreased pliancy and decreased affinity for dyes.

In an attempt to overcome the foregoing difficulties, it is now customary to size or otherwise apply a protective coating around filamentary bodies composed of synthetic materials before processing and/or fabricating goods therefrom. In the manufacture of woven crepe fabrics, for example, the desired number of warp threads are sized and wound upon a beam in the usual manner. The weft or filling threads are the elements in the completed crepe fabric that impart the crepe effect thereto and their preparation usually comprises subjecting a synthetic thread, yarn, filament or the like, to the following steps: 1. soaking, 2. conditioning, 3. spinning and twisting, 4. twist setting, and 5. copping or quilling. Synthetic fibers are manufactured in various sized filaments, a number of which are combined with a slight twist to produce a yarn having given denier. A filament 480 meters long and weighing 1/20 of a gram is called a single denier. The size of filling threads normally employed may vary from 100–300 deniers. In preparing filling yarns the first step comprises soaking skeins of the yarn in a fluid composition comprising a water-soluble material, such as glue, gelatine, etc., and a softener or plasticizer therefor, after which the yarn is hydro-extracted and dried. The second operation comprises conditioning the yarn in a humidified atmosphere so that the yarn will regain its normal moisture content and thus prepare it for the third step of spinning, during which a twist is imparted to the yarn that may run as high as 65 or more turns per inch. It might be stated here that usually about one-half of the filling yarns are given a right-hand twist and the remainder a left-hand twist so as to impart the desired crepe effect in the finished fabric. The fourth step involves setting of the twist by subjecting the twisted yarns to an atmosphere of steam and pressure for a suitable period of time after which the fifth step of copping is carried out, i. e., rewinding the yarn from spools onto quills, cops, shuttle bobbins or the like.

A warp and the filling threads prepared in the above-described manner are woven into a fabric having any desired design, count, width, etc., the left and right-hand twisted filling threads being alternated singly or in groups of any desired number. After the fabric has been woven, it is subjected to a so-called boil-off or scouring operation wherein the sizing material is dissolved or emulsified and removed from the fibers, thus allowing the filling threads to spring back to their most natural condition and thereby producing a kinked or creped effect in the resulting fabric.

While the herein before described procedure is being practiced with varying degrees of success, the fact remains that many difficulties arise therefrom. For example, (*a*) the sizing composition in the bath tends to decompose due to bacterial action, (*b*) the viscosity of the sizing bath is changed due to heating and reheating thus affecting the thickness of the film applied to the yarns, (*c*) the strength of the film-forming constituent is affected by ageing and heating the bath, (*d*) these changes in the properties of the bath render successive skeins of yarn non-uniform, (*e*) the sizing is not readily removed from the fibers at the proper time, (*f*) the resulting crepe or pebble effect is not even or deep enough, etc.; and in view of these and other disadvantages, there is a constant demand for improved sizes and improved processes of producing crepe-effects in woven and other fabrics.

The general object of the invention is to obviate the foregoing and other difficulties in the art.

Another object of the invention is to provide an improved process of sizing yarns, filaments, threads and the like in preparation for their fabrication into textiles.

A specific object of the invention is to pro-

vide an improved size and a commercial process of applying the same so as to produce a crepe fabric having improved crepe or pebble effects.

Another specific object of the invention is to provide a size which is substantially resistant to bacterial decomposition and which may be readily removed from the yarns during the boil-off or desizing operation.

A further object of the invention is to provide a sizing composition which may be heated and reheated for relatively long periods of time without deleteriously affecting the viscosity of the sizing bath or the film strength of the film-forming constituent or constituents contained therein.

Other objects will in part be obvious and will in part appear hereinafter.

According to the invention, there is provided a sizing composition comprising a sulfonated condensation product of an aromatic hydrocarbon, derivative or homologue thereof with a terpene hydrocarbon, alcohol, ketone, ester or other suitable terpene derivative and a plasticizer therefor such as a suitable sulfonated body or mixture of bodies. The invention further comprises applying the novel sizing composition of the invention to synthetic yarns in the manufacture of textile materials, more particularly crepe fabrics.

