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METHOD OF TREATING KERATINOUS YARNS AND COMPOSITIONS THEREFOR

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The present invention relates to an improved process of setting the twist of mechanically twisted keratinous yarns and particularly highly twisted keratinous yarns employed in the manufacture of pile fabrics, frieze rugs and the like.

The present invention also relates to a method of introducing kinks in highly twisted keratinous yarns and simultaneously therewith setting such kinks.

In the manufacture of twisted keratinous yarns such as twisted wool yarns which are employed in the production of high quality frieze rugs, pile fabrics and the like, it has been customary to set the twist of the yarns by physical and mechanical methods. However, such methods have not been entirely satisfactory both from the standpoint of cost and the permanency of the set which has been imparted to the twisted yarn. For example, it is known that while rugs and pile fabrics fabricated from the twisted yarns which have been set by the above processes are superior to rugs and pile fabrics fabricated from twisted yarns which have not been set, the yarns therein are not set with sufficient permanence to withstand a number of aqueous cleaning operations without losing a considerable amount of their twist. The result is that the rugs or pile fabrics which are cleaned in this manner undergo marked changes in the appearance of the pile structure.

The present invention provides a process which not only obviates the above described disadvantages, but also makes it possible to set the twist of keratinous yarns, which have been given a high degree of twist, to an extent which has not been possible by the prior art processes.

It is, accordingly, one object of the present invention to provide a simple and efficient process for setting the twist of mechanically twisted keratinous yarns such as wool yarns whereby the twisted yarn possesses, after fabrication into a pile fabric, a high degree of resistance to untwisting when such fabric is subjected to aqueous cleaning operations.

A further object of the present invention is the processing of highly twisted keratinous yarns such as wool yarns whereby the twist of the yarns is set and made resistant to untwisting when subjected to hot aqueous solutions.

A further object of the present invention is the introduction of kinks in highly twisted keratinous yarns and the simultaneous setting thereof whereby the yarn retains such kinks when fabricated into rugs and then cleaned by aqueous cleaning methods.

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A further object of the present invention is the preparation of novel twisted keratinous yarns which are resistant after fabrication into pile fabrics to untwisting when such fabrics are subjected to aqueous cleaning methods or are treated with hot aqueous solutions.

Still further objects and advantages of the invention will appear from the following description and the appended claims.

The present invention is carried out in general by first treating twisted keratinous yarns such as twisted wool yarns, twisted mohair yarns and the like at about 100° to 150° F. with an aqueous dispersion comprising a reducing agent capable of splitting the characteristic cystine linkage of keratin, preferably, reducing agent such as mercapto compounds having at least one hydroxyl group and containing from 2 to 6 carbon atoms, and a light stable thermoplastic resin which when dry is substantially nontacky.

As examples of suitable thermoplastic resins may be mentioned copolymers of styrene with butadiene or isoprene, vinyl resins such as polystyrene, polyvinyl butyral, polyvinyl acetate, polyvinyl acrylates, polyvinyl chloride and the like and vinylidene resins such as polyvinylidene chloride, polyvinyl methacrylates and the like or copolymers of vinyl compounds or vinylidene compounds or vinyl and vinylidene compounds.

After the keratinous yarns have been treated as described for a period of time sufficient to cause the reaction of about 1% to 6% by weight of reducing agent with the keratin, based on the dry weight of the keratinous yarn, the yarn is extracted to remove excess liquid therefrom and is then dried at a temperature between about 90° to 350° F. The yarns may be further finished, if desired, by washing them with an aqueous solution comprising an emulsifying agent and an oxidizing agent capable of reacting with the residual unreacted reducing agent in the yarns and then drying.

The invention may be practiced on dyed or undyed keratinous yarns. However, if the finished yarns are to be employed in a colored rug or pile fabric, they should be dyed prior to treatment because the finish imparted to the yarn prevents satisfactory dyeing.

