3,224,182

PROCESS FOR PRODUCTION OF CROSS-LINKED CELLULOSIC YARNS

Frederick R. W. Sloan, Parsonage, Manchester, England, and John Lightfoot Spencer-Smith, Doagh, Northern Ireland, assignors to Whitecroft Industrial Holdings Limited, Manchester, England

No Drawing. Filed Mar. 16, 1961, Ser. No. 96,107
Claims priority, application Great Britain, Mar. 23, 1960, 10,313/60
8 Claims. (Cl. 57—157)

This invention relates to the production of threads or yarns from regenerated cellulose filaments.

It is now common practice to chemically treat fabrics formed from cellulose yarns to improve the mechanical properties of the fabrics, notably crease resistance, or to enable lasting decorative mechanical effects to be obtained, such as embossing, schreinering, glazing or permanent pleats or for the production of drip-dry or "minimum iron" finishes.

A wide variety of chemicals are used in such treatments but they can be classified according to the reaction which occurs during the treatment.

The first type of treatment commonly used consists of applying a synthetic resin precursor which penetrates the fibres to the fabric and then curing the resin precursor to form a water insoluble resin which forms a continuous phase extending through the fibres of the fabric. Urea-formaldehyde or melamine-formaldehyde resins are used for this type of treatment.

The second type of treatment consists in applying a compound such as formaldehyde or dimethylol ethylene urea to the fabric. These compounds chemically react with the cellulose molecules and cross-link them to one another and consequently are often referred to as cross-linking agents.

The third type of treatment consists in applying a polymeric material which forms a water insoluble continuous phase on the fabric together with a cross-linking agent. Unlike the first type of treatment however, the continuous phase does not extend into the fibres of the fabric because the polymeric materials used in the treatment are too large to penetrate them. Such polymeric materials may be referred to as extra-fibre polymers.

The polymeric materials used in this type of treatment may be initially water soluble and are converted during treatment into a water insoluble state. For example hydroxyl group containing polymers such as polyvinyl alcohol or modified cellulose derivative react with the cross-linking agent and become insoluble. These polymers will also be cross-linked to the cellulose molecules of the fabric. Thermosetting resins such as unsaturated polyester resins, or acrylic resins may also be used. Dispersions of water insoluble polymers such as vinyl acetate, vinyl stearate copolymers may also be applied.

When such treatments are applied to the fabric the final resin produced or the chemical applied tends to have a non-uniform distribution in the fibres and this is also observed if the treatment is applied to yarns or threads instead of to fabrics. The alternative of applying such treatments to fibres or filament tow prior to processing into threads or yarns has been proposed, but such proposals have not been adopted in practice, because fibres or tows which have been treated with the reagents described above and dried or cured, are exceedingly difficult to process into threads or yarns, because the fibres or filaments tend to become welded together into groups.

According to the present invention, threads or yarns are produced by impregnating a continuous cellulose filament tow with a solution containing at least one reagent which undergoes a reaction with the cellulose to modify the elastic properties of the component filaments of the tow, such as a synthetic resin precursor or chemical cross-linking agent or an extra-fibre polymer and chemical cross-linking agent, drying the tow whilst maintaining the filaments in continuous relative movement, which prevents continuing contact between given points on adjacent fibres, and thereafter converting the tow into thread or yarn by conventional methods. The heat applied during the drying of the tow may be sufficient to cure the resin, i.e., to convert the resin precursors into the final resin, or to bring the cross-linking about or convert the extra-fibre polymer to an insoluble condition but if it is not sufficient, additional heating may be applied during the drying stage or the necessary heat for curing may be applied subsequently, e.g., after the thread or yarn has been produced or even after the thread or yarn has been converted into fabric or other finished articles. It is especially desirable to cure the resin or bring about cross-linking after the fabric has been woven when it is desired to apply mechanical effects such as embossing and permanent pleating to the finished fabric.

The tendency of the fibres to become welded together is further reduced by drying the tow at a temperature which is insufficient to cure the resin precursor completely, then passing the dried or partly dried tow through a known crimping device, such as a pair of rollers delivering the tow into a stuffing box, before curing the resin precursor. The crimp so introduced may be partly or wholly removed by applying tension to the tow during curing or it may be retained by curing the tow in its crimped form. Crimping the tow before curing is specially advantageous when treating filaments of fine denier, and if the crimping is not removed, increased resiliency may be imparted to the ultimate yarns. Chemically crimped filaments may be treated with a cross-linking agent by the process of the invention. Crimped yarns are especially suited for use as carpet yarns.

