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TREATMENT OF CELLULOSIC FIBERS

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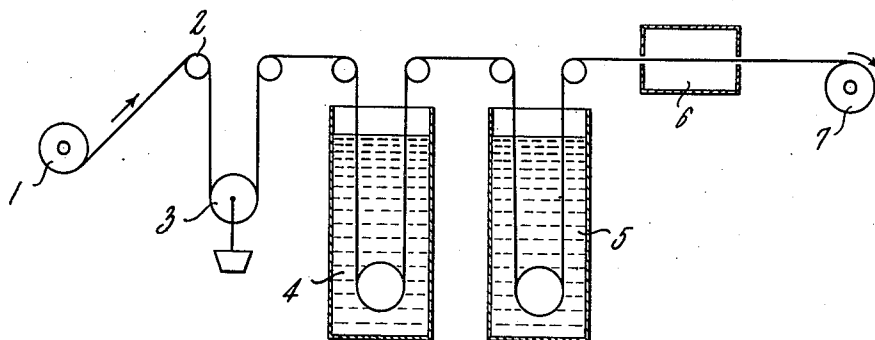


FIG. 1  
CONTINUOUS METHOD

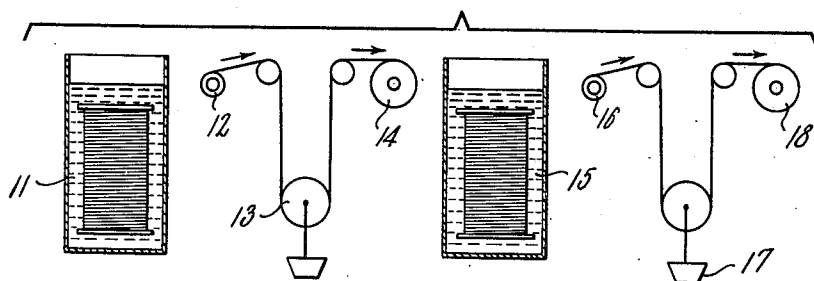


FIG. 2  
BATCH METHOD

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## UNITED STATES PATENT OFFICE

2,549,014

## TREATMENT OF CELLULOSIC FIBERS

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2 Claims. (Cl. 28-76)

1

This invention relates to improved cotton yarns and cords, and to a method of improving their tensile strength.

The invention resides in the discovery that cotton yarns and cords, and especially yarns and cords of grey cotton, may be endowed with markedly greater tensile strength per unit weight (measured at ordinary temperatures) when the yarn or cord is subjected to a combination of two treatments, namely: (a) treatment with an aqueous bath, preferably a slightly alkaline aqueous solution from the class consisting of dilute aqueous solutions of fixed alkalis, aqueous solutions of ammonia, and aqueous solutions of readily water-soluble amines; and (b) treatment with a hot non-aqueous bath consisting essentially of an organic solvent, preferably selected from those solvents having boiling points which will permit liquid treatment at 50-150° C. at atmospheric pressure, and at the same time will permit solvent volatilization during subsequent drying. The best results are obtained when tension is applied to the yarn or cord during part or all of the treatment in each bath, and during the subsequent drying. It has been found that the effects resulting from the combination of the two treatments are much greater than the sum of the individual effects of the two treatments upon separate yarns or cords.

Grey cotton refers to cotton fibers, yarn, cord, or thread containing substantially all the natural cotton waxes, and "cotton waxes" means that group of non-cellulosic water-insoluble components of the grey cotton which are characterized by extreme solubility in hot ethyl alcohol.

The aqueous bath (a) has the property of swelling the cellulose without materially altering the chemical composition of the cellulose fibers constituting the yarn or cord; the non-aqueous organic solvent bath (b) is capable of swelling or dissolving the so-called cotton waxes. Either bath may be used first in the successive treatments. The time of treatment in each bath should be for at least five minutes but not to an extent to substantially alter the final softness and pliability of the yarn or cord. The tension applied to the cord or yarn during the treatment should preferably not exceed 40% of the breaking stress of the untreated cord or yarn if the continuous method is used; in batch treatment on spools, higher tension may be required. The treated yarn or cord is subsequently dried under tension.