The twist and kink in the finished yarn is set quite permanently by the process described herein. When such yarn is fabricated into a pile fabric, it is resistant to untwisting when the fabric is subjected to rug shampooing and other aqueous cleaning operations or when the pile

fabric is wet out with hot aqueous solutions. In addition, the tensile strength and abrasion resistance of the yarns is not appreciably affected by such processing.

In carrying out the present invention, best results have been obtained by employing mercapto compounds having one or more hydroxyl groups and containing from 2 to 6 carbon atoms, since such compounds react with the keratin molecule more readily than other reducing compounds. These compounds are also used advantageously since they are compatible with most vinyl or vinylidene aqueous resin dispersions at the temperatures employed herein, that is such compounds do not ordinarily cause precipitation of the dispersed vinyl or vinylidene resin particles. Moreover, it has been found that such mercaptans do not adversely affect the light stability of vinyl or vinylidene resins. However, it is possible in some instances to employ other organic and inorganic reducing agents which are capable of splitting the cystine linkages of keratin. As examples of such reducing agents may be mentioned inorganic agents such as alkali metal bisulfites, sulfites, sulfides, hydrosulfides and cyanides and the like and organic reducing agents such as the lower aliphatic mercaptans, water soluble derivatives of sulfinic acids and lower mercapto aliphatic carboxylic acids and water soluble salts thereof such as thioglycollic acid, sodium thioglycollate, ammonium thioglycollate and the like. It is preferable, particularly when employing organic reducing agents of the type described to select compounds which have a solubility of at least 1% by weight in water since such compounds are employed in an aqueous dispersion and they do not react with the keratin to the extent desired if they are not sufficiently water soluble.

As examples of the preferred reducing agents employed in the present invention may be mentioned 2-mercapto ethanol, 1-mercapto sorbitol and 1-mercapto glycerol.

The organic or inorganic reducing agents employed herein are most effective when supplied in an amount sufficient to react with the wool or other keratin filler to the extent of about 1% to 6% by weight, based on the weight of the dry keratin yarn. This is readily accomplished when employing mercapto-alcohol compounds by treating about 1 part of the wool or keratin fiber with about 20 to 30 parts of an aqueous thermoplast dispersion containing about 1 to 4% by weight of mercapto-alcohol compound. The treatment is generally carried out at about 150° F. for 3 minutes or about 100° F. for 60 minutes or between such temperatures for proportionate periods of time depending on the temperature used. However, the same results may be accomplished at somewhat higher temperatures or for longer periods of time, and it is only necessary that the proper weight of mercapto alcohol or other reducing agent be reacted with the wool or keratin fiber and that the treatment be carried out at a temperature of at least 100° F.

A suitable test for determining the amount of reducing agent reacted with the wool or keratin yarn is to titrate aliquot portions of the aqueous treating solution with iodine, using starch as an indicator, and then calculate the actual quantity of reducing agent removed from the entire treating solution by reaction with the wool or keratin. This value is then converted to a per cent value based on the weight of dry wool or keratin fiber being treated.

In practicing the present invention, either "primary" or "secondary" aqueous dispersions of thermoplastic resin are employed. The term "primary" aqueous resin dispersion as employed herein is intended to cover resin dispersions which have been prepared by the emulsion polymerization of polymerizable monomeric compounds or mixtures of polymerizable, or copolymerizable monomeric compounds. "Secondary" aqueous resin dispersions, on the other hand, is a term employed herein to designate resin dispersions which have been prepared by dispersing either partially or completely polymerized solid or liquid resin particles in an aqueous solution. This may be accomplished by the use of homogenizing apparatus such as colloid mills and the like or by dissolving or dispersing the resin particles in an organic solvent or plasticizer for the resin, forming a water-in-oil type of emulsion or dispersion and then inverting said emulsion dispersion so as to form an oil-in-water emulsion or dispersion.

