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SIZING COMPOSITIONS

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This invention relates to sizing compositions and relates more particularly to compositions particularly adapted to be employed for the sizing of spun yarns comprising staple fibers having a basis of cellulose acetate or other organic derivative of cellulose.

An object of this invention is the provision of an improved composition for the sizing of spun yarns of staple fibers having a basis of cellulose acetate or other organic derivative of cellulose.

Another object of this invention is the provision of a composition for the sizing of fine count spun yarns of staple fibers having a basis of cellulose acetate or other organic derivative of cellulose to enable said fine count yarns to be woven as a warp in the production of fabric materials.

A further object of this invention is to provide a novel composition for the sizing of fine count spun yarns having a basis of cellulose acetate or other organic derivative of cellulose staple fibers which not only acts as a sizing composition but which also simultaneously lubricates and conditions the spun yarns for weaving operations.

Other objects of this invention will appear from the following detailed description.

As is well known in the art, warps of spun yarns are normally sized prior to being employed in weaving operations. The object of applying sizing compositions to the yarns is to form a film or coating thereon which strengthens the yarns and renders them more resistant to abrasion during weaving operations which tends to weaken or sever the yarns and to produce end breaks. In spite of the application of sizes of varying composition to spun yarns comprising staple fibers of cellulose acetate or other organic derivative of cellulose, an excessive number of end breaks is encountered in the weaving of fine count warp yarns and, particularly, yarns of 20/1^s or finer. Since the end breaks have to be knotted together to continue weaving, the quality of the fabric is reduced and, in the past, has been so serious as to render the weaving of spun yarns of cellulose acetate or other organic derivative of cellulose staple fiber of a count of 20/1^s or finer commercially impractical.

We have now found that if warps of spun yarns of 20/1^s count and finer are sized with an improved composition having both a sizing and lubricating action on said spun yarns, the warps obtained may be woven into commercially acceptable fabric in an efficient and economical manner with a substantially reduced number of end breaks.

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The novel sizing composition of our invention comprises an aqueous solution of polyvinyl alcohol containing a humidifying agent such as glycerine, sorbitol, pentaerythritol, polyethylene glycol and urea, and having a lubricant composition and a conditioning composition dispersed therein. The film-forming polyvinyl alcohol in our novel sizing composition acts to bind the staple fibers comprising the spun yarn while the lubricant and conditioning compositions incorporated in the size reduce frictional and static effects which tend to impair fabric quality.

The lubricant composition which we employ comprises a plurality of components including mineral oil, an oxidized vegetable oil, a dialkyl phenol, an alkylolamine and a higher fatty acid. The lubricant composition may also contain an acyl derivative of an ester of a hydroxylated higher fatty acid in which the acyl radical is derived from a lower fatty acid. The conditioning composition comprises the desalted, dehydrated product, diluted with mineral oil, obtained on reacting oleum with a mixture of mineral oil, a vegetable oil and a higher fatty acid to which has been added a penetrating agent such as a dialkyl phenol and which has been neutralized with an alkali such as sodium hydroxide prior to salt removal and dehydration.

The lubricant composition is preferably prepared, for examule, by mixing mineral oil with a suitable quantity of oxidized peanut oil, or oxidized sperm oil, oxidized olive oil or oxidized teaseed oil, adding a dialkyl phenol, preferably branch chained, such as, di-secondary or di-tertiary-butyl phenol or di-secondary or di-tertiary-amyl phenol, an alkylolamine such as triethanolamine, and a higher fatty acid such as oleic acid. Butyl acetyl ricinoleate or other acyl derivative of an ester of a hydroxylated higher fatty acid may also be added. Usually, the mineral oil comprises 50 to about 75% by weight of the mixture, the oxidized vegetable oil is about 20% by weight, the dialkyl phenol about 10% by weight and the acylated higher fatty acid ester about 10% by weight. The long chain fatty acid and alkylolamine constitute the remainder and are usually added in stoichiometric proportions so as to form an alkylolamine soap of the fatty acid. A very small amount of a blending agent such as oleyl alcohol may also be employed. The preparation of the lubricant compositions described and which constitutes one of the components of our novel yarn size is more particularly described in U. S. Patent No. 2,385,423, all of the compositions disclosed therein being suitable for use as the lubri-

cant component in the preparation of our novel sizing composition. The compositions specified in Examples I, III, IV and V are particularly suitable.

