

1

3,118,060

**CONTROL OF FINISH ON FIBROUS MATERIAL BY THE USE OF A FLUORESCING SUBSTANCE**

Elias Klein, Mobile, Ala., assignor, by mesne assignments, to Courtaulds, Limited, London, England, a British company

No Drawing. Filed Oct. 16, 1959, Ser. No. 846,794

5 Claims. (Cl. 250—71)

This invention relates to a process for finishing fibrous materials and in particular to a process for controlling the amount of finishing agent applied to fibrous materials.

In the production of synthetic fibers, whatever the chemical composition, it is customary to apply one or more external finishing agents at some stage in the process. These finishes are used to add desirable properties such as lubricity, hand or scoop to the fibrous material, to serve as static suppressors or for any of a variety of other functions.

For economic reasons and to maintain consistent quality of product it is desirable to control the amount of agent which is applied to the fiber. However, because of the rather complex chemical structure of many finishes, chemical analysis of the treated fiber for the purpose of determining the amount of finish that has been applied is not usually practicable.

It is an object of the present invention to provide a simple and economical method for controlling the amount of finish applied to fibrous material.

It is another object of the invention to provide a method for controlling the amount of finish applied to a fibrous material, which avoids chemical analysis of the treated fiber.

Other objects will appear from a consideration of the following specification and claims.

In accordance with the invention, a method is provided for controlling the amount of finish applied to fibrous material which comprises adding to the finish prior to its application to the fiber, a fluorescent material which is miscible with the finish but not substantive to the fiber, and irradiating the fiber after the finish has been applied to actuate the fluorescent substance. The degree of fluorescence is then a measure of the amount of finish which has been applied to the fiber. This can be measured by visual comparison with a sample or by fluorimeter, depending on the accuracy required.

The invention is applicable to a great variety of fibers including fibers of natural and regenerated cellulose, cellulose esters, e.g. cellulose acetate, cellulose ethers, acrylic fibers, such as fibers made from acrylonitrile polymers and copolymers, nylon, and polyester fibers such as fibers made from polyethylene terephthalate.

The fiber may be treated with the finish in the form of staple, as a continuous filament, as a tow, or after it has been made into yarn or fabric.

The finishing agent may be selected from among the wide variety of materials known to the art. In particular the invention is applicable to so-called "external" finishing agents, i.e. those finishes whose effects are obtained by deposits and alterations on the peripheries of the fibers. The specific agent used will depend on the type of fiber being processed and on the qualities which it is desired to impart.

Among the external finishes to which the present invention may be applied are softening agents, film-forming finishes, surface deposits and corrective finishes.

Softening agents are applied to improve the hand of textiles, subduing harshness and coarseness. They generally include surface active substances having a hydrophobic group derived from vegetable, animal or mineral oils and a hydrophilic group. Typical hydrophobic groups are dodecyl, tetradecyl, hexadecyl, octadecyl, heptadec-

2

enyl, stearyl, and oleyl groups. Common hydrophilic groups include sulphate, sulfonate, phosphate, carboxyl ( $-\text{COOH}$ ) amino sulphate ( $-\text{NH}_2\text{HSO}_4$ ), substituted ammonia ( $-\text{NH}_2\text{R}$ ,  $-\text{NHRR}'$ ,  $-\text{NR}'\text{R}''\text{R}'''$ ) and polyoxyethylene, for example, polyoxyethylene and polyoxypropylene groups.

Film-forming finishes are usually applied to add abrasive resistance to textiles and are normally applied after the fabric has been formed. These materials include starch, synthetic resins such as urea-formaldehyde, polyvinylacetate, polyvinyl alcohol, polyacrylate and methacrylate and polystyrene resins, proteins such as gelatins and glues, and gums such as gum arabic, gum tragacanth and Tragosol.

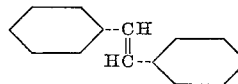
Surface deposit finishes include dulling and delustering agents. These are normally dispersions of pigments such as  $\text{TiO}_2$ ,  $\text{ZnO}$  and  $\text{ZnS}$  in oils or other media.

Corrective finishes include water repellents, flame inhibitors, moth repellents, dimensional stabilizers, crush retarders, and antiseptic agents. Obviously the chemical nature of such agents will vary widely.

Although it is possible to use the invention with all of the above types of finishes, it finds its principal use in connection with softening agents.

The fluorescent material will be chosen having regard to the nature of the finishing agent and of the fiber. It should preferably dissolve in the finishing agent. In the case of softening agents, most are oily materials and are applied as aqueous dispersions or emulsions. Thus in this particular instance the fluorescent material is preferably water insoluble. The material ought not to be substantive to the fiber, so that the fiber will not take up more of the fluorescent material than of the finishing agent. Many substances meet these requirements. Those which are commercially available and practical to use include stilbene, substituted stilbenes, anthracenes and commercial brightening agents classified in the Colour Index, Second Edition, under C.I. Fluorescent Brighteners 68, 69 and 70.