Suitable fixed alkalis for making the aqueous bath slightly alkaline are sodium hydroxide, so-

2

dium carbonate, trisodium phosphate. Concentrations of from 0.1% to 2% are useful, higher concentrations giving no added benefit and being disadvantageous because they may impair the tensile strength. Such tendering of the cords is avoided by using instead a volatile base which is completely removed in the subsequent drying of the cord.

The time of treatment with the aqueous bath may range up to about 2 hours when the aqueous solution is used heated, and much longer when the temperature of the solution approximates room temperature. The duration generally should be such as not to degrade, that is, reduce the strength of, the fibers.

The time of treatment with the non-aqueous organic solvent ranges from about 5 minutes to about 2 hours, there being no advantage obtained by more prolonged treatment. It is best that at least a substantial proportion of the naturally occurring cotton waxes remain in the yarn or cord after the treatment. In practice, the cord after the solvent treatment usually contains upwards of 40% of the ethanol-extractable material initially present.

The organic solvent of (b) may be any well-known resin solvent or gum solvent, or mixture of such, preferably (but not necessarily) boiling in the range from about 100° C. to about 180° C., and being capable of dissolving cotton waxes. Very good results are obtained with alkyl acetates in which the alkyl group comprises from 3 to 5 carbon atoms, for example, amyl acetate, or with toluene, trichloroethylene, pyridine, or butanol. Other solvents such as xylene, solvent naphtha, petroleum ether, amyl alcohol, cyclohexanone, diacetone alcohol, diethyl ketone, butyl ethyl ketone, propyl acetate, butyl acetate, isobutyl acetate, butyl lactate, ethyl butyrate, "methyl Cello-solve," diethyl carbonate, ethylene chlorohydrin, amyl ethyl ether, dichlorodiethyl ether, isoamyl ether, morpholine, piperidine, and dioxane may also be used.

The two treatments (a) and (b) may be combined successively in a continuous process, shown diagrammatically in Figure 1 of the drawing, whereby the yarn or cord is passed first through one bath and then through the other, and is finally dried. Alternatively, the yarn or cord may be passed through or under a spray of the solutions, in succession.

Alternatively, the two treatments may be applied in a batch process, shown diagrammatically by Figure 2 of the drawing, in which yarn or cord

wound in packages is treated first in one bath; the yarn or cord is then subjected to tension (as by re-winding under tension), and is then treated by the second bath. Thereafter, the yarn or cord is again subjected to tension (as by re-winding under tension), and is dried under tension. In this batch process higher tensions (up to 75% of the breaking stress of the untreated material) may be applied, since the tension at fixed elongation rapidly decreases immediately after winding. By suitable adjustments of the factors of time and tension, products may be made by the batch process that are identical with those made by the continuous method described above.

The yarn or cord may be so treated as to have either high tensile strength and low stretch, or a less increased tensile strength and a higher stretch. The yarns and cords may be used in the manufacture of sewing threads, tire cords, and many kindred products. The yarns and cords processed according to the invention may be employed as such, or combined with other processing, for the fabrication of various articles, e. g., hose, tires, belting, or other strain-resisting elements, and for the preparation of various fabrics, woven, knitted, or braided.

The invention may be applied to grey cotton in various forms, e. g., single yarn, plied yarns, cords, and cables.

The expression "stranded" cotton fibres is to be understood as meaning cord, yarn, plied yarn, strand, thread, string, or the like.

The treated yarns or cords may be subsequently rubberized, as in the manufacture of tire carcasses, but it will be apparent that the invention may be adapted to various uses not requiring rubberizing, and that the production of unrubberized fabrics including the treated cotton is also within the broad scope of the invention.

The following examples are given to illustrate the invention, without confining it thereto, the parts or concentrations being by weight. In Examples 1 to 15 the strength tests are made at 70° F. and 65% relative humidity (except as noted in Example 12). The calculation of percent increase in strength is made on the denier basis (except in Example 12).

