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TEXTILE MATERIALS AND METHOD OF PREPARING SAME

William Whitehead, Cumberland, Md., assignor to Celanese Corporation of America, a corporation of Delaware

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This invention relates to textile materials containing filaments or fibers of organic esters of cellulose and to the method of preparing the same by altering the physical and chemical composition of the said filaments or fibers.

An object of the invention is the preparation of fibers or filaments of organic esters of cellulose that are particularly suitable for the building of strong uniform yarns. Another object of the invention is the preparation of staple length fibers of an organic ester of cellulose that need less anti-static finish and which fibers are softer and fuller of hand. Other objects of the invention will appear from the following detailed description.

The formation of "spun" yarn from comparatively short lengths or staples of filaments or threads composed of organic esters of cellulose presents serious difficulty. This is due in part to the fact that such organic esters of cellulose have a very high di-electric constant and are very poor conductors of electricity, and when the filaments are rubbed together during the various operations involved in mixing, carding, combing and spinning a heavy charge of static electricity is produced. Because of the static electrical charge, the staple fibers containing organic esters of cellulose do not adhere to each other readily and cause great difficulty in the textile operations.

The difficulty encountered in forming "spun" yarn from the staple fibers of organic esters of cellulose is also partly due to the nature of the surface of the organic ester of cellulose fibers. The filaments, from which the staple fibers are cut, are formed with a smooth slippery surface which gives to the fibers an undesired coefficient of friction, i. e. the ease with which the fibers slip over each other, thus preventing efficient carding and drafting operations and making for a weaker yarn.

I have found that if the organic ester of cellulose fibers or filaments are treated with a saponifying agent under certain conditions and then treated with an anti-static dressing the tendency to generate static electricity during the textile operations is reduced so that it is no greater than, and even below, that of cotton and wool. Moreover, I have found that such treatment of the fibers eliminates the difficulty encountered in carding and drafting operations for it removes the tendency of the fibers to repel each other and to adhere to the metal of the carding and drafting devices.

I have also found that the fibers treated in accordance with this invention have the desired surface texture to produce the coefficient of friction necessary for satisfactory carding and drafting by the woolen, worsted, cut silk or cotton method of forming slivers, rovings and yarns. The modification of the texture of the fibers, in accordance with this invention, permits the fibers to be formed into small size or count and results in the production of stronger yarns than can be formed from the untreated fibers. For instance, organic ester of cellulose fibers may be formed into yarns of 30 count or finer measured by the cotton method of measuring yarns. By treating the fibers in accordance with this invention there is a remarkable improvement not only in respect to easier processing and cleaner yarn but fly is cut down to a minimum both in the yarn processing and in the weaving operations. Yarns processed in accordance with this invention have less tendency to slub up on the loom.

In accordance with this invention, I modify fibers or filaments of organic esters of cellulose by treating the same with a saponifying agent under such conditions that the acidyl value of the organic ester of cellulose is lowered by less than two percent in eight hours of treatment. After the fibers have been treated with the saponifying agent they may be lubricated or treated with any suitable dressing but preferably with the dressing and in the manner described in U. S. applications S. Nos. 84,976 filed June 12, 1936 and 102,812 filed September 26, 1936.

While this invention is applicable to the preparation of yarns, fabrics and other textile materials containing organic esters of cellulose, it is of particular importance in connection with staple fibers made of organic esters of cellulose wherein the advantages pointed out above are gained. Examples of organic esters of cellulose are cellulose acetate, cellulose formate, cellulose propionate and cellulose butyrate. Fabrics and yarns containing organic esters of cellulose may be treated in accordance with this invention to increase the resistance of slip between warp and weft and to produce a soft full hand. Fabrics which have been treated in accordance with this invention or which are formed of fibers or filaments that have been treated in accordance with this invention have the advantage that they do not attract dirt or lust and do not tend to cling to a person wearing them. Fabrics may be treated in the piece on a winch or otherwise, preferably before dyeing, while yarns may be treated in packages such as bobbins, spools, cones, hanks, etc. However, as this invention is of par-

ticular importance in the preparation of staple fiber containing cellulose acetate, it will be described with reference to the same.

Artificial filaments of organic esters of cellulose may be formed from solutions of the same in suitable solvents by extruding said solutions through orifices into a drying or evaporative atmosphere as in dry spinning, or by extrusion through orifices into a bath containing a precipitating liquid as in wet spinning. These filaments may then be torn, broken or cut into suitable lengths, say from ½ inch to 10 inches or longer, to suit the particular type of process by which they are to be formed into yarns. The artificial filaments may be of any suitable weight per unit length, say from 1 to 30 or more denier, examples of which are 1.9, 2.5, 3 or 5 denier. A mixture of yarns having different deniers may be employed.