The above described resin dispersions may comprise a small amount of plasticizer for the resin depending upon the characteristics of the particular resin being used. When the resin used drys to a flexible film no plasticizer need be employed therewith since the treated wool or keratin yarn comprising such resin will be sufficiently soft and flexible for its intended use. However, when resins such as polystyrene, polyvinyl acrylates and the like are employed in accordance with the present invention, it is desirable to employ a small amount of plasticizer therewith since the treated wool will have a softer feel and more flexibility in such instances.

Best results are obtained when from about 1% to 10% by weight of resin solids, based on the weight of dry wool or keratin yarns, are deposited on the wool or keratin yarn. The twisted yarn may be treated with the aqueous dispersion of reducing agent and resin in a variety of ways. A suitable method consists in immersing skeins of the twisted wool or keratin yarns in the dispersion until the desired reaction between the reducing agent and wool is effected and then centrifuging or otherwise extracting the treated skeins until the desired amount of resin has been deposited on the wool yarn. Generally, aqueous dispersions comprising from about 2 to 16% by weight of resin solids are suitable for the treatment described above depending upon the method of extraction used.

A further understanding of the present invention will be obtained from the following examples which are intended to be illustrative, but not limitative of the scope of the present invention, parts and percentages being by weight unless otherwise specified.

Example I

A "primary" aqueous polystyrene dispersion was first prepared as follows:

Forty parts of monomeric styrene, 1.8 parts of sodium stearyl benzene sulfonate as emulsifying agent, 0.07 part of NaH_2PO_4, 0.28 part of Na_2HPO_4, 0.10 part of $K_2S_2O_8$ (catalyst) and 53.55 parts of water were heated in an inert atmosphere of nitrogen at a temperature between about 90° to 95° C. for a period of several hours. The resulting dispersion contained about 40% polystyrene solids and it was diluted until it contained about 4% polystyrene solids.

About 2% of 2-mercapto ethanol was added to the dilute dispersion of polystyrene (as pre-

pared above) and the dispersion was heated to 130° F. A skein of highly twisted wool carpet yarn was immersed in the heated dispersion for about 5 minutes, after which, it was centrifuged and thoroughly dried. About 25 parts of dispersion were employed for 1 part of carpet yarn. The treated yarn was woven into a pile fabric which was then subjected to several aqueous cleaning operations. The twist and kink of the treated yarn was not substantially affected by such cleaning operations.

By preparing an aqueous dispersion containing about 25% dibutyl phthalate (plasticizer) based on the styrene monomer in the preparation of the polystyrene dispersion by emulsion polymerization and then treating a highly twisted wool carpet yarn as immediately described above, it is possible to produce a softer, yet non-tacky yarn.

Example II

A "primary" aqueous polyvinyl acetate dispersion was prepared as follows:

Eight hundred parts of water, 10 parts of methyl cellulose (emulsifying agent), 10 parts of gum arabic (emulsifying agent), 2 parts of sodium salt of the sulphonic acid of dioctyl succinic ester (surface tension depressant), 930 parts of monomeric vinyl acetate and 1 part of benzoyl peroxide were stirred rapidly and the temperature of the emulsion was raised to between 60° to 80° C. and maintained between such temperatures for a period of 2 hours. The resulting dispersion or emulsoid was quite stable and contained about 50% solids. The dispersion was then diluted until it contained 6% polyvinyl acetate solids.

Twenty-five parts of the 6% polyvinyl acetate dispersion (prepared as given above) to which was added 3%, based on the dispersion, of 2-mercapto ethanol was heated to about 130° F. One part of highly twisted wool carpet yarn in skein form was immersed in this dispersion for 5 minutes, after which, it was centrifuged until it contained about 100%, based on the weight of the yarn, of the dispersion and the yarn was then dried. The treated yarn was woven into a frieze rug which was then given several aqueous cleanings. The kink and twist of the treated yarn was found to be set substantially fast to such aqueous cleaning operations.

