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PRODUCTION OF ORGANIC DERIVATIVE OF
CELLULOSE STAPLE FIBERS BY SOLVENT
DYEING, MECHANICAL CRIMPING, AND
CUTTING IN THAT ORDERCyril M. Croft, Ocotlan, Mexico, assignor to Cella-
nese Corporation of America, a corporation of
DelawareNo Drawing. Application June 12, 1947,
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3 Claims. (Cl. 19—1)

1

This invention relates to the dyeing of textile materials and relates more particularly to an improved process for the production of dyed staple fiber materials having a basis of cellulose acetate or other organic derivative of cellulose.

Staple fibers having a basis of cellulose acetate, or other organic derivative of cellulose, are widely employed in the textile art either alone or in admixture with various other textile fibers for the production of spun yarns by spinning operations employing the cotton, woolen or worsted system. The spun yarns obtained may then be woven into fabrics and the latter dyed in solid shades or, if other textile materials are present, they may be cross-dyed, to yield novel effects. In many applications, however, as in the production of patterned carpeting or suiting materials, the staple fiber spun yarns of cellulose acetate or other organic derivative of cellulose must be dyed prior to weaving since cross-dyeing or printing of such heavy fabric or pile materials is not commercially feasible.

Dyed cellulose acetate or other organic derivative of cellulose staple fibers are usually produced from continuous filaments having a basis of cellulose acetate or other organic derivative of cellulose by first crimping the filaments to impart resiliency and elasticity thereto, and then cutting the crimped continuous filaments to the desired staple fiber length followed by dyeing. However, the dyeing of staple fiber materials in the mass usually twists, snarls, tangles and mats the staple fibers so badly that when the dyed staple fibers are opened and carded in preparation for the spinning operations whereby the spun yarns are formed, a serious amount of tearing and the like results. As a consequence, the average fiber length of the dyed staple fiber materials is seriously reduced and a considerable amount of waste in the form of extremely short fibers must be removed and discarded. In addition, the dyeing of staple fiber requires the use of a rather large volume of dye-liquor due to the voluminous nature of the staple fiber.

It is, accordingly, an important object of this invention to provide an improved process for the production of dyed staple fibers having a basis of cellulose acetate or other organic derivative of cellulose.

Another object of this invention is the provision of a continuous process for the rapid and efficient production of dyed cellulose acetate or other organic derivative of cellulose staple fiber materials, whereby the quantity of short ends of staple fiber removed during the opening and pick-

2

ing of said fibers when forming yarns is reduced to a minimum and the resiliency and elongation of the dyed staple fibers remain unaffected.

Other objects of this invention will appear from the following detailed description.

I have found that dyed staple fibers having a basis of cellulose acetate or other organic derivative of cellulose may be produced efficiently and economically, and in a continuous manner, by an improved process which comprises dyeing a tow or bundle of continuous filaments of cellulose acetate or other organic derivative of cellulose material as said filaments emerge from the spinning cabinet or metier in which they are formed, washing the dyed continuous filaments directly after dyeing, crimping the same and then severing the dyed filaments to desired staple fiber lengths. The dyeing operation is effected most advantageously if the dyebath employed comprises an aqueous solution of a dyestuff for said cellulose acetate or other organic derivative of cellulose filaments containing a lower aliphatic alcohol and a salt of thiocyanic acid which has a swelling action on said filamentary materials. Preferably, I employ driven squeeze rolls to compress the bundle of filaments as it enters the dyebath so that the compressed bundle will expand in the dyebath and be thoroughly penetrated. Squeeze rolls are also preferably employed to remove excess dye liquor as the dyed filaments leave the dyebath. The squeeze rolls comprise a driven lower roll and a freely floating upper roll, the lower rolls being each driven at a controlled uniform speed to avoid any tension on the filaments during the dyeing operation. After the excess dye liquor is removed by the squeeze rolls and the dyed filaments are washed directly as they leave the dyebath, a lubricating and anti-static finish is then usually applied to said continuous dyed filaments. The dyed and conditioned filaments are then dried to a moisture content of 20 to 30% by weight, crimped and the crimped continuous filaments are then dried to a moisture content of about 6% by weight, cut to staple fiber lengths and the dyed cellulose acetate or other organic derivative of cellulose staple fiber materials thus obtained then collected in a suitable manner. By my novel process a greatly increased rate of production may be attained, excessive tangling and snarling are avoided and, when spun yarns are prepared by subjecting the dyed staple fibers to the sequence of textile operations employed for the production of said spun yarns, very little waste in the form of excessively short fibers is obtained.

The lower aliphatic alcohols which are suitable for use in forming the dyebath are those alcohols which are solvents for the dyestuffs employed and which exert at least a swelling or softening action on the organic derivative of cellulose. While I prefer to employ ethyl alcohol in the dye liquor, other alcohols such as methyl alcohol, propyl alcohol, isopropyl alcohol, n-butyl alcohol, secondary butyl alcohol, tertiary butyl alcohol, n-amyl alcohol, secondary amyl alcohol and tertiary amyl alcohol, or mixtures of these alcohols may be used. The amount of the alcohol used in the dye liquor will vary depending upon the alcohol or alcohols used. I have found that optimum results are obtained when the lower aliphatic alcohol comprises at least 75% by volume of the dyebath.