The invention accordingly comprises the several steps and relation of one or more of such steps with respect to each of the others, and a composition of matter possessing the characteristics, properties and relation of components, all of which will be exemplified in the following detailed disclosure, and the scope of the invention will be indicated in the claims.

The present invention is particularly adapted to the manufacture of the crepe type fabrics composed wholly or partially of non-fibrous cellulosic materials. The warp threads may be composed of any suitable material such, for example, as cellulose acetate, cellulose nitrate, partially or completely de-esterified cellulose esters, cellulose ethers, cellulose oxy or hydroxy ethers, regenerated cellulose, casein, synthetic resins, etc. The filling threads, which are the elements that impart the crepe or pebble effect to the finished fabric, are preferably composed of a hydrophilic colloidal material such as regenerated cellulose which may be obtained in a known manner from viscose, cuprammonium solutions of cellulose, or by the partial or complete de-esterification of cellulose esters or by other suitable processes. Other water or alkali swelling cellulose derivatives may also be used as filling threads such as alkali soluble cellulose ethers and the like.

The film-forming constituent in the sizing composition may comprise any suitable water soluble product obtained by the sulfonation of a condensation product of a terpene or derivative thereof with any suitable aromatic hydrocarbon or their homologues. More specifically, the film-forming ingredient may comprise a sulfonated product derived from the condensation of any suitable mono or polynuclear hydrocarbon or homologue such, for example, as benzene, toluene, xylene, naphthalene, phenols, cresols, amines, naphthols, etc., as well as their corresponding sulphonated derivatives, with any suitable open chain or cyclic terpenes or their alcohols, ketones, esters, etc. such, for example, as terpin, terpin hydrate, pinene, terpinene, terpinolene, dipentene, terpineol, borneol, fenchyl alcohol, etc., or with crude materials rich in terpenes or terpene alcohols such as turpentine and pine oil respectively. The expression "terpenic compound" is used generically in the present specification and claims to describe the terpenic bodies of the aforementioned type. Sulfonation of the condensation product may be dispensed with in some instances where the hydrocarbon employed in the condensation is pre-sulfonated. The aforementioned condensation products may be modified by further condensation with a compound which is capable of reacting with two molecules of the substituted sulphonic acid by forming a carbon or sulfur bridge between two aromatic nuclei, such for example as aliphatic aldehydes as formaldehyde or acetaldehyde, etc., aromatic aldehydes as benzaldehyde, etc., ketones as acetone, etc. or sulfurhalides as sulfur-monochloride, etc.

The condensation product may be sulfonated either during the condensation process or subsequently thereto by any suitable sulfonating agent, such as sulfuric acid, fuming sulfuric acid, acetyl sulfuric acid, chlorsulfonic acid, oleum, etc., or the hydrocarbon may be sulphonated prior to the condensation process.

For a fuller understanding of the nature of the water-soluble film-forming constituent employed in the sizing composition of the invention, the following examples are given to illustrate suitable methods of producing the same and they are not to be construed in a limiting sense.

Example I

To a mixture of 108 parts of cresylic acid, 200 parts of ethylene chloride and 154 parts of pine oil are added slowly and with rapid stirring at 15° C. to 20° C. 200 parts of 20% oleum. This mixture is allowed to stand over night at room temperature and then poured into 1500 cc's. water containing 200 grams of sodium chloride. In about two hours separation takes place and the sulfonation mass (bottom layer) is drawn off and neutralized with NaOH and solvent removed.

Example II

To a mixture of 108 parts of M-cresol and 154 parts of turpentine are added slowly and with rapid stirring at 15° C. to 20° C. 210 parts of 100% sulphuric acid. The mixture is well stirred for two hours and is then allowed to stand at room temperature for 24 hours. The reaction mass which is now clearly soluble in water is then washed substantially free from acid with salt solution and is then neutralized with caustic soda solution and the solvent distilled off.

Example III

To a solution of 128 parts of naphthalene in 154 parts of pine oil and 300 parts of ethylene chloride are added with rapid stirring between 20° C. and 30° C. 260 parts of chlorsulphonic acid. The reaction mass is stirred until the evolution of hydrogenchloride has ceased and is then allowed to stand over night at room temperature. The reaction mass is then washed with salt solution, neutralized with soda and the solvent is distilled off.