Example III

One part of highly twisted wool carpet yarn in skein form was immersed for 5 minutes in 30 parts of an aqueous dispersion containing 5% polyvinyl butyral (prepared as a "secondary" aqueous resin dispersion) and 1.5% of 2-mercapto ethanol. The dispersion was maintained at a temperature of approximately 150° F. during the immersion period. Thereafter the skein of yarn was centrifuged and dried. The treated yarn was woven into a frieze rug. The kink and twist of the treated yarn was found to be set substantially fast to aqueous rug cleaning operations.

The polyvinyl butyral employed in this example was prepared by condensing butyraldehyde with a partially hydrolyzed polyvinyl acetate and contained on a weight basis 17 to 20% hydroxy groups calculated as polyvinyl alcohol, less than 3% acetate groups calculated as polyvinyl acetate and the remainder substantially butyraldehyde acetal. The aqueous polyvinyl butyral dispersion was prepared by mixing the resin with butyl ricinoleate (plasticizer) in a Baker-Perkins mill and then slowly adding water to the mix until first a water-in-oil and then an oil-in-water dispersion is formed.

It is apparent, however, that other polyvinyl butyrals differing in hydroxy, acetate and acetal composition from the specific polyvinyl butyral described above may also be employed. Moreover, the aqueous polyvinyl butyral dispersion may be prepared in other ways as, for example, by a homogenizing procedure using a colloid mill for dispersion of the resin particles.

Various modifications may be made in the process described herein and it is intended that the invention be limited only by the scope of the appended claims.

What is claimed is:

1. A process of setting the twist of twisted keratinous yarns which consists in treating said yarns with an aqueous dispersion comprising from 2 to 16% by weight of dispersed particles of a thermoplastic resin selected from the group consisting of vinyl resins and vinylidene resins, which resins are substantially non-tacky when dry, and from about 1 to 4% of an aliphatic mercapto alcohol containing from 2 to 6 carbon atoms which are attached to each other in a carbon-to-carbon linkage, said compound consisting of a mercapto group, at least one hydroxyl group, carbon atoms and hydrogen atoms, until from about 1 to 6% by weight, based on the weight of the dry yarn, of said mercapto compound has reacted with said yarn, the temperature of said dispersion being above about 100° F. during said treatment; extracting said yarns to remove excess liquid therefrom and to deposit from about 1 to 10% by weight of said resin particles based on the dry weight of the yarns; and then drying said yarns.

2. A process of setting the twist of twisted keratinous yarns which consists in treating said yarns with an aqueous dispersion comprising from 2 to 16% by weight of dispersed polystyrene particles and from about 1 to 4% by weight of an aliphatic mercapto alcohol containing from 2 to 6 carbon atoms which are attached to each other in a carbon-to-carbon linkage, said compound consisting of a mercapto group, at least one hydroxyl group, carbon atoms and hydrogen atoms, until from about 1 to 6% by weight, based on the weight of the dry yarns, of said mercapto compound has reacted with said yarns, the temperature of said dispersion being between about 100 and 150° F. during said treatment; extracting said yarns to remove excess liquid therefrom and to deposit from about 1 to 10% by weight of said polystyrene particles based on the dry weight of the yarns; and then drying said yarns.

3. A process of setting the twist of twisted keratinous yarns, which consists in treating said yarns with an aqueous dispersion comprising from about 2 to 16% by weight of dispersed polyvinyl acetate particles and an aliphatic mercapto alcohol containing from 2 to 6 carbon atoms which are attached to each other in a carbon-to-carbon linkage, said compound consisting of a mercapto group, at least one hydroxyl group, carbon atoms and hydrogen atoms, until from about 1 to 6% by weight of said mercapto compound, based on the weight of the dry yarns, has reacted with the yarns, the temperature of said dispersion being between about 100 and 150° F. during said treatment; extracting said yarns to remove excess liquid therefrom and to deposit from about 1 to 10% by weight of polyvinyl acetate particles,

based on the dry weight of the yarns; and then drying said yarns.