The continuous relative movement during drying in addition to preventing the fibres welding into groups, also ensures a uniform distribution of the reagent. It may be imparted to the filaments in a variety of ways. For treatment with a resin precursor or extra fibre polymer and a cross-linking agent the maximum possible relative movement should be given. A particularly suitable method in these cases is to use a hot flue drier in which the tow is passed over rollers. The tow can also be dried by a hot air current such as in a loop drying machine. This method is particularly suitable for treatment of crimped fibres, where a longitudinal tension on the tow is undesirable.

If desired the tow may be treated after impregnation and before drying with a surface active material which will produce water repellency such as modified melamine stearamide.

The conversion of the dried and cured tow into yarn may be carried out by conventional steps including stapling and converting the filament tow into a sliver then converting the sliver into a twisted or untwisted roving, spinning the yarn and then doubling the yarn if necessary. Such steps may be carried out on conventional flax, cotton or worsted type machinery, the staple length being conventional for the type of machinery chosen. Such steps may also include adjustment of the moisture content and the use of any conventional processing aids. Alternatively fibre may be treated in the form of a continuous filament rove and then spun into staple fibre yarn on a direct spinner. The filaments may also be twisted and wound to form a continuous filament yarn. The filaments may of course be blended with other fibres or filaments to form a blended yarn.

The yarns so produced may be converted into knitted or woven fabrics and such fabrics exhibit improved properties particularly of abrasion resistance and strength when compared with fabrics treated with similar reagents after fabrication. The yarns so produced are also particularly suitable as tuft or pile yarns for the manufacture of carpets, because of the high resiliency and abrasion resistance which they exhibit either in the manufacture of carpets or of fabrics. The yarns produced in accordance with the invention may of course be employed in association with other yarns not produced in accordance with the invention.

The fibres may be dyed before or after conversion into yarn or after conversion into fabrics, and when used in the production of fabrics the fabrics may also be treated with the same or similar resin precursors or cross-linking agents.

The following examples illustrate the invention.

Example 1

This example illustrates the use of a synthetic resin precursor.

(A) Urea and formaldehyde are reacted together at pH 7 and a molecular ratio of about 1:1.6. When the reaction has proceeded to the extent that 3% or less of free formaldehyde is present in the reaction mixture the solution is stabilised by dilution to 75% volume. It then contains approximately 42% solids.

(B) A linear ester condensation product is prepared by reacting polyethylene glycol (molecular weight 400), with maleic anhydride in equimolecular amounts by the method described in British specification No. 790,837.

Mix A is further diluted with water to 6% solids and 5% w./v. of B is added to the diluted A mix together with sufficient ammonium dihydrogen phosphate catalyst to give a pH of about 3.2 on the fibre after curing. A length of tow composed of 18 denier filament is padded through the solution (mangle expression 80%) dried on the hot flue and cured for three minutes at 140° C. on a conventional baking stove. The tow is then stapled and processed into yarn by any of the methods previously described. When woven into carpet the resultant pile has much improved resiliency.

Example 2

This example illustrates the use of an initially water insoluble extra-fibre polymer and a cross-linking agent.

A length of rayon tow composed of 3 denier fibre was impregnated with the following solution:

15% w./v. dimethylol ethylene urea (50% solids)
1% polyvinyl acetate polyvinyl stearate co-polymer dispersion (Vinyl Products Limited)
2% polyester catalyst
0.5% modified melamine stearamide (BIP Chemicals Limited)

The polyester catalyst was a triethylene glycol citric acid supplied by British Paints Ltd. under the name "Wresinol 11542."

The material was squeezed, dried, crimped and cured, and processed into yarn by any of the methods previously indicated.

The yarn subsequently woven into cloth was treated as follows:

After singeing and desizing the material was dyed with a dye-stuff obtainable under the name "Procion Brilliant Blue R," the material was then impregnated with the following solution:

1% w./v. polyvinyl alcohol (98% hydrolysed)
5% cyclic ethylene urea
2% polyester catalyst (Wresinol 11542)

The material was dried and cured for two minutes at 140° C. The resultant cloth had a very good dry crease resistance comparable to normal treated cloth but had much improved abrasion resistance and stability against shrinkage, moreover it had a very soft distinctive handle not obtainable by normal methods.

Example 3

This example illustrates the use of a cross-linking agent alone.

A length of rayon tow composed of 18 denier filament was impregnated with the following solution:

20% dimethylol ethylene urea (50% solids)
1% catalyst H7 (Rohm & Haas buffered zinc nitrate)

The material is squeezed, dried on a hot flue drier and cured for 2 minutes at 150° C. by a second passage through the hot flue drier. The tow is then processed into yarn by any of the methods already described. The resultant yarn is used for carpet manufacture and is resilient, has a very soft handle and good abrasion resistance.

