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PROCESS FOR COATING POLYESTER FIBERS WITH A MIXTURE OF LANOLIN AND AN OILY SILICONE

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ABSTRACT OF THE DISCLOSURE

The process for imparting a durable, aesthetically-pleasing cashmere-like feel to polyester fabrics which comprises coating the fibers of the fabric before being made into yarn with an aqueous emulsion of lanolin and a specific oily silicone with the weight ratio of silicone to lanolin being within the range of 1.5 to 3, and then heating the coated fibers to cross-link the silicone and to provide the improved durable aesthetics.

This invention is concerned with the surface treatment of synthetic fibers. It is particularly concerned with the surface treatment of polyester fibers to add softness and slickness to the fibers.

Certain natural fibers such as cashmere, lambs wool, mohair and alpaca have surface characteristics which make them especially suitable for the manufacture of fabrics with outstanding aesthetic values. These fabrics have a smooth, soft, slick feel or "hand" and good draping qualities which make them luxurious. The synthetic fibers do not possess these qualities to a high degree unless the surfaces of the fibers are modified after spinning.

It is an object of the present invention to modify the surface of synthetic fibers to impart softness and slickness to the fibers to give them the aesthetic qualities of the more luxurious of the natural fibers.

This object is accomplished in the present invention by applying to the fibers, preferably in the form of a tow before cutting into staple, a finish consisting of lanolin and a silicone resin which further polymerizes on the fiber.

The modification of the surface of a fiber at the point of manufacture to produce some particular effect in fabrics prepared from the fiber presents many problems. First it is necessary that the fiber process well in the operations necessary to convert it into a fabric. If it is cut into staple, the staple should card well without forming neps and without fouling up the cards. If the tow is converted to sliver by the Turbo process or the Pacific Converter process, it should provide a sliver with good drafting properties and not produce an excessive amount of short fibers or loose fibers "fly." Many finishes which impart softness, smoothness and slickness to fibers are not satisfactory because the staple fibers will not card.

A further requirement is that the fibers be dyeable. Not only must they take up the dyestuff under normal conditions of dyeing but they should dye uniformly, free of streaks or blotches. And since most fabrics go through hot-wet treatments as in dyeing, usually in the presence of detergents, it is essential that any finish which is to modify the properties of the final fabric must be fast to these wet treatments. Also, when the fabrics are used, especially if used in garments, they will frequently be laundered or dry-cleaned. The desirable finish must be durable to such treatments if the full benefits of the finish are to be realized.

The finish of the present invention, which meets these rigid requirements, comprises a mixture of 1.5 to 3 parts of certain silicone resins per part of lanolin. About 5 to 10% emulsions of these materials are used. Silicone alone

or in higher ratios than stated imparts an oily feel to the fibers and results in poor fiber processability due to lack of yarn cohesion. Lower ratios of silicone to lanolin give finishes with a less pleasant hand and which are not durable to laundering and dry-cleaning. Satisfactory results are obtained when about 0.5 to 2.0%, and preferably 0.75 to 1.50%, of the finish is applied to the fibers.

The preferred method of carrying out this invention consists of passing a tow of the synthetic fiber through an emulsion of the silicone resin and lanolin, squeezing out the excess emulsion, crimping the fibers in a stuffer-box crimper while still wet, either cutting the wet tow into staple or first drying and then cutting. After drying, it is heated further to cross-link the resin on the fiber. The staple is then converted to fabric by the usual methods.

Lanolin is the protective covering on sheep's wool and is recovered in wool scouring. It is widely used in cosmetics and its use as a fiber or fabric finish is disclosed in a number of patents, notably Canadian Patents 540,273 and 581,754 and German Patent 1,004,584.

The emulsion of lanolin and silicone can be readily prepared by the use of a small amount of any of a number of surface-active agents of the anionic, cationic or non-ionic type. The silicone must be cross-linkable after it is applied to the fiber and this property is gained by copolymerizing the following silanes: alkyl trichlorosilane, dialkyl dichlorosilane, trialkyl chlorosilane and alkyl hydrogen dichlorosilane which are represented by the following formulas, respectively: R_3SiCl, R_2SiCl_2, $RSiCl_3$ and $RHSiCl_2$.