#### Example 1.—Continuous process—Figure 1

18/4/3 cotton tire cord supplied from any conventional tube or spindle 1 is passed first through a tensioning device consisting of a snubbing bar 2 and a dead load pulley 3. Weight attached to the pulley is sufficient to maintain a tension of 0.9 lb. on the cord in transit. The cord is then passed through a bath 4 consisting of amyl acetate heated to a temperature of 90° C. The cord traverses the amyl acetate bath at the rate of one foot per minute; each individual point on the cord is in contact with the amyl acetate for about 10 minutes. The cord is next passed through a bath 5 consisting of an aqueous 0.28% solution of ammonia maintained at 75° C., the length of path likewise being such that the cord is in contact with the ammonia for about 10 minutes. The cord is next passed through a chamber 6 in which dry air at 80° C. is circulated, the cord remaining in this chamber for about 2½ minutes. The cord is finally wound on a spool at the positive drive wind-up mechanism 7 which provides the power necessary to pull the cord through the equipment. The cord so treated shows substantial improvements in strength and in fatigue life, with a

corresponding reduction in stretch as illustrated by the following test data:

|                                                                                                 | Untreated<br>18/4/3<br>Cord | 18/4/3 Cord<br>Treated as<br>Described<br>in<br>Example 1 |
|-------------------------------------------------------------------------------------------------|-----------------------------|-----------------------------------------------------------|
| Strength:                                                                                       |                             |                                                           |
| in lbs.                                                                                         | 17.6                        | 23.5                                                      |
| in gms./denier                                                                                  | 2.02                        | 2.94                                                      |
| Per cent increase in strength                                                                   |                             | 46                                                        |
| Stretch:                                                                                        |                             |                                                           |
| at 10 lbs., per cent                                                                            | 5.94                        | 3.45                                                      |
| at break, per cent                                                                              | 8.97                        | 8.50                                                      |
| Fatigue life: Cycles flexing at 250° F. to<br>produce failure                                   | 2,870                       | 13,320                                                    |
| Per cent increase in fatigue life                                                               |                             | 365                                                       |
| Tension applied during treatment, as per<br>cent of breaking strength of untreated<br>grey cord |                             | 5                                                         |

#### Example 2.—Batch process—Figure 2

An 1800 yard length of 7.8/1 cotton yarn wound on a metal spool with a perforated barrel is immersed in a bath 11 consisting of an aqueous 0.14% solution of ammonia at room temperature (25° C). The package is allowed to remain in the bath for 2 hours to permit penetration. The package is then removed from the bath and placed on a positive drive variable speed let-off spindle 12. The wet yarn is then passed through the dead load tensioning pulley 13 and rewound on a second spool on a positive drive fixed speed spindle 14. During the respooling operation just described the speed of the let-off spindle 12 is varied by manual control so as to maintain the tensioning pulley 13 at a uniform height. The weight of the load and pulley 13 is such that a tension of about 1.9 lbs. is maintained on the yarn in transit between let-off 12 and wind-up 14. The average rate of wind-up is about 150 yards per minute.

The respooled yarn is then immersed in a bath 15 of amyl acetate for 4 hours at 110° C., after which it is again respooled. The second respooling operation is comparable to the first, except that the load applied 17 is such that the yarn in transit between the let-off spindle 16 and the wind-up spindle 18 is maintained at a tension of 1.7 lbs.

The rewinding operation is a convenient way of applying more tension to the strand after immersion in bath 11, but other ways may be used.

After the final respooling the yarn is dried on the spool at 70° F. and 65% relative humidity. This is a convenient procedure for small packages; for larger packages the yarn may be dried by passing hot, dry air through the packages.

Yarn treated by the method of Example 2 shows a substantial increase in strength and a corresponding reduction in stretch, as illustrated by the following test data:

|                                                                                                 | 8/1 Yarn<br>Un-<br>treated | 8/1 Yarn<br>Treated as<br>Described<br>in Ex-<br>ample 2 |
|-------------------------------------------------------------------------------------------------|----------------------------|----------------------------------------------------------|
| Strength:                                                                                       |                            |                                                          |
| in lbs.                                                                                         | 2.87                       | 4.80                                                     |
| in gms./denier                                                                                  | 1.81                       | 3.24                                                     |
| Per cent increase in strength                                                                   |                            | 79                                                       |
| Stretch at break, per cent                                                                      | 6.01                       | 2.44                                                     |
| Tension applied during treatment, as per<br>cent of breaking strength of untreated grey<br>cord |                            | 60-66                                                    |

If desired, either or both of the respooling operations at 14 and 18 may be eliminated, although better results follow such respooling.

5

In a modification of the static or batch process, the cord may be kept in fixed position and the respective solutions either sprayed or circulated thereon in succession.