Example 4

This example illustrates the use of a thermosetting extra fibre polymer.

A length of tow composed of 4½ denier fibre is impregnated with the following solutions:

20% dimethylol ethylene urea
2% of a thermosetting acrylic resin dispersion (Primal HA–8, Rohm & Haas)
0.5% modified melamine stearamide
3% polyester catalyst

The material is squeezed, dried, crimped and cured in the normal way and converted into yarn.

When woven into cloth the resultant material has improved stability and abrasion resistance over cloth treated in the conventional way, and has about the same level of crease recovery. Again a characteristic handle is obtained.

Example 5

This example illustrates the use of a cross linking agent on chemically crimped filaments.

A length of rayon tow composed of 18 denier chemically crimped filament is impregnated with the following solution:

15% w./v. cyclic ethylene urea
2% w./v. polyester catalyst (Wresinol 11542)

The material is impregnated, squeezed, and piled on to a small jay box where it is allowed to delay for about 3 minutes (this allows the crimp to fully develop). The material is then dried on the loop drying machine or any other similar type of machine where lengthwise tension is not applied, and then cured by passage through the same type of machine at 150° C. for a duration of about 2 minutes. The treated tow is then processed into yarn by any of the methods previously described.

Example 6

This example illustrates the use of an initially water soluble extra fibre polymer.

A length of rayon tow composed of 18 denier filament is impregnated with the following solution:

15% w./v. cyclic ethylene urea
0.5% w./v. polyvinyl alcohol (Alcotex 99120)
2% w./v. polyester catalyst (Wresinol 11542)

The material is squeezed and dried on the hot flue dryer and cured for 2 minutes at 150° C. by a second passage through the same type of machine. The tow is then processed into yarn by any of the methods described and the resultant yarn used for carpet manufacture.

We claim:

1. A process for the production of cellulosic yarns, which comprises the steps of impregnating a continuous cellulose filament tow with a solution containing a cross-linking agent, drying the tow while maintaining the fila-

ments in continuous relative movement and converting the tow into yarn.

2. A process as in claim 1 wherein said solution contains both a cross-linking agent and an additional reagent which forms a synthetic resinous compound with a portion of the cross-linking agent.

3. A process as in claim 1 wherein said solution contains a cross-linking agent for the cellulose and a water soluble polymer which reacts with the cross-linking agent to form a water insoluble continuous phase.

4. A process for the production of cellulosic yarns which comprises the steps of impregnating a continuous cellulose filament tow with a solution containing a cross-linking agent selected from the group consisting of formaldehyde and dimethylol ethylene urea, drying the tow while maintaining the filaments in continuous relative movement and converting the tow into yarn.

5. A process for the production of cellulosic yarns which comprises the steps of impregnating a continuous cellulose filament tow with a solution containing both a cross-linking agent selected from the group consisting of formaldehyde and dimethylol ethylene urea and an additional reagent selected from the group consisting of urea and melamine, drying the tow while maintaining the filaments in continuous relative movement and converting the tow into yarn.

6. A process for the production of cellulosic yarns which comprises the steps of impregnating a continuous cellulose filament tow with a solution containing both a cross-linking agent selected from the group consisting of formaldehyde and dimethylol ethylene urea and water soluble polyvinyl alcohol, drying the tow while maintaining the filaments in continuous relative movement and converting the tow into yarn.

7. A process for the production of cellulosic yarns which comprises the steps of impregnating a continuous cellulose filament tow with a solution containing a cross-linking agent, drying the tow while maintaining the filaments in continuous relative movement by passing the tow through a hot flue drier provided with a series of rollers over which the tow passes, and converting the tow into yarn.

8. A process for the production of cellulosic yarns which comprises the steps of impregnating a continuous filament tow with a solution containing a cross-linking agent, drying the tow while maintaining the filaments in continuous relative movement by passing the tow through a loop drier, and converting the tow into yarn.

References Cited by the Examiner

UNITED STATES PATENTS

2,514,187	7/1950	Bosomworth.	
2,892,219	6/1955	Thiele	19—66
2,877,739	3/1959	Payne	19—66
2,972,220	2/1961	Flack et al.	19—66
2,978,788	4/1961	Keefe	28—75
3,039,167	6/1962	Lund	28—76

MERVIN STEIN, *Primary Examiner.*

DONALD W. PARKER, *Examiner.*