A range of silicones can be made by the following procedure: One or more of the chlorosilanes shown above, preferably the methyl chlorosilanes and methyl hydrogen chlorosilanes, is dissolved in dry benzene using 75 parts of benzene to 1 part of the silane. The benzene solution is cooled to 20° C. and 67.5 parts of isopropyl alcohol are added over a period of several minutes with stirring. Then 75 parts of water are added slowly. The polymer is washed several times with water; then the benzene is distilled off at a pressure of 3 mm. and a temperature up to 125° C. The final product is an oily polymer and when alkylhydrogen chlorosilane is included in the silane mixture, the resulting polymer is capable of condensing further by virtue of the hydrogen atoms on the silicon atoms.

The polymers made by this process can be represented by the following general formula:

$$(R)_mH_nSiO_{\frac{4-m-n}{2}}$$

For purposes of this invention m should have a value of 1.0 to 1.75 and n a value of 0.25 to 1.25; R is usually CH_3. Commercial silicones sold under the trade names "Decetex" 104 and ET–5317 are also satisfactory for use in this invention.

More wash-fast results are obtained if a catalyst is used with the silicone to hasten its further polymerization. Traces of certain metal salts of organic acids serve as catalysts. Cobalt, manganese and lead salts of 6- to 10-carbon fatty acids or branched-chain acids are satisfactory. Salts of 2-ethylhexanoic acid are widely used and commercial catalysts are available for the silicone resins. Organic amines such as morpholine are also suitable catalysts.

Lanolin can be used as such or in the form of certain derivatives such as the reaction products of lanolin and ethylene oxide. The polyhydroxyethyl lanolin is more readily emulsified than lanolin itself. Also certain commercial lanolin emulsions can be used such as "Lanolized" WW. sold by Scholler Brothers.

On carrying out this invention the lanolin and the silicone are mixed and added to water containing an emulsifying agent, with vigorous stirring. Alternately, the emulsifying agent can be added to the lanolin and silicone and this mixture then stirred into water. Anionic or nonionic emulsifying agents are preferred. Long chain alcohol sulfates, long chain alkyl sulfonates, alkylphenolpolyethylene glycols, sorbitan mono stearate or oleate, are types that can be used. Small amounts of a long-chain unsaturated alcohol such as hexadecenyl alcohol or octadecenyl alcohol when used with the emulsifying agent will improve the ease of forming the emulsion.

The emulsion of silicone and lanolin is applied to a tow of the fiber to be treated by running the tow through the diluted emulsion, and then through squeeze rolls to remove excess emulsion. The coated tow is crimped by passing it through a stuffer-box crimper to impart about 6 to 15, preferably 8 to 10, crimps per inch. The tow is then dried in a relaxed state and cured at 140° C. for 6 minutes to cross-link the silicone. It is next cut into 3-inch staple.

The cured staple fiber is then carded and spun on the worsted system and the yarn is knit into a fabric; the aesthetics are determined before and after washing, dyeing, and dry-cleaning. The following examples will illustrate the invention in more detail.

EXAMPLE I

A copolymer is made by esterifying (by ester interchange) ethylene glycol and a mixture of terephthalic acid dimethyl ester and sulfonated isophthalic acid dimethyl ester containing 2 mol percent of the latter. The copolymer ester is made by the method disclosed in U.S. Patent 3,018,272 to Griffing and Remington. This is a basic-dyeable copolymer.

The copolymer is spun into a 3 denier per filament fiber by conventional melt spinning and drawing methods. A 15,000 denier tow of this fiber is passed, at 25 yards per minute through an aqueous emulsion containing, by weight, 3.5% silicone, 3.5% lanolin and 0.05% cobalt 2-ethylhexanoate.

The silicone used in this example has a ratio of Si—H to Si—CH_3 groups of 26 to 74. The emulsion is formed by adding the silicone and the lanolin to water containing 0.2 part of an emulsifying agent made by first reacting a mixture of octadecyl, octadecenyl, hexadecyl and hexadecenyl alcohols with 6 mols ethylene oxide, then esterifying with sulfuric acid. The sulfate ester is poured into a solution of sodium hydroxide to form the sodium salt, carefully avoiding a pH below 7.

