

## UNITED STATES PATENT OFFICE

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## PROCESS OF IMPROVING COTTON

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Processes are known for treating vegetable fibrous materials, such as cotton fabric and the like, with alkali and carbon bisulphide, partly in order to convert these materials into a parchment-like product, partly to give them a durable finish. These processes consist in impregnating the fibrous materials, particularly fabrics, with strong alkali solution and then subjecting them to the action of carbon bisulphide in vapour form, or in first treating the material with concentrated alkali solution, then with carbon bisulphide and finally mercerizing under tension.

According to my invention very valuable properties are imparted to cotton in hank or fabric form when it is treated with an alkali solution and carbon bisulphide in such a manner that in the course of this treatment there is applied continuously or temporarily a temperature which does not substantially exceed  $+5^{\circ}$  C., for example a temperature between  $0^{\circ}$  and  $-25^{\circ}$  C. or lower.

According to the nature of the cotton material and the mode of operation, particularly the duration of the action of the carbon bisulphide in presence of the alkali solution there are obtained by the present process either highly translucent effects with a very silky lustre or a more or less stiffening finish or both.

Two operations are essential to the process: (1) the treatment of the cotton with carbon bisulphide in presence of an alkali solution and (2) the action of a low temperature in presence of an alkali solution.

These two operations may be carried out separately or simultaneously.

The cotton may be subjected to the treatment in the form of fabrics, or of yarns in hanks or cops or warps.

The cotton or the material consisting of or containing cotton may be treated by the present process in raw condition or after preliminary treatment (for example boiled, wetted or not wetted, unbleached or bleached with oxidizing or reducing bleaching agents, mercerized or not mercerized). It may also previously be treated with a hydrolyzing or gelatinizing agent, for example a strong mineral acid, such as sulphuric acid of  $49-60^{\circ}$

Baumé specific gravity or phosphoric acid of  $55-57^{\circ}$  Baumé or higher specific gravity or hydrochloric acid of  $24^{\circ}$  Baumé specific gravity or nitric acid of  $43-46^{\circ}$  Baumé or higher specific gravity, or hot solution of zinc chloride of  $60^{\circ}$  Baumé specific gravity or an ammoniacal solution of cupric oxide of high concentration.

The process may be applied in diverse ways. As examples of the conduct thereof—which, however, the invention is not intended to be confined—the following varieties are adduced:

(1) The cotton material is treated with carbon bisulphide, which, if desired, may be diluted with a suitable diluent, for instance benzene, petrol, chloroform, petroleum ether, carbon tetrachloride or the like. The cotton material impregnated with carbon bisulphide is then treated with an alkali solution, for example, an alkali solution of normal temperature or of a temperature higher than this, for example  $+30^{\circ}$  to  $+50^{\circ}$  C., and, finally, the alkali solution is brought to a temperature which does not substantially exceed  $+5^{\circ}$  C., for example to a temperature between  $0^{\circ}$  and  $-25^{\circ}$  C. or lower. It is also possible to proceed so that the material treated with carbon bisulphide and alkali solution at room temperature or higher, for example at  $+30^{\circ}$  to  $50^{\circ}$  C., is introduced into an alkali solution previously cooled to a temperature not substantially exceeding  $+5^{\circ}$  C., for example to  $0^{\circ}$  to  $-25^{\circ}$  C. or lower.

(2) The cotton material, previously treated with carbon bisulphide as in (1), is introduced into an alkali solution which, from the very beginning, has a temperature not substantially exceeding  $+5^{\circ}$  C., for example  $0^{\circ}$  to  $-25^{\circ}$  C. or lower.

(3) The cotton material is immersed in an alkali solution which, at the outset or after introduction of the cotton, receives an addition of carbon bisulphide; the cotton is treated with this mixture for some time at room temperature or at a higher temperature, for example at  $+30^{\circ}$  to  $+50^{\circ}$  C. and the mass is then cooled to a temperature not substantially exceeding  $+5^{\circ}$  C., for example to  $0^{\circ}$  to  $-25^{\circ}$  C. or lower.

(4) The cotton material is treated with an alkali solution having a temperature not substantially exceeding  $+5^{\circ}\text{C}$ ., for example  $0^{\circ}$  to  $-25^{\circ}\text{C}$ . or lower, to which alkali solution, before or after it comes in contact with the cotton material, carbon bisulphide is added.

(5) The cotton is impregnated with an alkali solution and, where desirable, after removing the excess of solution (for example by squeezing or centrifuging), is subjected to the action of carbon bisulphide in liquid or vapour form and, finally, treated with an alkali solution at a temperature not substantially exceeding  $+5^{\circ}\text{C}$ ., for example at  $0^{\circ}$  to  $-25^{\circ}\text{C}$ . or lower.