### Example 3

7.5/1 cotton yarns are treated, as described in Example 2, except that:

(a) Variations are made in the aqueous bath to show that while water may be used for the aqueous bath, further improvements are gained by use of an aqueous bath made slightly alkaline by the addition of small amounts of bases, e. g., ammonia, ethylene diamine, or caustic soda;

(b) The tension maintained is 2.0 lbs., or about 69% of the strength of the untreated yarns.

|   | Baths                                          | pH   | Strength |           |             | Stretch At Break |
|---|------------------------------------------------|------|----------|-----------|-------------|------------------|
|   |                                                |      | Lbs.     | Gms./Den. | Improvement |                  |
| A | Untreated yarn                                 |      | 2.90     | 1.83      | Per cent    | Per cent         |
| B | Amyl acetate treatment only.                   |      | 3.21     | 2.19      | 20          | 4.6              |
|   | Amyl acetate treatment following treatment by: |      |          |           |             |                  |
| C | Water                                          | 7.0  | 3.80     | 2.52      | 38          | 1.6              |
| D | 0.14% Ammonia                                  | 9.0  | 4.59     | 3.13      | 71          | 2.2              |
| E | 1% Ethylene diamine                            | 11.2 | 4.31     | 2.90      | 59          | 1.9              |
| F | 1% Caustic soda                                | 11.5 | 4.91     | 3.37      | 84          | 2.2              |

### Example 4

7.5/1 cotton yarn is treated as described in Example 3, except that various non-aqueous baths are used, as noted below, at 90° C., to show that a variety of commercial organic solvents may be used for the non-aqueous bath.

|   | Baths                                   | Strength |           |             | Stretch at Break |
|---|-----------------------------------------|----------|-----------|-------------|------------------|
|   |                                         | Lbs.     | Gms./den. | Improvement |                  |
| A | Untreated grey yarn                     | 2.90     | 1.83      | Per cent    | Per cent         |
| B | Aqueous 0.14% Ammonia bath only.        | 3.56     | 2.46      | 34          | 2.3              |
|   | Aqueous 0.14% Ammonia bath followed by: |          |           |             |                  |
| C | Toluene                                 | 3.94     | 2.76      | 51          | 1.9              |
| D | Tetrachloroethylene                     | 4.04     | 2.86      | 56          | 1.9              |
| E | Amyl acetate                            | 4.59     | 3.13      | 71          | 2.2              |
| F | Pyridine                                | 4.86     | 3.17      | 73          | 2.5              |

Examples 3 and 4 together show that the results obtainable by the combination of the baths are more than additive. Adding the per cent improvement of Example 3 (B) to that of Example 4 (B), gives a predicted total of 54% increase in strength, whereas in reality Example 3 (D), and Example 4 (E), show the combination treatment to give 71% improvement in strength. Note also that this increase did not require more than a tension of about 2 lbs., which is noteworthy considering that the disadvantage of most prior art treatments of cotton strands, attempting to approach similar strength, is that they require tensions so high as to closely approach the breaking strength of the strands. For example, in British Patent No. 545,716, to get a 31% strength improvement requires such a high tension as to bring about a 75% loss in stretch.

6

### Example 5

10/3/2 cotton cord is treated as described in Example 1, except that:

(a) The applied tension was 4.75 lbs. (equal to 31% of the breaking strength of the untreated cord); and

(b) The time of treatment in each bath is varied, at noted below, to show that time of treatment in either bath in excess of 5 minutes and up to 20 minutes is not critical in the continuous process.

|                             | A Untreated | B Treated | C Treated | D Treated | E Treated |
|-----------------------------|-------------|-----------|-----------|-----------|-----------|
| Time of Treatments:         |             |           |           |           |           |
| In aqueous 0.28% ammonia    |             | 5'        | 10'       | 10'       | 20'       |
| In Amyl acetate             |             | 5'        | 5'        | 10'       | 20'       |
| Strength:                   |             |           |           |           |           |
| lbs.                        | 15.4        | 18.7      | 19.5      | 19.4      | 19.2      |
| gms./den.                   | 1.82        | 2.54      | 2.61      | 2.58      | 2.57      |
| Stretch at break, per cent. | 14.0        | 5.6       | 5.3       | 5.4       | 5.4       |