As examples of the salts of thiocyanic acid which may be employed as swelling agents for the organic derivative of cellulose materials, there may be mentioned ammonium thiocyanate, sodium thiocyanate and potassium thiocyanate, as well as other salts such as zinc thiocyanate. Optimum results are achieved employing sodium thiocyanate, and preferably, in amounts of from 25 to 35 grams per liter of dyebath.

Various dyestuffs for organic derivative of cellulose materials may be employed in accordance with my novel process. These dyestuffs may be acid dyes which are ordinarily used for the dyeing of wool and which do not possess any substantial affinity for organic derivative of cellulose materials when applied by the methods usually employed for applying said acid dyestuffs, as well as those dyes which are relatively insoluble in water and which are usually applied to organic derivative of cellulose materials in the form of aqueous dispersions. Mixtures of these dyestuffs may also be employed. When these dyestuffs are applied to the cellulose acetate or other organic derivative of cellulose continuous filaments by this dyeing method in aqueous alcoholic solutions containing a salt of thiocyanic acid which is a swelling agent for said materials, in accordance with my novel process, the dyeing of said filamentary materials not only takes place very rapidly but the shades obtained are uniform and level and of increased depth and brilliance as well. The speed at which dyeing may be effected enables the continuous filaments to be gathered together as a thick tow as the same emerge from the spinning cabinets in which they are formed and to be entered into the dyebath and dyed in said form so that the whole operation from spinning to collecting the dyed, cut staple comprises a continuous process.

The most advantageous results are obtained in accordance with my novel process when employing acid dyestuffs. Examples of acid dyestuffs which may be employed in the production of dyed staple fiber materials are Artol Blue BL (C. I. No. 833), Artol Blue 6G (Sandoz), Gycolan Black GAL (Geigy), Cloth Fast Brown GRA (Ciba), Artol Red BL (Sandoz), Sulfonine Cyanine Black DN (General Dyestuff), Artol Blue GL (C. I. No. 833), Azo Rubinole 3GP (Sandoz), Neolan Orange R (P. R. No. 146), Alizarine Sky Blue B (C. I. No. 1088), and Vitrolan Orange R (P. R. No. 146).

The concentration of dyestuff in the dyebath will, of course, vary not only with the final shade desired in the staple fiber material but also on the length of time the tow of continuous filaments is present in the dyebath and may be from about 1% up to about 30%. The temperature of the

dyebath during the dyeing operation is preferably maintained between 25 and 32° C. and optimum results are obtained when the temperature is about 30° C. The dyebath is continuously replenished as it is used up in dyeing the tow passing through said bath, the composition of the makeup dyebath being carefully controlled with respect to both the concentration of the dyestuff and alcohol and the temperature so that an excessive variation in the temperature of the dyebath is not effected when the makeup solution is added thereto.

The dyebath is applied to the tow of continuous filaments by introducing the tow into the dyebath by means of squeeze rolls and then guiding the tow through the dyebath along a path defined by a series of submerged guide rolls. On leaving the dyebath the excess dye liquor is expressed by means of a second set of squeeze rolls. Any size tow can be dyed in accordance with my process and any number of individual tows may be dyed at the same time depending upon the width of the padding rolls. Usually, from about six to twelve or more strands of tow may be dyed conveniently in a single operation, the apparatus employed being adjusted for the denier of the tow being dyed.

The tow of continuous filaments which comes from the spinning cabinet or metier in which the individual filaments are formed is ordinarily in a somewhat moist condition and contains residual acetone, the volatile solvent employed in the spinning operation, together with some water which is placed thereon when the lubricating finish and/or an anti-static finish is applied thereto in emulsion form, for example. The most desirable results are obtained from the point of view of even dyeing in level shades if the tow of continuous filaments is thoroughly dried and cooled prior to dyeing. The tow may be dried by passing the same through a tube through which hot air at 80 to 90° C. is circulated, preferably, in the same direction as the movement of the tow. The dry tow is then preferably cooled so that it is at a uniform temperature, e. g. about room temperature, prior to entering the dyebath since an excessively high and/or uneven temperature also effects the levelness of the dyeing. Cooling may be effected conveniently by blowing the tow with a stream of cool compressed air. When cooled it may then be entered into the dyebath.

The dyeing is effected by passing the tow through the dye liquor under submerged guide rolls. The squeeze rolls employed are all preferably synchronized so that no tension will be placed upon the filaments during the dyeing operation. The speed at which dyeing takes place depends upon the rate at which the dyestuff is absorbed by the filaments. I have found that for dark shades a speed of about 25 yards per minute is a practical maximum, while for lighter shades speeds up to about 40 yards per minute may be employed.