The emulsion is formed by adding 10 parts of the silicone-lanolin mixture to 100 parts of water containing the emulsifying agent. After emulsification, more water is added to give a silicone content and a lanolin content of 3.5% each. The catalyst is added to the diluted emulsion with the temperature not over 40° C.

The treated tow is then passed through squeeze rolls (leaving 20 parts of the emulsion on 100 parts of fiber or a loading of about 1.4% of solids on the fiber). It is next passed through a stuffer-box crimper to impart about 8 to 10 crimps per inch and dried in a relaxed state on a belt passing through an oven. The dried tow is cured 6 minutes at 140° C. then cut into 3-inch staple and the latter is carded and spun on the worsted system to yield a 1/15 worsted count yarn with 7 turns per inch. No difficulty is encountered in carding and spinning this fiber.

The yarn is knit into a 22 course per inch fabric. The aesthetics of the fabric are judged by a panel of judges, in the gray state, after scouring and after dyeing. The hand of the gray fabric is found to be soft and smooth but slightly greasy and somewhat lacking in slickness. After scouring and after dyeing, the fabric is slightly improved but not sufficiently slick.

EXAMPLE II

The same fiber prepared in Example I is treated with an aqueous emulsion containing, by weight, 4.1% silicone and 2.7% lanolin. About 1.4% of finish, on the dry fiber weight, is left on the fiber after squeezing out the excess. After processing as in Example I, a fabric of improved slickness is obtained which closely approximates the hand of cashmere. The slickness and softness of this fabric is not appreciably diminished by 5 scourings in soap for 20 minutes at 40° C.

This process is especially suitable for finishing polyester fibers such as those of poly(ethylene terephthalate) or polyester formed of dicarboxylic acid other then terephthalic acid, but it will have merit on other synthetic fibers. The finish is useful, not only for providing a pleasant and luxurious feel to fabrics made from the fibers but also to facilitate the processing of the fibers into yarns. The fibers possess the optimum degree of cohesiveness and the correct slip to make them highly satisfactory for carding and spinning where uniform yarns are desired.

Knit fabrics made from fibers treated by the process of the present invention can be napped to make fur-like fabrics which are especially suitable for fur ironing to straighten the fur pile.

The following table gives additional data showing the preferred ratio of components of the finish.

Finish composition	Subjective evaluation	Remarks
100% silicone	Too oily	Poor fiber processability, good durability to laundering and dry cleaning.
100% lanolin	Too greasy	Not durable to dry cleaning.
50/50 silicone/lanolin	Slightly greasy	Durable to laundering, not durable to dry cleaning.
60/40 silicone/lanolin	Smooth and slick, closely approximates cashmere.	Durable to laundering and dry cleaning.
75/25 silicone/lanolin	Smooth and slick but inferior to 60/40.	Do.
80/20 silicone/lanolin	Slick but too oily	

From the foregoing discussion, description and data, it is evident that the present discovery provides a significant advance in the art of improving the aesthetics of synthetic fibers. While the invention has been described with regard to specific detail, it will be appreciated that changes can be made without departing from its scope.

What is claimed is:

1. In the production of synthetic fabrics of fibers of a polyester polymer including the steps of extruding the polymer through a spinneret having capillaries, quenching the extruded polymer to form filaments, combining the filaments to form a tow, drawing and crimping the filaments, making a yarn of the tow, and then forming said fabric by knitting or weaving; the improvement for imparting a durable aesthetically pleasing cashmere-like hand to the fabric which comprises coating the fibers of the tow before making the yarn with an aqueous emulsion of lanolin and an oily silicone having the unit formula

$$(R)_mH_nSiO_{\frac{4-m-n}{2}}$$

where m has a value of 1.0 to 1.75, n has a value of 0.25 to 1.25 and R is lower alkyl, there being 1.5 to 3 parts by weight of silicone per part of lanolin in the emulsion; said fibers being coated with sufficient solution to provide a coating of from about 0.5 to about 2 percent by weight, based on the fibers, of the combined lanolin and silicone; and then heating the coated fibers to cross-link the silicone; said aqueous solution being further characterized as having a catalytic amount of a cobaltic catalyst to enhance silicone polymerization and provide a more wash-fast coating.

2. The process as in claim 1 wherein the coated fibers are heated to a temperature af about 140° C. for a sufficient time interval to cross-link the silicone.

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