### Example 6

10/3/2 cotton cord is treated as described in Example 1, except that the temperature of the non-aqueous bath is varied as noted to show that the temperature of the non-aqueous bath should be substantially above room temperatures, and preferably not lower than about 50° C.

|                                        | A Untreated | B Treated | C Treated | D Treated |
|----------------------------------------|-------------|-----------|-----------|-----------|
| Temperature of amyl acetate bath, ° C. |             | 25        | 55        | 90        |
| Strength:                              |             |           |           |           |
| lbs.                                   | 14.3        | 14.9      | 18.8      | 19.5      |
| gms./den.                              | 1.76        | 1.79      | 2.41      | 2.54      |
| Stretch at break, per cent.            | 13.1        | 9.1       | 9.1       | 7.5       |

### Example 7

10/3/2 cord is treated as described in Example 1, except for the variations in tension as noted below, to show that in the continuous method the strength is improved as the tension applied is increased up to a value equal to around 20% of the strength of the untreated material; at higher tensions the stretch is further reduced without any apparent further increase in strength.

|                                                | A Untreated | B    | C    | D    | E    | F    |
|------------------------------------------------|-------------|------|------|------|------|------|
| Tension applied during treatment, lbs.         |             | 0    | 1    | 3    | 5    | 8    |
| Strength:                                      |             |      |      |      |      |      |
| lbs.                                           | 15.2        | 17.1 | 18.3 | 19.2 | 19.5 | 19.2 |
| gms./den.                                      | 1.79        | 2.00 | 2.36 | 2.54 | 2.62 | 2.56 |
| Stretch at break, per cent.                    | 13.6        | 17.8 | 8.0  | 5.9  | 5.0  | 4.2  |
| Tension as per cent of untreated grey strength |             | 0    | 7    | 20   | 33   | 53   |

### Example 8

7.1/1 cotton yarn is treated as described in Example 2, except that the tension applied is varied as noted below to show that the strength of the yarns treated according to the batch method improves as the tension applied is increased up to about 60% of the breaking strength of the untreated yarn. At this point it appears that higher applied tension would result in a substantial reduction in stretch without a compensating increase in strength.

7

|                                                                       | A Un-<br>treat-<br>ed | B       | C    | D    | E    | F    |
|-----------------------------------------------------------------------|-----------------------|---------|------|------|------|------|
|                                                                       |                       | Treated |      |      |      |      |
| Tension applied during treatment, lbs.....                            |                       | 0       | ¾    | ¾    | 1½   | 2    |
| Strength:                                                             |                       |         |      |      |      |      |
| lbs.....                                                              | 2.6                   | 3.4     | 3.8  | 4.3  | 4.6  | 4.7  |
| gms./den.....                                                         | 1.67                  | 2.08    | 2.42 | 2.79 | 3.12 | 3.27 |
| Stretch at break, per cent.....                                       | 5.4                   | 5.4     | 4.6  | 4.3  | 3.4  | 2.4  |
| Tension initially applied as per cent of untreated grey strength..... |                       | 0       | 15   | 25   | 58   | 77   |

*Example 9*

10/3/2 cotton cords are treated as described in Example 1, except that:

(a) A tension of 4.2 lbs. is applied, equivalent to 27% of the breaking strength of the untreated cord; and

(b) The various cords are treated by a single bath or by both baths as indicated.

This example shows further that the improvement gained by the use of the aqueous and non-aqueous baths in succession is substantially greater than the improvement gained by treatment with either bath alone. The effect of the invention as disclosed is likewise greater than would be predicted from the separate effects of the aqueous and non-aqueous treatments.

8

and cords treated by the continuous procedure (as in Example 1).

|                                        | Un-<br>treated | Treated By—                |                  |
|----------------------------------------|----------------|----------------------------|------------------|
|                                        |                | Con-<br>tinuous<br>Process | Batch<br>Process |
| 10/3/2 COTTON CORD                     |                |                            |                  |
| Tension applied:                       |                |                            |                  |
| lbs.....                               |                | 4.5                        | 10.0             |
| as per cent of untreated strength..... |                | 30                         | 66               |
| Strength:                              |                |                            |                  |
| lbs.....                               | 15.2           | 19.7                       | 19.7             |
| gms./den.....                          | 1.79           | 2.65                       | 2.62             |
| Stretch at break, per cent.....        | 13.6           | 4.6                        | 4.4              |
| 7.1/1 COTTON YARN                      |                |                            |                  |
| Tension applied:                       |                |                            |                  |
| lbs.....                               |                | 1.0                        | 2.0              |
| as per cent of untreated strength..... |                | 38                         | 77               |
| Strength:                              |                |                            |                  |
| lbs.....                               | 2.6            | 4.9                        | 4.7              |
| gms./den.....                          | 1.67           | 3.35                       | 3.27             |
| Stretch at break, per cent.....        | 5.4            | 2.5                        | 2.4              |

