

1

2,990,291

**PROCESS OF PREPARING PIGMENT DELUSTERED
CELLULOSE ACETATE YARN**

Henry William Bartholomay, Waynesboro, Va., assignor
to E. I. du Pont de Nemours and Company, Wilmington,
Del., a corporation of Delaware
No Drawing. Filed June 22, 1960, Ser. No. 37,838
2 Claims. (Cl. 106—192)

This invention relates to delustered cellulose acetate filaments of low abrasivity, and is more specifically concerned with cellulose acetate filaments containing hard particulate delustrants which, nevertheless, show little tendency to abrade metal surfaces upon continuous moving contact of the filaments with said surfaces.

Delustered synthetic yarns find widespread use in the textile industry. Although numerous chemical and physical procedures have been employed in delustering cellulose acetate yarns, it is generally found that such yarns delustered with finely-divided inorganic pigments, homogeneously dispersed in the spinning solution, have the best uniformity and stability to luster changes. Unfortunately, the most desirable pigment for delustering purposes is calcined titanium dioxide, which is a hard material, and cellulose acetate yarns containing these pigment particles rapidly abrade the metal parts of textile machinery which are in moving contact with these yarns. Previous attempts to treat such delustering pigments to reduce their abrasive nature have had a detrimental effect on their whiteness and covering power, and generally decreased the stability of dispersions of the delustrant in cellulose acetate spinning solutions to the extent of producing poor spinning continuity and poor uniformity of yarn quality. These treatments in the past were generally chemical in nature and were effected in the pigment manufacturing step. All efforts to reduce abrasivity by these means result in either optical or stability deficiencies.

It is an object of this invention to provide a process for preparing pigment delustered cellulose acetate yarns and fibers of reduced abrasiveness while retaining and even enhancing desirable whiteness and covering power. It is a further object to provide a spinning solution of cellulose acetate containing a uniform dispersion of titanium dioxide particles having whiteness and covering power acceptable for yarn delustering purposes, said solution being spinnable with good continuity to form delustered yarn of reduced abrasivity. Other objects and advantages will become apparent from the disclosure and claims which follow.

The objects of this invention are accomplished by the improvement, in the process of preparing a spinning solution of cellulose acetate and spinning the solution into filaments, of sand grinding a slurry of hard, white, inorganic pigment in an acetone solution of cellulose acetate, containing 10–40 parts by weight of pigment and 2–6 parts of cellulose acetate per 100 parts of slurry, by high speed agitation with an amount of 20–40 mesh sand which provides an average liquid distance between sand granules in the slurry of 0.5–2.0 millimeters and then separating the sand to produce a pigment dispersion of 0.1–1.0 micron particle size having a viscosity of 1 to 30 poises, and intimately mixing this initial dispersion with at least 10 times its weight of an acetone solution of cellulose acetate having at least 4 times greater concentration of cellulose acetate than that of the dispersion to provide a spinning solution suitable for extrusion to form delustered filaments. The abrasiveness of the filaments is reduced to a remarkable extent, in comparison with previous filaments containing comparable amounts of delustering pigment. The whiteness and covering power are at least equivalent and generally superior to previous delustered filaments.

2

The grinding step is suitably carried out with a sand grinder of the type shown in Hochberg et al. U.S. Patent No. 2,855,156, issued October 7, 1958, and assigned to the assignee of the present invention, but critical process conditions must be observed. The white inorganic pigment particles used for preparing delustered filaments exist mainly as strongly bonded aggregates which must be broken down and thoroughly dispersed and stabilized against reagglomeration prior to satisfactory use as delustering agents. The grinding conditions are extremely important, seemingly subtle variations of critical features exerting great effect on the abrasive nature and delustering qualities of the particles and the stability of the dispersed particles toward re-agglomeration. Re-agglomeration of dispersed pigment particles upon mixing the initial dispersion with the spinning solution is avoided by the critical selection of a ratio of pigment concentration in the initial dispersion to pigment concentration in the spinning solution of at least 10 to 1 and preferably less than 100 to 1, about 20–80 to 1 being generally desirable, and the selection of a critical ratio of cellulose acetate concentration in the initial dispersion to cellulose acetate concentration in the delustered spinning solution of about 1 to 5, the concentration in the latter being at least 4 times and preferably less than 10 times the concentration of cellulose acetate in the initial dispersion.

The abrasive nature of the delustered yarn is measured by a standardized silver wire test wherein filamental yarn of 150 denier to be tested is run over a 28 gage wire of "fine" silver at a tension of 55 grams and a speed of 550 yards per minute. The total number of yards of delustered yarn run over the wire before the wire is severed is noted and taken as a measure of the abrasivity of the yarn. A low value for this number is indicative of a highly abrasive yarn. For currently commercial delustered cellulose acetate yarn, a value of 9,000 is generally considered acceptable although higher numbers i.e., less abrasive yarns, have long been sought.