Example IV

To a mixture of 108 parts of commercial cresylic acid, 136 parts of dipentene and 250 parts of carbontetrachloride 200 parts of fuming sulphuric acid are added between 15° C. and 20° C. and the mass is stirred for 6 hours. To this mixture is then added 29 parts of acetone with rapid stirring, at 20° C.–30° C. The reaction is com-

pleted when the mass is clearly soluble in water which requires about 10 hours. The product of reaction is then washed and neutralized and the solvent is distilled off.

Example V

To a mixture of 94 parts of phenol, 154 parts of pine oil and 200 parts of ethylene chloride are added with rapid stirring 250 parts of fuming sulphuric acid, keeping the temperature during the reaction between 20° C. and 30° C. The reaction is completed when the mass is clearly soluble in water which required about 4 hours. At the end of this period 53 parts of benzaldehyde is added, keeping the temperature below 30° C. The mass is agitated for 2 hours and then allowed to stand for 24 hours without agitation. During this time condensation of the benzaldehyde with the substituted sulphonic acids becomes complete. The mass is then washed and neutralized and the solvent is distilled off in the usual manner.

Example VI

A mixture of 108 parts of U. S. P. cresol, 154 parts of turpentine, 250 parts of ethylene chloride and 260 parts of chlor-sulphonic acid are subjected to reaction at a temperature of 15° C.–20° C. for 4 hours or until the product is clearly soluble in water. 68 parts of sulphur monochloride is then added slowly and with rapid stirring, while the temperature is maintained between 20° C.–30° C. The mass is then stirred for an additional period of 3 hours and is then washed and finished as cited in Example I.

Example VII

To a solution of 128 parts of naphthalene in 154 parts of pine oil and 200 parts of ethylene chloride are added with rapid stirring at 20° C.–30° C. 300 parts of chlorsulphonic acid. The mixture is then allowed to stand at room temperature for 24 hours. At the end of this period the reaction mass is diluted with 100 parts of water and 38 parts of a 40% formaldehyde solution are then added slowly and with stirring. During this operation the temperature is kept between 20° C. and 40° C. The mass is then stirred for 1 hour or until the formaldehyde odor has completely disappeared. The product of reaction is then washed in salt solution and neutralized and the solvent is distilled off.

Example VIII

92 parts of toluene are converted into toluene sulphonic acid by acting on it with 100 parts of fuming sulphuric acid at 100° C. Thereupon the reaction mixture is cooled to 20° C. and a solution of 154 parts of alpha-terpineol in 200 parts of ethylene chloride is added. The mixture is then agitated and while maintaining the temperature between 15° C. and 20° C., 100 parts of fuming sulphuric acid are added. The mixture is then stirred for 4 hours at 20° C.–30° C. At the end of this period the product of reaction is poured into 1500 parts of a 20% salt solution and well stirred. The mass separates into two layers, the lower of which containing the sulphonated condensation product is withdrawn and neutralized with caustic soda solution and the solvent distilled off.

Example IX

108 parts of M-cresol are converted into the corresponding sulphonic acid by acting on it with 200 parts of 100% sulphuric acid at 120° C. The reaction mixture is then cooled to 20° C. and a solution of 154 parts of pine oil in 200 parts of carbon tetrachloride is added. The mixture is stirred well and then 100 parts of fuming sulphuric acid are added, keeping the temperature between 15° C. and 20° C. At the end of six hours the product of reaction is poured into 2000 parts of a 20% salt solution and well agitated. The mass separates into two layers, the lower of which containing the sulphonated condensation product is withdrawn and neutralized with a caustic potash solution and the solvent distilled off.

Example X

To a mixture of 208 parts of naphthalene sulphonic acid and 200 parts of ethylene chloride are added with rapid stirring at 20° C.–30° C. 154 parts of pine oil, followed by 100 parts of chlorsulphonic acid. The mixture is then allowed to stand for 24 hours. At the end of this period the reaction mass is diluted with 100 parts of water and 38 parts of 40% formaldehyde solution are then added slowly and with stirring, keeping the temperature between 20° C. and 40° C. The mass is then stirred for 1 hour or until the formaldehyde odor has completely disappeared. The product of reaction is then washed in salt solution and neutralized and the solvent is distilled off.