The tow coming from the dyebath is passed through the squeeze rolls to remove excess dye liquor and then directly into a washing trough containing cold water where any surface dye, excess solvent or swelling agent is washed off. After the initial washing, the tow is again passed through a set of squeeze rolls and washed with warm water in a second trough. The washed tow then passes through another set of squeeze rolls where excess water is removed, after which a lubricating and anti-static finish is applied thereto. Following application of the finish, the

5

excess is removed by a set of squeeze rolls, the tow passed through skying rolls, dried to a moisture content of 20 to 30% by weight and then into containers. It is then ready for processing through the crimping device. The latter may comprise a closed box provided with an entry port through which the continuous filaments are stuffed under pressure. The period during which the tow is present in the crimping box tightly packed and under pressure with a controlled amount of moisture thereon is sufficient to impart a permanent random crimp thereto. The crimped filaments are continuously withdrawn from the stuffing box at the same rate at which they are entered. The crimped tow removed from the stuffing box is then dried to a moisture content of about 6% by weight and then fed into a cutting device where it is cut into staple fiber lengths. The lower moisture content enables the tow to be stretched fairly taut so that it may be cut into uniform lengths without affecting the crimp effect. After the cut staple fibers are collected they are subjected to further processing into spun yarns.

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

Example

A dyebath is prepared by dissolving $\frac{1}{4}$ grams per liter of Artol Blue GL, 4 grams per liter of Cloth Fast Orange G and $1\frac{1}{2}$ grams per liter of Neolan Orange R in a mixture comprising 250 parts by volume of water and 750 parts by volume of 95% ethyl alcohol, and then 25 to 30 grams per liter of sodium thiocyanate are added thereto. The bath is maintained at a temperature of 30° C. and the volume adjusted to the amount of tow being dyed. A tow of continuous cellulose acetate filaments coming directly from the spinning cabinet or metier in which said filaments are formed is dried with air heated to 80 to 90° C., the dry tow cooled to about 25° C. with compressed air and then dyed with the above dyebath. The tow is immersed in the dyebath for 1 to 3 seconds, being guided over submerged rolls and passed through said bath at a rate of 25 yards per minute. Excess dye liquor is expressed from the dyed tow by means of squeeze rolls. The dyed tow is washed directly as it leaves the dyebath in cold water, followed by washing in warm water. The excess water is removed and a suitable lubricating and anti-static finish is then applied to the continuous tow. After drying to about 25% moisture content the tow is then crimped. The crimped tow is dried to about 6% moisture content and then severed into staple fiber averaging 1 to 3 inches in length, or any other desired length. The staple fibers obtained are dyed a tan shade and are of superior fastness to gas fading, washing, dry cleaning and light.

While my invention has been more particularly described in connection with the production of dyed cellulose acetate staple fibers, filaments having a basis of other organic derivatives of cellulose may also be dyed in accordance with my process. Examples of other organic derivatives of cellulose are cellulose esters such as cellulose propionate, cellulose butyrate and mixed esters such as cellulose acetate-propionate and cellulose acetate-butyrate, while examples of cellulose ethers are ethyl cellulose and benzyl cellulose.

It is to be understood that the foregoing de-

6

tailed description is given merely 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. Process for the production of dyed staple fiber materials, which comprises dyeing a tow of continuous organic derivative of cellulose filaments with a dyebath comprising an aqueous solution of a lower aliphatic alcohol containing a dyestuff for said organic derivative of cellulose filaments and a salt of thiocyanic acid which is a swelling agent therefor, washing the dyed filaments directly after dyeing, applying lubricating and anti-static conditioning agents to said dyed, continuous filaments, drying the tow to a moisture content of from about 20 to 30% by weight, imparting a crimp to the dyed and conditioned filaments by mechanically deforming the filaments under pressure, drying the crimped tow to a moisture content of about 6% by weight, and severing the crimped, dyed continuous filaments into staple fiber lengths.

2. Process for the production of dyed staple fiber materials, which comprises dyeing a tow of continuous cellulose acetate filaments with a dyebath comprising an aqueous solution of a lower aliphatic alcohol containing a dyestuff for said cellulose acetate filaments and a salt of thiocyanic acid which is a swelling agent therefor, washing the dyed filaments directly after dyeing, applying lubricating and anti-static conditioning agents to said dyed, continuous filaments, drying the tow to a moisture content of from about 20 to 30% by weight, imparting a crimp to the dyed and conditioned filaments by mechanically deforming the filaments under pressure, drying the crimped tow to a moisture content of about 6% by weight, and severing the crimped, dyed continuous filaments into staple fiber lengths.

3. Process for the production of dyed staple fiber materials, which comprises dyeing a dry tow of continuous cellulose acetate filaments with a dyebath comprising an aqueous solution containing at least 75% by volume of ethyl alcohol, a dyestuff for said cellulose acetate filaments and from 25 to 35 grams per liter of a salt of thiocyanic acid which is a swelling agent therefor, washing the dyed filaments directly after dyeing, applying lubricating and anti-static conditioning agents to said dyed, continuous filaments, drying the tow to a moisture content of from about 20 to 30% by weight, imparting a crimp to the dyed and conditioned filaments by mechanically deforming the filaments under pressure, drying the crimped tow to a moisture content of about 6% by weight and severing the crimped, dyed continuous filaments into staple fiber lengths.

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