The treatment of the staple fibers comprises the effecting of a slow and minute saponification of the same and then the application of a dressing having anti-static properties. The saponification treatment may be accomplished under a number of conditions but these conditions should be such that the acidyl value of the organic ester of cellulose of the fibers is lowered by less than two percent in eight hours. For instance, in treating cellulose acetate fibers having an acetyl value of 54.5%, calculated as acetic acid, it is preferable that after eight hours' treatment the acetyl value of the fibers be above 53% and still more preferable that the acetyl value be about 54%. The saponification in all instances should be so slight that there is substantially no change in affinity for cotton dyestuffs. This slight and slow saponification treatment may be accomplished by employing weak saponifying agents, dilute concentration of saponifying agents, maintaining a low temperature during saponification, or combinations of these expedients. The cellulose acetate may have any suitable acetyl value before saponification, say from 40 to 57 or more acetyl value. Throughout this specification acetyl values is expressed in terms of acetic acid derivable by saponification.

Although any saponifying agent such as sodium or potassium hydroxide, or secondary and tertiary amines such as di- and tri-ethanolamine may be employed in the treatment of cellulose acetate, it is preferable to employ ammonium hydroxide. Ammonium hydroxide in an amount equal to 2 to 5% by weight of the staple fiber in a bath 8 to 20 times the weight of the fibers and maintained at about 28° C. is particularly suitable in the treatment of cellulose acetate fiber having an acetyl value of about 54.5% in that after 8 hours of treatment the acetyl value of said cellulose acetate fiber is between 53.9 and 54.2 and the desired physical alteration of the fibers is also accomplished. Faster treatments do not effect the desired change in the properties of the fibers. In place of ammonium hydroxide, there may be employed from 10 to 20%, based on the weight of the fibers, of tri-ethanolamine or the equivalent of any other saponifying agent.

After treatment with the saponifying agent the fibers are washed neutral and then coated with an anti-static dressing. Any anti-static dressing employed for use with organic esters of cellulose may be employed. However, the dressing disclosed in U. S. application S. No. 84,976 is preferred. This dressing comprises a solution or an emulsion containing from 0.5 to 10 parts of an inorganic electrolyte, from 60 to 90 parts of a lubricant, and from 10 to 35 parts of a lubricating aid such as a sulphonated compound of the fatty alcohol type. By the term "sulphonated fatty alcohol" is meant the fatty alcohols that contain either or both the HSO_3 and the SO_4 groups. The lubricant may be mineral oil, a mineral oil and a fatty acid, or mineral oil, fatty acid and a soap. The inorganic electrolyte may be magnesium chloride, magnesium citrate, magnesium nitrate, magnesium chlorate, ammonium citrate, calcium chloride, calcium nitrate, zinc chloride, or a mixture of two or more of these. The emulsion of lubricant, electrolyte and lubricating aids may be diluted to any suitable concentration or viscosity by the addition of water. The amount of such emulsion applied to or incorporated in the fibers is preferably from 0.5% to 10%, based upon the weight of the fibers, of the substantially water-free emulsion.

The staple fibers of organic esters of cellulose after the above described saponification may be treated with the anti-static dressing by placing the same in bags which are immersed in a bath containing the dressing, or the dressing may be sprayed upon the fibers. Other methods may be employed for applying the dressing. By immersing the staple fiber in a bath containing the dressing and by regulating the time of immersion and temperature of the bath a fiber may be produced having a crinkly or curled character.

After the treatment with the emulsion, the short lengths of filaments or fibers are then subjected to a suitable spinning operation, such as those operations used for the spinning of the short lengths of natural silk, cotton or wool fibers to form threads or yarns. Any of the well-known systems may be employed to form the threads or yarns, such as the cotton system, the spun silk system, the Bradford system, the woolen system, etc. These spinning operations also include the preliminary treatments necessary to present the fibers in the form required for the actual operation of twisting them into yarns. This may include carding, lapping, mixing, spinning, opening up, etc.