By "substituted stilbenes" are meant compounds in which one or both the phenyl groups of the stilbene mole-



cule have from 1 to 5 substituents selected from the group consisting of amino, hydroxy, alkyl, or alkoxy. Preferably the substituent chain will have not more than say six carbon atoms.

The fluorescent material is normally mixed with the finish in proportions of from about 0.1 to about 1% on the weight of the finishing agent. This leads to a concentration of fluorescent material on the fiber of between about 1 and about 100 parts per million.

The finish containing the fluorescent material may be applied by any conventional means. Normally, as indicated above, the finish is applied as an aqueous emulsion containing say 0.1 to 2.0% by weight of the finishing agent. Depending on the physical form of the fibrous material this emulsion may be applied as a spray, a bath or by means of padding rolls, to name but a few conventional instrumentalities.

After the finishing agent has been applied the fiber is normally squeezed to remove excess agent. It may then be passed under a suitable source of ultra-violet radiation; for example a Hanovia lamp. An operator may be stationed nearby to visually compare the material being produced with a standard sample. Alternatively a fluorimeter may be installed to measure directly the intensity of the fluorescence.

It may also be desirable to measure the composition of the finishing liquor actually applied to the fiber. This

3

may be done by adding the fluorescent material to the finishing agent before the agent is dispersed in the medium by means of which it is to be applied, then irradiating the dispersion and comparing the resulting fluorescence to a standard, or measuring it directly by means of a fluorimeter.

The invention will be further described with reference to the following specific examples which are given for purposes of illustration only and are not to be taken as in any way limiting the invention beyond the scope of the appended claims.

#### Example I

Viscose rayon staple fiber, newly spun, is sprayed with an aqueous emulsion containing 0.5% polyglycol stearate. This emulsion is prepared by dispersing 1 gm. Blanchophor AS (C.I. 68) in 1000 gms. of molten stearate and then dispersing the stearate in an appropriate amount of water. The concentration of stearate in the solution is checked by diluting a sample 1.5 times with ethanol, irradiating the sample with ultra-violet light and comparing the fluorescence with a standard sample.

The fiber is squeezed to retain 0.4% finish on the fiber, dried at 80-90° C. and then visually compared under ultra violet light with a standard having the correct amount of finish. Using this system a constant quality of fiber is obtained.

#### Example II

A finishing liquor is prepared by dissolving 5 grams of trans-stilbene in 1000 gms. of Sapamine EH and then dispersing the mixture in sufficient water to form a 0.4% emulsion Sapamine EH is a concentrated aqueous polyethylene glycol monopalmitate dispersion containing about 50% solids. Washed newly spun viscose staple fiber is immersed in the liquor, squeezed and dried. It is then compared with a standard under ultra violet light. By this means good control of the amount of finish applied is obtained.

What is claimed is:

1. In a process wherein a finish is applied to fibrous material, a method for determining the amount of finish which has been applied to the material which comprises adding to the finish, prior to the application of said finish to said fibrous material, a predetermined proportion of a fluorescent substance, miscible with said finish but not

4

substantive to said fibrous material, applying said finish to said material, irradiating said material to which said finish containing said fluorescent substance has been applied with radiation capable of actuating said fluorescent substance, and measuring the intensity of the fluorescence.

2. The process claimed in claim 1 and comprising measuring the intensity of fluorescence by visually comparing the fibrous material being processed with a standard.

3. In a process wherein a water-insoluble finish is applied to fibrous textile material as an aqueous dispersion a method for controlling the amount of finish applied which comprises adding to the finish, prior to its application to said textile material a predetermined proportion of a water insoluble fluorescent substance, soluble in said finish but not substantive to said textile material, applying said finish containing said fluorescent substance to said textile material, drying said material, irradiating said material after applying the finish thereto to develop fluorescence from said fluorescent substance, and measuring the intensity of the resulting fluorescence.

4. The process claimed in claim 3 wherein the intensity of the resulting fluorescence is measured by comparison with a standard.

5. In a process wherein a softening agent is applied to fibrous material as an aqueous dispersion, a method for controlling the quantity of softening agent applied to the material which comprises adding to the softening agent, prior to the application of said agent to said fibrous material, between about 0.1 and about 1.0% of a water insoluble fluorescent substance miscible with said agent but not substantive to said fibrous material, applying said agent containing said substance to said material, subjecting said fibrous material to ultra violet radiation to develop fluorescence from said fluorescent substance, and measuring the intensity of the fluorescence.

#### References Cited in the file of this patent

##### UNITED STATES PATENTS

2,412,817	Lacey et al. ....	Dec. 17, 1946
2,469,961	Gottshalck .....	May 10, 1949
2,671,250	Fidell .....	Mar. 9, 1954
2,920,202	Motter .....	Jan. 5, 1960