*Example 12*

This example shows the strength, and strength improvement, of yarns treated by my invention and tested both bone dry and at 65% humidity. The data show that the treated yarn is less af-

|                                         | A Un-<br>treated | B Treated          | C Treated         | D Treated          |
|-----------------------------------------|------------------|--------------------|-------------------|--------------------|
| Aqueous bath.....                       |                  | 0.28% Ammonia..... |                   | 0.28% Ammonia..... |
| Non-aqueous bath.....                   |                  |                    | Amyl acetate..... | Amyl acetate.....  |
| Strength:                               |                  |                    |                   |                    |
| lbs.....                                | 15.7             | 16.6               | 17.4              | 20.4               |
| gms./den.....                           | 1.87             | 2.16               | 2.13              | 2.64               |
| Stretch at break, per cent.....         | 14.0             | 6.0                | 10.8              | 5.9                |
| Per cent increase in grey strength..... |                  | 15                 | 14                | 41                 |

*Example 10*

18/4/3 cotton cord is treated as described in Example 1, except that the tension applied is 2.2 lbs., corresponding to 12% of the breaking strength of the untreated cord, to show that the order in which the cord is treated in the two baths is immaterial.

|                                 | A Un-<br>treated | B Treated          | C Treated          |
|---------------------------------|------------------|--------------------|--------------------|
| 1st Treatment.....              |                  | 0.28% Ammonia..... | Amyl acetate.....  |
| 2nd Treatment.....              |                  | Amyl acetate.....  | 0.28% Ammonia..... |
| Tension used.....               |                  | 2.2 lbs.....       | 2.2 lbs.....       |
| Strength:                       |                  |                    |                    |
| lbs.....                        | 18.0             | 23.5               | 23.2               |
| gms./den.....                   | 2.06             | 2.86               | 2.86               |
| Stretch at break, per cent..... | 9.8              | 6.3                | 6.4                |

*Example 11*

This example shows that by suitable adjustment of the applied tension, yarns and cords treated by the batch procedure (as in Example 2) may be made equal in quality to comparable yarns

50 fected than the untreated yarn by lowering the moisture content.

7.8/1 yarn is treated exactly as described in Example 2.

|                              | Stretch at<br>Break | Strength | Increase in<br>strength |
|------------------------------|---------------------|----------|-------------------------|
| Tested at 70° F., 65% R. H.: | Per cent            | Pounds   | Per cent                |
| untreated yarn.....          | 6.1                 | 2.66     |                         |
| treated yarn.....            | 2.6                 | 4.93     | 85                      |
| Tested at 70° F., 0% R. H.:  |                     |          |                         |
| untreated yarn.....          | 3.1                 | 1.37     |                         |
| treated yarn.....            | 2.2                 | 3.90     | 185                     |

*Example 13*

This example shows the relative effects of processing, according to this invention, cotton yarn composed of grey fibers (containing the native cotton waxes), and cotton yarn from which the non-cellulosic constituents have first been extracted with ethanol. Note that even in the case of the extracted cotton there is an increase in tensile strength, but materially less than when grey cotton is treated.