In order to further illustrate my invention, but without being limited thereto, the following specific example is given:

Example

Cellulose acetate fibers of 54.5% acetyl value, calculated as acetic acid, are saponified by treating the fibers for eight hours at 28° C. with 4.7% of its weight of ammonia in a bath containing 10 parts by weight of water for each part by weight of fiber. This reduces the acetyl value to 54.05%. The staple fibers are washed neutral and then immersed in a bath of anti-static dressing containing 70 parts of 20 viscosity mineral oil and 30 parts of a sulphonated fatty alcohol and 3 parts of a saturated solution of magnesium chloride. The concentration of the anti-static dressing in the aqueous bath is such that there is deposited on the fibers after removal of the water about 2%, based on the weight of the fiber, of the dressing. The fibers are then processed to yarns by the cotton method of forming yarns. The fibers are found to have the right surface texture for drawing the same into very fine yarns having good strength and uniform diameter. The fibers are noticeably improved in the amount of fly produced in the carding and drafting operations.

Instead of treating the staple fiber by immersing the same in a bath of a saponifying agent, hanks, spools or other packages of continuous filament organic ester of cellulose yarn may be treated in a saponifying bath and these continuous filaments cut to staple fiber. In this method it is preferable to cut the continuous filaments into staple fiber while the same are still wet as this reduces the tendency of the fiber to develop a static electric charge.

In another modification of my invention I carry out the slow saponification of the fibers by immersing them in a saponification bath containing from 4 to 6%, preferably 4.7%, concentration of triethanolamine on the weight of the staple fiber. The saponification bath is preferably from 8 to 20 times the weight of the staple fiber. As soon as the staple fibers have been wetted by the liquid they are withdrawn from the bath and allowed to stand wet for from 6 to 14 hours. The fibers may be centrifuged before allowing to stand. This centrifuging may reduce the liquid content of the fiber to from 40 to 80% by weight of the staple fiber. After standing a desired length of time, say from 6 to 14 hours, the small amount of residual triethanolamine may be washed out and the staple fiber then dipped in the anti-static finish. Instead of washing out the residual triethanolamine the anti-static finish may be formed with a small amount of free fatty acid such as oleic acid and then the fibers immersed in this, thus neutralizing the triethanolamine with the oleic or other fatty acid forming a soap. The soap formed in this manner acts as an electrolyte on the fibre and aids in the anti-static property of the fiber. By this means more staple fiber may be turned out with a same amount of equipment and floor space. Furthermore, by this method of slowly saponifying the staple fiber one obtains a better acetate stability as evidenced by autoclave tests.

In place of triethanolamine in the above described modifications of my invention I may employ any of the non-volatile organic bases such as mono, di- or tri-amines or the tertiary or quaternary substituted ammonia. Obviously these reagents will be used in equivalent amounts to that specified for triethanolamine.

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

Having described my invention what I desire to secure by Letters Patent is:

1. In a method of forming spun yarn containing short lengths of filamentary materials comprising organic esters of cellulose, the step of subjecting filamentary materials to treatment with a saponifying agent under such conditions that the acidyl value of the materials is lowered less than 2% in eight hours of treatment, the amount of saponification effected not exceeding a lowering of the acidyl value by 2%, whereby the tendency of the treated materials to generate static electricity during textile operations is reduced.

2. In a method of forming spun yarn containing short lengths of filamentary materials comprising cellulose acetate, the step of subjecting filamentary materials to treatment witha saponifying agent under such conditions that the acetyl value of the materials is lowered less than 2% in eight hours of treatment, the amount of saponification effected not exceeding a lowering of the acetyl value by 2%, whereby the tendency of the treated materials to generate static electricity during textile operations is reduced.

3. In a method of forming spun yarn containing comparatively short lengths of organic ester of cellulose fibers, the steps of subjecting the organic ester of cellulose fibers to a saponifying agent under such conditions that the acidyl value of the fibers is lowered less than 2% in eight hours, interrupting the saponification before the lowering of the acidyl value exceeds 2%, applying an anti-static dressing to the fibers and spinning the fibers to a yarn by the cotton method of spinning yarns.

4. In a method of forming spun yarn containing comparatively short lengths of cellulose acetate fibers, the steps of subjecting the cellulose acetate fibers to a saponifying agent under such conditions that the acetyl value of the fibers is lowered less than 2% in eight hours, interrupting the saponification before the lowering of the acetyl value exceeds 2%, and applying an anti-static dressing to the fibers.

5. In a method of forming spun yarn containing comparatively short lengths of organic ester of cellulose fibers, the steps of subjecting the organic ester of cellulose fibers to a saponifying agent under such conditions that the acidyl value of the fibers is lowered less than 2% in eight hours, interrupting the saponification before the lowering of the acidyl value exceeds 2%, washing the fibers neutral and immersing the fibers in a bath containing an anti-static dressing.