4. A process of setting the twist of twisted keratinous yarns which consists in treating said yarns with an aqueous dispersion comprising from about 2 to 16% by weight of dispersed polyvinyl butyral particles and from about 1 to 4% by weight of an aliphatic mercapto alcohol containing from 2 to 6 carbon atoms which are attached to each other in a carbon-to-carbon linkage, said compound consisting of a mercapto group, at least one hydroxyl group, carbon atoms and hydrogen atoms, until from about 1 to 6% by weight of said mercapto compound, based on the weight of the dry yarns, has reacted with said yarns, the temperature of said dispersion being between about 100 and 150° F. during said treatment; extracting said yarns to remove excess liquid therefrom and to deposit from about 1 to 10% by weight of said polyvinyl butyral particles based on the weight of the dry yarns; and then drying said yarns.

5. A process of setting the twist of twisted keratinous yarns which consists in treating said yarns with an aqueous dispersion comprising from about 2 to 16% by weight of dispersed particles of a thermoplastic resin selected from the group consisting of vinyl resins and vinylidene resins, which resins are substantially non-tacky when dry, and from about 1 to 4% by weight of 2-mercapto ethanol until from about 1 to 6% by weight of 2-mercapto ethanol, based on the weight of the dry yarns, has reacted with said yarns, the temperature of said dispersion being between about 100 and 150° F. during said treatment; extracting said yarns to remove excess liquid therefrom and to deposit from about 1 to 10% by weight of said resin particles based on the weight of the dry yarns; and then drying said yarns.

6. A process of setting the twist of twisted keratinous yarns which consists in treating said yarns with an aqueous dispersion comprising from about 2 to 16% by weight of dispersed particles of a thermoplastic resin selected from the group consisting of vinyl resins and vinylidene resins, which resins are substantially non-tacky when dry, and from about 1 to 4% by weight of 1-mercapto glycerol until from about 1 to 6% by weight of 1-mercapto glycerol, based on the weight of the dry yarns, has reacted with said yarns, the temperature of said dispersion being between about 100 and 150° F. during said treatment; extracting said yarns to remove excess solution therefrom and to deposit from about 1 to 10% by weight of said resin particles; and then drying said yarns.

7. A process of setting the twist of twisted keratinous yarns which consists in treating said yarns with an aqueous dispersion comprising from about 2 to 16% by weight of dispersed particles of a thermosetting resin selected from the group consisting of vinyl resins and vinylidene resins, which resins are substantially non-tacky when dry, and from about 1 to 4% by weight of 1-mercapto sorbitol until from about 1 to 6% by weight of said mercapto sorbitol, based on the weight of the dry yarns, has reacted with said yarns, the temperature of said dispersion being between about 100 and 150° F. during said treatment; extracting said yarns to remove excess liquid therefrom and to deposit from about 1 to 10% by weight of said resin particles based on the weight of the dry yarns; and then drying said yarns.

8. A composition for treating keratinous yarns consisting of an aqueous dispersion comprising from about 2 to 16% by weight of dispersed particles of a thermoplastic resin selected from the group consisting of vinyl resins and vinylidene resins, which resins are substantially non-tacky when dry, and from about 1 to 4% by weight of an aliphatic mercapto alcohol containing from 2 to 6 carbon atoms which are attached to each other in a carbon-to-carbon linkage, said mercapto alcohol consisting of a mercapto group, at least 1 hydroxyl group, carbon atoms and hydrogen atoms.

9. A composition for treating keratinous yarns consisting of an aqueous dispersion comprising from about 1 to 4% by weight of 2-mercapto ethanol and from about 2 to 16% by weight of dispersed particles of a thermosetting resin selected from the group consisting of vinyl resins and vinylidene resins, which resins are substantially non-tacky when dry.

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