The conditioning composition employed in the sizing composition of my invention is obtained by reacting a mixture of mineral oil, a vegetable oil such as olive oil, rice oil or peanut oil, and a long chain aliphatic acid such as oleic acid, ricinoleic acid or palmitic with fuming sulfuric acid such as 20% oleum, at a temperature no greater than about 20° C. Under these conditions at least part of the aliphatic acid and the vegetable oil employed are sulfated and/or sulfonated. At the completion of the sulfation and/or sulfonation reaction, an alkylated phenol, such as dibutyl or diamyl phenol is added to the reaction mixture to stabilize the same and to act as a penetrant and an anti-oxidant. The addition of the alkyl phenol is followed by the addition of a sufficient amount of an alkali metal hydroxide to neutralize all of the sulfuric acid remaining and the major portion of the free aliphatic acid. The batch is then stirred until reaction ceases and a clear oil is formed.

The sulfated and/or sulfonated reaction product obtained in the manner described above is subjected to a de-salting and dehydration treatment before it is diluted with mineral oil and employed as a component of my novel sizing composition. The de-salting and dehydration is effected by diluting the neutralized reaction product with an organic solvent such as anhydrous isopropyl alcohol which is a non-solvent for the inorganic salts present and which forms an azeotrope with water. The alcohol addition causes the salts to be precipitated from solution. The precipitated salts are then filtered out and the isopropyl alcohol distilled off, the water which is present in the mixture coming off as an azeotrope with the isopropyl alcohol. Preferably, the temperature of the sulfonated mixture being distilled should not exceed about 110° C. during the distillation operation. If any water remains, an additional quantity of anhydrous isopropyl alcohol is then added and the diluted mixture again distilled to remove the remaining water as well as the alcohol. Prior to distillation, a filter aid such as diatomaceous earth or bentonite, for example, may be added and the solution filtered to improve the clarity and remove any inorganic salts still present. The still residue remaining after distillation comprises the composition employed as the salt-free, dehydrated component. The latter is diluted with added mineral oil prior to use in forming our novel size. To obtain the desired degree of dilution about 40 parts by weight of the salt-free, dehydrated product are added to about 60 parts by weight of mineral oil.

To form the novel sizing composition of our invention from about 25 to 85 parts by weight of polyvinyl alcohol are mixed with about 10 to 40 parts by weight of glycerine, 6.5 to 19.5 parts by weight of the multicomponent lubricant composition and 3.5 to 10.5 parts by weight of the mineral oil-diluted desalted, dehydrated sulfated and/or sulfonated reaction product and the whole then diluted with 400 to 1200, and, preferably about 800 parts by weight of water. After thorough mixing, the size composition is in suitable dilution for application to a warp of spun yarns having a basis of cellulose acetate or other organic derivative of cellulose staple fibers. The size may be applied to the warp in the usual manner employing a slasher or the like.

The components of our novel size composition are not entirely compatible and the composition is preferably agitated during application of the size to a warp. Our novel composition contains a balance of oil components which are not compatible with the polyvinyl alcohol film which is formed and, on drying, the oil components in the size composition are, in part, forced to the surface. Thus, the polyvinyl alcohol film acts to bind the yarn fibers while the oil components impart the desired lubricity.

In order further to illustrate our invention, but without being limited thereto, the following examples are given:

Example I

A mixture suitable for use as the lubricant composition component in forming the novel size of my invention is formed by thoroughly mixing the following components in the proportions given:

	Parts by weight
Paraffin base white mineral oil (50 seconds viscosity)	55.05
Oxidized peanut oil	20.2
Oleic acid	10.35
Di-tertiary-amyl phenol	4.55
Triethanolamine	4.80
Butyl acetyl ricinoleate	5.05

Example II

The de-salted, dehydrated component of our novel sizing composition is prepared as follows: A mixture of 117 parts by weight of acid-refined white mineral oil of about 50 Saybolt viscosity at 100° F., 89 parts by weight of oleic acid, and 43 parts by weight of raw peanut oil are cooled to 8 to 10° C. in a suitable vessel provided with a cooling jacket and then 27 parts by weight of 20% oleum are added over a period of one-half hour. The temperature rises due to the exothermic nature of the reaction but the maximum temperature is held to below 25° C. by cooling. The reaction mixture is then stirred for an additional 45 minutes with the temperature held at 20° C. To the reaction mixture, there are now added 21 parts by weight of diamyl phenol, followed by the added to 75 parts by weight of a 25% by weight aqueous solution of sodium hydroxide. The resulting neutralized mixture is cooled to room temperature with stirring.