6. In a method of forming spun yarn containing comparatively short lengths of cellulose acetate fibers, the steps of subjecting the cellulose acetate fibers to a saponifying agent under such conditions that the acetyl value of the fibers is lowered less than 2% in eight hours, interrupting the saponification before the lowering of the acetyl value exceeds 2%, washing the fibers neutral and immersing the fibers in a bath containing an anti-static dressing.

7. In a method of forming spun yarn containing short lengths of filamentary materials comprising organic esters of cellulose, the step of immersing filamentary materials in a bath containing a saponifying agent equivalent to 2 to 5%, based on the weight of the organic ester of cellulose materials, of ammonium hydroxide and maintaining the bath at room temperature such that the acidyl value of the materials is lowered less than 2% in eight hours, the amount of saponification effected not exceeding a lowering of the acidyl value by 2%, whereby the tendency of the treated materials to generate static electricity during textile operations is reduced.

8. In a method of forming spun yarn containing short lengths of filamentary materials comprising cellulose acetate, the step of immersing filamentary materials in a bath containing a saponifying agent equivalent to 2 to 5%, based on the weight of the cellulose acetate materials, of ammonium hydroxide and maintaining the bath at room temperature such that the acetyl value of the materials is lowered less than 2% in eight hours, the amount of saponification effected not exceeding a lowering of the acetyl value by 2%, whereby the tendency of the treated materials to generate static electricity during textile operations is reduced.

9. In a method of forming spun yarn containing comparatively short lengths of organic ester of cellulose fibers, the steps of immersing the fibers in a bath containing a saponifying agent equivalent to 2 to 5% by weight of the staple fiber

of ammonium hydroxide, the amount of saponification effected not exceeding a lowering of the acidyl value by 2%, maintaining the bath at room temperature, washing the fiber neutral and incorporating therewith an anti-static dressing.

10. In a method of forming spun yarn containing comparatively short lengths of cellulose acetate fibers, the steps of immersing the fibers in a bath containing a saponifying agent equivalent to 2 to 5% by weight of the staple fiber of ammonium hydroxide, the amount of saponification effected not exceeding a lowering of the acetyl value by 2%, maintaining the bath at room temperature, washing the fiber neutral and incorporating therewith an anti-static dressing.

11. In a method of forming spun yarn containing comparatively short lengths of organic ester of cellulose fibers, the steps of immersing the fiber for four to eight hours in a bath containing 2 to 5% by weight of the staple fiber of ammonium hydroxide while maintaining the bath at about room temperature, washing the fiber neutral and incorporating therewith an anti-static dressing.

12. In a method of forming spun yarn containing comparatively short lengths of cellulose acetate fibers, the steps of immersing the fiber for four to eight hours in a bath containing 2 to 5% by weight of the staple fiber of ammonium hydroxide while maintaining the bath at about room temperature, washing the fiber neutral and incorporating therewith an anti-static dressing.

13. A staple length fiber of organic esters of cellulose having a modified surface due to a slow saponification and containing as a finish an emulsion comprising an electrolyte, a mineral oil and a sulphonated alcohol compound.

14. A staple length fiber of cellulose acetate having a modified surface due to a slow saponification and containing as a finish an emulsion comprising an electrolyte, a mineral oil and a sulphonated fatty alcohol compound.

15. In a method of forming spun yarn containing short lengths of organic ester of cellulose fibers, the step of subjecting the organic ester of cellulose fiber to a saponifying bath containing triethanolamine, removing the fiber from the bath and reducing the liquid content to between 40 and 80% on the weight of the fiber, allowing the staple fiber to stand in contact with the remaining liquid for from 6 to 14 hours and then coating the fibers with an anti-static finish containing enough free fatty acid to neutralize the triethanolamine.

16. In a method of forming spun yarn containing short lengths of cellulose acetate fibers, the step of subjecting the cellulose acetate fiber to a saponifying bath containing triethanolamine, removing the fiber from the bath and reducing the liquid content to between 40 and 80% on the weight of the fiber, allowing the staple fiber to stand in contact with the remaining liquid for from 6 to 14 hours and then coating the fibers with an anti-static finish containing enough free fatty acid to neutralize the triethanolamine.

17. In a method of forming spun yarn containing short lengths of organic ester of cellulose fibers, the step of subjecting the organic ester of cellulose fiber to a saponifying bath containing triethanolamine, removing the fiber from the bath and reducing the liquid content to between 40 and 80% on the weight of the fiber, allowing the staple fiber to stand in contact with the remaining liquid for from 6 to 14 hours, washing the fibers substantially free of triethanolamine and applying to the fibers an anti-static finish.

WILLIAM WHITEHEAD.