In the following example, which illustrates a specific embodiment of the invention, parts and percentages are by weight unless otherwise indicated:

Example

A premixture slurry was prepared by adding 2,500 parts of titanium dioxide of Du Pont 33 grade to a solution of 500 parts of cellulose acetate of yarn grade quality in 7,000 parts acetone while stirring at a rate sufficient to prevent much TiO_2 pigment from settling out. The slurry thus obtained was pumped at a rate of 25 parts per minute through the bottom of a sand grinding apparatus similar to that shown in FIGURE 1 of U.S. Patent 2,855,156, loaded with 3,700 parts of 20–40 mesh Ottawa sand. The residence time of the slurry in the sand grinder was about one hour. The impeller shaft r.p.m. was 900. The average dimension of liquid between sand granules was about 1 mm. as determined by theoretical calculations based on assuming sphericity of the granules. The initial dispersion of TiO_2 particles of 0.1–1.0 micron size emanating from the top of the sand grinder was intimately mixed with 615,000 parts of a 25% solution of cellulose acetate in acetone by paddle agitation to produce a delustered spinning solution containing 0.4% TiO_2 , 24.6% CA, and 75% acetone. The delustered spinning solution was dry spun through a 48-hole spinneret having 0.045 mm. diameter orifices, to produce a yarn of 150 denier. The delustered cellulose acetate yarn thus obtained exhibited excellent whiteness and covering power and, when tested for its abrasiveness toward a metal-wearing surface via the silver wire test, it was found that 22,000 yards of this yarn could be passed over the wire before severing the wire.

To illustrate the improvement obtained by the dis-

persing procedure of the present invention in comparison with the results obtained by ball milling, a 200-gram portion of the premixture slurry of the example was placed in a one-quart size ballmill containing 400 balls averaging 1/4" in diameter. The ballmill was rotated at a rate of 50 r.p.m. for 48 hours. The dispersion thus obtained, resembling the initial dispersion of the example in weight composition, was used to produce a delustered cellulose acetate spinning solution of the same concentration as that of the example and was dry spun in the same manner. The yarn obtained was found to have an unsatisfactorily high abrasivity as indicated by the fact that only 8,600 yards of yarn were required to cut through the standard silver wire.

The present invention provides a very satisfactorily delustered cellulose acetate yarn, produced from the spinning of a solution of cellulose acetate containing 0.3 to 2.0% homogeneously dispersed, hard, white, inorganic pigment particles of 0.1 to 1.0 micron size, which possesses hitherto-unobtainably low abrasive properties in the range of 20,000 to 30,000 as measured in the above described test.

Since many different embodiments of the invention may be made without departing from the spirit and scope thereof, it is to be understood that the invention is not limited by the specific illustrations except to the extent defined in the following claims.

I claim:

1. In the process of dispersing hard, white, inorganic pigment in cellulose acetate spinning solution and spinning the solution to form delustered cellulose acetate

yarn, the improvement for reducing the abrasiveness of the resulting yarn which comprises sand grinding a slurry of the inorganic pigment in an acetone solution of cellulose acetate, containing 10-40 parts by weight of pigment and 2-6 parts of cellulose acetate per 100 parts of slurry, by high speed agitation with an amount of 20-40 mesh sand which provides an average liquid distance between sand granules in the slurry of 0.5-2.0 millimeters and then separating the sand to produce a pigment dispersion of 0.1-1.0 micron particle size having a viscosity of 1 to 30 poises, and intimately mixing this initial dispersion with at least 10 times its weight of an acetone solution of cellulose acetate having at least 4 times greater concentration of cellulose acetate than that of the initial dispersion to provide a spinning solution suitable for extrusion to form delustered filaments.

2. A spinning solution for preparing delustered cellulose acetate filaments of reduced abrasiveness which comprises an acetone solution of cellulose acetate and 0.3 to 2.0% by weight of homogeneously dispersed, hard, white, inorganic pigment particles of 0.1 to 1.0 micron size prepared as defined in claim 1.

References Cited in the file of this patent

UNITED STATES PATENTS

2,581,414	Hochberg	Jan. 8, 1952
2,701,211	Taylor et al.	Feb. 1, 1955
2,819,173	Dithmar	Jan. 7, 1958
2,855,156	Hochberg	Oct. 7, 1958
2,868,662	Ingham et al.	Jan. 13, 1959