The sulfonated reaction mixture obtained is then desalted by the addition thereto of anhydrous isopropyl alcohol to precipitate the inorganic salts present and the latter are then removed by filtration. The filtered solution is distilled under vacuum and the isopropyl alcohol and water are taken off as an azeotropic mixture with the still temperature held at a maximum of 110° C. The still residue is diluted with an equal volume of anhydrous isopropyl alcohol, about 0.1 to 0.4% by weight of a diatomaceous earth filter aid are added and the mixture filtered again. The isopropyl alcohol present and any remaining water are removed by repeating the distillation under vacuum at a maximum still temperature of 110° C. The still residue comprises the desalted and dehydrated reaction product which, after dilution with mineral oil in a ratio of 40 parts by weight of the still residue to 60 parts by weight of mineral oil, is incorporated in said diluted form as a component of our novel sizing composition.

Example III

In order to prepare our novel sizing composition, the following are mixed together:

	Parts by weight
Polyvinyl alcohol	54
Glycerine	10
Lubricant composition of Example I	13
Mineral-oil diluted desalted and dehydrated reaction product of Example II	7
Water	800

After mixing thoroughly to disperse the components in the water the size is ready for use in a slasher. During use, the size is preferably continuously agitated as it is applied to the warp.

A 43 inch warp of 2752 ends of 20/1^s spun yarn of a blend consisting of 45% 2 denier per filament, 30% by weight of 3 denier per filament and 25% by weight of 5.5 denier per filament cellulose acetate staple fiber, all of an average fiber length of 2 inches, is sized with the above size composition. The sized warp is then woven into a tabby weave fabric with a filling of 54 picks per inch of the above blended 20/1^s spun yarn. In a total length of 183 yards only 34 end breaks were found to have occurred for an average of only 0.19 end breaks per running yard. Normally, without the use of our novel size, end breaks of over 1.0 per running yard are encountered when trying to weave the 20/1^s spun yarn as a warp.

While the application of the novel size of our invention has been described more particularly in connection with the sizing of fine count spun cellulose acetate yarns, equally valuable results may be obtained where the spun yarns have a basis of other organic derivative of cellulose materials. Examples of other organic derivative of cellulose materials are cellulose esters such as cellulose propionate, cellulose butyrate, cellulose acetate-propionate and cellulose acetate-butyrate as well as cellulose esters such as ethyl cellulose and benzyl cellulose. Furthermore, while the use of our novel size for the sizing of warps of fine count spun yarns has been stressed, it is to be understood that our novel size may also be employed for the sizing of coarser counts of organic derivative of cellulose staple fiber spun yarns, spun yarns of fibers of other textile materials or even continuous filament yarns.

It is to be understood that the foregoing detailed description is given merely by way of illustration and that many variations may be made therein without departing from the spirit of our invention.

Having described our invention, what we desire to secure by Letters Patent is:

1. A composition for the sizing of textile yarns, comprising a mixture in water of polyvinyl alcohol, a humidifying agent; a lubricant composition comprising a mixture of mineral oil, an oxidized vegetable oil, a dialkyl phenol, an alkylolamine and a higher fatty acid; and a conditioning agent comprising the desalted and dehydrated alkali-neutralized reaction product of oleum with a mixture of a higher fatty acid, a vegetable oil and mineral oil, which reaction product has been diluted with mineral oil.

2. A composition for the sizing of textile yarns, comprising a mixture in water of polyvinyl alcohol, a humidifying agent; a lubricant composition comprising a mixture of mineral oil, an oxidized vegetable oil, a dialkyl phenol, an alkylolamine, a higher fatty acid and a blending agent; and a conditioning composition comprising the desalted and dehydrated alkali-neutralized reaction product of oleum with a mixture of a higher fatty acid, a vegetable oil and mineral oil having penetrating agent incorporated therein, which reaction product has been diluted with mineral oil.