[7.8/1 cotton yarns.]

|                                                        | Not extracted |                  | Extracted with ethanol, 72 hrs. |                  |
|--------------------------------------------------------|---------------|------------------|---------------------------------|------------------|
|                                                        | A             | B                | C                               | D                |
| Per cent Extractable Material in Yarn before treatment | 1.75          | 1.75             | 0.25                            | 0.25             |
| Treatment                                              | None          | ( <sup>1</sup> ) | None                            | ( <sup>1</sup> ) |
| Per cent Extractable Material in yarn after treatment  | 1.75          | 0.90             | 0.25                            | 0.10             |
| Strength:                                              |               |                  |                                 |                  |
| in lbs.                                                | 2.80          | 4.82             | 3.44                            | 4.40             |
| in gms./den.                                           | 1.84          | 3.31             | 2.37                            | 3.09             |
| Per cent increase in strength                          | 0             | 80               | 29                              | 68               |
| Stretch at break, per cent                             | 5.4           | 2.7              | 5.4                             | 2.3              |

<sup>1</sup> As in Ex. 2.**Example 14**

This example further illustrates that the effects obtained are more than the sum of the effects from treating with water alone and from treating with the organic solvent alone. In this example 10/3/2 cotton cords are treated, respectively, with water alone, with amyl acetate alone, and with amyl acetate followed by water; the tension applied during the bath treatments is about 27% of the breaking strength of the untreated cord.

|                              | Tension (lbs.) | Strength |       | Per Cent Stretch (at Break) | Per Cent Increase in Strength |
|------------------------------|----------------|----------|-------|-----------------------------|-------------------------------|
|                              |                | lbs.     | g./d. |                             |                               |
| Untreated unwetted grey cord |                | 15.7     | 1.87  | 14.0                        |                               |
| Amyl acetate alone           | 4.2            | 17.4     | 2.13  | 10.8                        | 14                            |
| Water alone                  | 4.2            | 16.6     | 2.17  | 5.7                         | 16                            |
| Amyl acetate and then water  | 4.2            | 19.0     | 2.58  | 4.8                         | 38                            |

**Example 15**

12.4/1 cotton yarns are treated as described in Example 2, except that:

(a) Variations are made in the aqueous swelling bath to show that, while dilute aqueous ammonia may be used, further improvements in strength and stretch may be gained by use of a more concentrated aqueous solution of ammonia;

(b) The tension applied is 1.0 lb., or about 67% of the breaking strength of the untreated yarn:

|                                                    | 12.4/1 Yarn Untreated |      | 12.4/1 Yarn Treated as described in Example 15 |      |
|----------------------------------------------------|-----------------------|------|------------------------------------------------|------|
|                                                    | A                     | B    | C                                              |      |
| Concentration of ammonia in swelling bath per cent |                       |      |                                                |      |
| Strength:                                          |                       |      |                                                |      |
| in lbs.                                            |                       | 1.57 | 2.63                                           | 2.85 |
| in gms./den.                                       |                       | 1.69 | 2.93                                           | 3.17 |
| Stretch at break, per cent                         |                       | 3.55 | 2.45                                           | 3.00 |
| Per cent increase in grey strength                 |                       |      | 73                                             | 88   |
| Per cent reduction in grey stretch                 |                       |      | 31                                             | 16   |

It will be apparent that by this invention it is possible to treat cotton yarns and cords inexpensively, by a simple two-step chemical process

to provide cotton textile materials with greatly increased strength, and without the use of excessively high tension or prolonged washing.

Having thus described my invention, what I claim and desire to protect by Letters Patent is:

1. A method of improving the tensile strength of stranded grey cotton fibers which comprises subjecting them to successive contact with two liquids, one being a dilute aqueous ammonia solution, and the other being an organic liquid having the property of dissolving cotton waxes comprising an alkyl acetate the alkyl group having from 3 to 5 carbon atoms, the solvent treatment being carried out at a temperature substantially above 25° C., but not in excess of 150° C., the entire liquid treatment allowing maintenance in the fibers of a substantial proportion of the original cotton waxes, and drying the fibers, and maintaining the stranded fibers under a marked tension materially less than the breaking strength of the untreated stranded fibers during at least part of the liquid treatment.

2. A method of improving the tensile strength of stranded grey cotton fibers which comprises subjecting them to successive contact with two liquids, one being a dilute aqueous ammonia solution, and the other being an organic liquid having the property of dissolving cotton waxes comprising amyl acetate, the solvent treatment being carried out at a temperature substantially above 25° C., but not in excess of 150° C., the entire liquid treatment allowing maintenance in the fibers of a substantial proportion of the original cotton waxes, and drying the fibers, and maintaining the stranded fibers under a marked tension materially less than the breaking strength of the untreated stranded fibers during at least part of the liquid treatment.

HENDRIK ROMEYN, JR.

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