In preparing the sizing composition of the invention a desired amount of a condensation product, of the type hereinbefore described, is admixed with or otherwise has incorporated therein, a suitable plasticizer therefor, such for example as sulfonated castor oil soap, sulfonated cocoanut oil, or any other suitable sulfated fatty oil, wax or soap thereof, as well as mahogany soaps, sulfated alcohols, pine oil or any mixture of these or other substances, which function to effectively soften or plasticize the sizing deposited on the yarn so that the original pliability or flexibility of the yarn will not be materially affected.

For a fuller understanding of the nature and the objects of the invention, reference is made to the following specific examples of the sizing composition of the invention and their application with respect to the treatment of textiles. It is to be understood that these examples are given merely to illustrate the invention and they should not be construed in a limiting sense.

Example A

	Pounds
Sulfonated cresol-pine oil condensation product obtained as in Example I (dry weight)	9
Sulfonated castor oil soap do	3
Water	500

A regenerated cellulose filling yarn (150 denier 40 filament Bright) is soaked in the above composition and dried. The sized yarn is then conditioned, twisted and finally woven with a suitable warp into a 96x50, 4x4 crepe fabric which is subsequently scoured to remove the size. The resulting crepe fabric is characterized by having a fine even, deep pebble presenting an enhanced appearance with respect to prior crepe fabrics.

Example B

	Pounds
Sulfonated cresol-turpentine condensation product obtained as in Example II (dry weight)	10
Sulfonated sperm oil do	4
Water	500

A 96x50 fabric having a 2x2 crepe produced from a filling yarn (150 denier 40 filament Bright) sized with the above composition, in accordance with the process under Example A, exhibits an even and deep pebble effect. A deeper, finer and more regular pebble effect may be obtained by employing ½ the amount of plasticizer (sulfonated sperm oil) given above, when the filling yarn is woven into a 111—52 2x2 crepe fabric.

Example C

	Pounds
Sulfonated condensation product obtained as in any of Examples I to X--(dry weight)--	3
Sulfonated fatty oil or wax----------do----	2
Water ----------	500

Creped fabrics produced from filling yarn sized with any of the above compositions present an even pebble and full hand.

Example D

As a warp sizing the following composition may be used:

	Pounds
Sulfonated condensation product---------- ----------(dry weight)--	35
Sulfonated cocoanut oil----------do----	8
Sulfonated castor oil and pine oil------	500

This particular composition has proven valuable in sizing cellulose acetate warp yarns.

It is within the purview of the invention to apply the size to the yarns in any suitable manner as by soaking a skein in a bath or by continuously running the yarn through a bath or over a coated roller or by spraying or in any other suitable manner. The same bath may be used continuously or intermittently from day to day and the concentration of the solids may be kept within the operable percentage range by periodically adding given quantities of the condensation product and plasticizer.

Many advantages over the heretofore known practices result from employing the hereindescribed invention, a few of which are enumerated as follows:

1. More even and deeper pebble effects are obtained in the finished crepe fabrics. For example, a 150 denier yarn gives pebble effects equivalent to that heretofore obtained with a 200 denier yarn.

2. The sizing composition may be subjected to repeated heatings and may be used for a prolonged period of time without materially affecting its film strength or viscosity.

3. The sizing composition is not subject to bacterial decomposition as is commonly the result when using glue, gelatine and like materials.

4. The sizing is readily removed from the yarns during the scouring operation.

5. The film-forming constituent displays certain wetting properties which aids in obtaining a more even sizing.

6. The film-forming constituent is produced synthetically from relatively pure raw materials thereby assuring uniformity in the sizing from batch to batch.

The expression "yarn" is used herein to include yarns, threads, filaments, fibers and like textile materials.

Since certain changes in carrying out the above process and certain modifications in the composition which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all the generic and specific features of the invention herein described and all statements of the scope of the invention, which, as a matter of language, might be said to fall therebetween; and that they are intended to be inclusive in scope and not exclusive, in that if desired other materials may be added to our novel composition of matter herein claimed without departing from the spirit of the invention. Particularly it is to be understood that in said claims, ingredients or components recited in the singular are intended to include compatible mixtures of said ingredients wherever the sense permits.