3. A composition for the sizing of textile yarns, comprising a mixture in water of polyvinyl alcohol, a humidifying agent; a lubricant composition comprising a mixture of mineral oil, an oxidized vegetable oil, a dialkyl phenol, an alkylolamine, a higher fatty acid, an acyl derivative of an ester of a hydroxylated higher fatty acid in which the acyl radical is derived from a lower fatty acid, and a blending agent; and a conditioning composition comprising the desalted and dehydrated alkali-neutralized reaction product of oleum with a mixture of a higher fatty acid, a vegetable oil and mineral oil having a penetrating agent incorporated therein, which reaction product has been diluted with mineral oil.

4. A composition for the sizing of textile yarns, comprising a mixture in water of polyvinyl alcohol, glycerine; a lubricant composition comprising a mixture of mineral oil, an oxidized vegetable oil, a dialkyl phenol, an alkylolamine, a higher fatty acid, an acyl derivative of an ester of a hydroxylated higher fatty acid in which the acyl radical is derived from a lower fatty acid and a blending agent; and a conditioning composition comprising the desalted and dehydrated alkali-neutralized reaction product of oleum with a mixture of a higher fatty acid, a vegetable oil and mineral oil having a penetrating agent incorporated therein, which reaction product has been diluted with mineral oil.

5. A composition for the sizing of textile yarns, comprising a mixture in about 800 parts by weight of water of about 25 to 85 parts by weight of polyvinyl alcohol, about 10 to 40 parts by weight of glycerine; about 6.5 to 19.5 parts by weight of a lubricant composition comprising a mixture of mineral oil, an oxidized vegetable oil, a dialkyl phenol, an alkylolamine, a higher fatty acid, an acyl derivative of an ester of a hydroxylated higher fatty acid in which the acyl radical is derived from lower fatty acid and a blending agent; about 3.5 to 10.5 parts by weight of a conditioning composition comprising the desalted and dehydrated alkali-neutralized reaction product of oleum with a mixture of a higher fatty acid, a vegetable oil and mineral oil having a penetrating agent incorporated therein, which reaction product has been diluted with mineral oil.

6. A composition for the sizing of textile yarns, comprising a mixture in water of polyvinyl alcohol, glycerine; a lubricant composition comprising a mixture of mineral oil, oxidized peanut oil, diamyl phenol, triethanolamine, oleic acid, butyl acetyl ricinoleate and oleyl alcohol; a conditioning composition comprising the desalted, dehydrated sodium hydroxide neutralized reaction product with oleum of a mixture of oleic acid, peanut oil and mineral oil having a diamyl phenol incorporated therein, which reaction product has been diluted with mineral oil.

7. A composition for the sizing of textile yarns, comprising a mixture in about 400 to 1200 parts by weight of water, of about 25 to 85 parts by weight of polyvinyl alcohol, about 10 to 40 parts by weight of glycerine; about 6.5 to 19.5 parts by weight of a lubricant composition comprising a mixture of mineral oil, oxidized peanut oil, diamyl phenol, triethanolamine, oleic acid, butyl acetyl ricinoleate and oleyl alcohol; about 3.5 to 10.5

parts by weight of a conditioning composition comprising the desalted, dehydrated sodium hydroxide neutralized reaction product with oleum of a mixture of oleic acid, peanut oil and mineral oil, and having a diamyl phenol incorporated therein, which reaction product has been diluted with mineral oil.

8. A composition for the sizing of textile yarns, comprising a mixture in about 400 to 1200 parts by weight of water, of about 25 to 85 parts by weight of polyvinyl alcohol, about 10 to 40 parts by weight of glycerine; about 6.5 to 19.5 parts by weight of a lubricant composition comprising a mixture of mineral oil, oxidized peanut oil, diamyl phenol, triethanolamine, oleic acid, butyl acetyl ricinoleate and oleyl alcohol; about 3.5 to 10.5 parts by weight of a conditioning composition comprising the desalted, dehydrated sodium hydroxide neutralized reaction product with oleum of a mixture of oleic acid, peanut oil and mineral oil, and having a diamyl phenol incorporated therein, which reaction product has been diluted with about 60% by weight of mineral oil on the weight of the composition.

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