Having described our invention, what we claim as new and desire to secure by Letters Patent is:

1. A process of sizing textiles, which comprises applying thereto a sizing composition containing as the film forming constituent a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives.

2. A process of sizing textiles, which comprises applying thereto a sizing composition comprising a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives as the film forming constituent and a plasticizer therefor.

3. A process of sizing textile yarns, which comprises applying thereto a sizing composition comprising an aqueous solution of a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives, the condensation product serving as the film forming constituent.

4. A process of sizing textile yarns, which comprises applying thereto a sizing composition comprising an aqueous solution of a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives and a plasticizer therefor, the condensation product serving as the film forming constituent.

5. In a process of preparing warp and filling yarns for weaving into a crepe fabric, the step which comprises sizing such yarns with a composition containing as the film forming constituent a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives.

6. In a process of preparing filling yarns for weaving into a crepe fabric, the step which comprises applying to a yarn composed of a hydrophilic colloidal material, a composition containing as the film forming constituent a water-soluble salt of a sulfonated condensation product of

a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives.

7. A process of preparing filling yarns for weaving into crepe fabrics, comprising sizing a yarn composed of a hydrophilic cellulosic material with a composition comprising a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives as the film forming constituent and a plasticizer therefor and twisting the yarn.

8. A process of preparing filling yarns for weaving into crepe fabrics, comprising sizing a yarn composed of a hydrophilic cellulosic material wtih a composition comprising a water-soluble salt of a sulfonated condensation product of a cresol with turpentine as the film forming constituent and a plasticizer therefor and twisting the yarn.

9. A process of preparing filling yarns for weaving into crepe fabrics, comprising sizing a yarn composed of a hydrophilic cellulosic material with a composition comprising a water-soluble salt of a sulfonated condensation product of a cresylic acid with pine oil as the film forming constituent and a plasticizer therefor and twisting the yarn.

10. A process of preparing filling yarns for weaving into crepe fabrics, comprising sizing a yarn composed of a hydrophilic cellulosic material with a composition comprising a water-soluble salt of a sulfonated condensation product of a cresol with pine oil and an aldehyde as the film forming constituent and a plasticizer therefor and twisting the yarn.

11. A sizing bath for warp and filling yarns to be woven into crepe fabrics, comprising an aqueous solution of a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives as the film forming constituent and a plasticizer for the condensation product.

12. A sizing bath for warp and filling yarns to be woven into crepe fabrics, comprising an aqueous solution of a water-soluble salt of a sulfonated condensation product of cresol with turpentine as the film forming constituent and a plasticizer for the condensation product.

13. A sizing bath for warp and filling yarns to be woven into crepe fabrics, comprising an aqueous solution of a water-soluble salt of a sulfonated condensation product of cresylic acid with pine oil as the film forming constituent and a plasticizer for the condensation product.

14. A sizing bath for warp and filling yarns to be woven into crepe fabrics, comprising an aqueous solution of a water-soluble salt of a sulfonated condensation product of a cresol with pine oil and an aldehyde as the film forming constituent and a plasticizer for the condensation product.

15. A sizing bath for warp and filling yarns to be woven into crepe fabrics, comprising an aqueous solution of a water-soluble salt of a sulfonated condensation product of a cresol with pine oil and formaldehyde as the film forming constituent and a plasticizer for the condensation product.

16. A textile yarn bearing a size comprising as the film forming constituent a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives.

17. A filling yarn adapted to be woven into a crepe fabric comprising a twisted yarn bearing a size comprising as the film forming constituent a water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives.

18. A filling yarn adapted to be woven into a crepe fabric comprising a twisted hydrophilic cellulosic yarn bearing a size comprising as the film forming constituent a plasticized water-soluble salt of a sulfonated condensation product of a terpenic compound with an aromatic compound selected from the group consisting of the mono and polynuclear hydrocarbons and their hydroxy, carboxylic, alkyl, amino and sulfonic acid derivatives.

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