(6) The cotton is impregnated with an alkali solution and, where desirable, after removing the excess of solution (for example by squeezing or centrifuging), subjected to the action of a mixture of an alkali solution and carbon bisulphide at a temperature not substantially exceeding  $+5^{\circ}\text{C}$ ., for example at  $0^{\circ}$  to  $-25^{\circ}\text{C}$ . or lower. The cotton material treated according to one of the foregoing illustrative examples of carrying out the present process is, of course, washed or acidified and washed and dried in the usual manner. It may also be dried or steamed before washing.

All the subsidiary operations usual in mercerizing or proposed for that purpose may also be applied to the present process, for example, glazing, printing, mechanical beating and the like.

By reserving desired portions by any known method (for example, by printing substances adapted for the purpose, such as albumin or acid), pattern effects may be obtained according to the present process.

Mixed goods, that is to say goods composed of vegetable and animal fibres, may also be subjected to the present process.

By suitable selection of the conditions of operation there may be obtained silky effects far surpassing those obtained by the usual mercerizing process. At the same time, provided that the duration of the treatment with carbon bisulphide in presence of alkali solution is not too long, there occurs no, or only negligible, stiffening of the cotton goods, which, if it appears at all, may be eliminated easily, if desired, by a suitable subsequent mechanical treatment.

For obtaining the silky lustre, stretching of the goods is essential. It is immaterial whether the tension is applied before the cotton material is subjected to the treatment with carbon bisulphide in presence of alkali solution or after this treatment has been completed.

If the operation is directed to production of a finish, there is obtained, according to the duration of the action of the carbon bisulphide in presence of alkali solution a light,

medium or stiff finish, which on continued treatment acquires the fullness and stiffness of book-cloth. This finish may be enhanced by adding to the alkali, before or during the action of the carbon bisulphide a small proportion of cellulose, for example sulphite-cellulose of mercerized cellulose or a cellulose conversion product, for example cellulose hydrate. The cellulose body swollen or suspended in the lye passes into solution under the action of carbon bisulphide in the cold and enriches very considerably the finish.

The following examples illustrate the invention:—

(1) Unbleached or bleached cotton fabric is treated in the stretched state for several seconds up to 15 minutes with carbon bisulphide, alone or diluted with a suitable agent (for instance with an equal weight or twice its weight of benzene). Then, where desirable after squeezing out the surplus carbon bisulphide, the fabric is immersed in caustic soda solution of 5–15 per cent strength, for example of 10 per cent strength, of room temperature and treated therewith for a few seconds up to 10 minutes. The caustic soda solution, in which the fabric is immersed, is then cooled to  $-5^{\circ}$  to  $-15^{\circ}\text{C}$ . and kept at this temperature for a few seconds up to 10 minutes, after which it is washed or soured (for example with sulphuric acid of 5–10 per cent strength) and washed and dried.

It is also possible to proceed so that the fabric, after having been treated with an alkali solution at room temperature, is introduced into another, similarly concentrated alkali solution cooled from the first to  $-5^{\circ}$  to  $-15^{\circ}\text{C}$ .

By the foregoing treatment a superior silky lustre is imparted to the fabric.

(2) The mode of operation is as in Example (1), except that the material, previously treated with carbon bisulphide is introduced into a caustic soda solution of 5–15 per cent strength (for example 10 per cent) which is cooled from the first to  $0^{\circ}$  to  $-10^{\circ}\text{C}$ .

(3) The mode of operation is as in Example (1), except that the treatment of the fabric, previously treated with carbon bisulphide, with caustic soda solution at room temperature is continued for a longer period, for example  $\frac{1}{2}$ –2 hours. The result of this modification of the mode of operation is that in addition to the silky lustre the fabric acquires a slight stiffening finish.

(4) The mode of operation is as in Example (1), except that the material, previously treated with carbon bisulphide, is treated with a caustic soda solution of  $+30^{\circ}$  to  $+50^{\circ}\text{C}$ . for a few seconds up to five minutes, after which the solution is cooled as in Example (1).

(5) The mode of operation is as in Example (1), (2), (3) or (4), except that the fabric is stretched only after the carbon bi-

sulphide treatment or after the treatment with alkali at room temperature or higher.

longer period, if the sulphuric acid is cooled to 0° C. or lower.

The treatment with sulphuric acid may also be preceded by a mercerizing of the fabric, for example with caustic soda solution of 10 per cent strength at a low temperature or with a strong caustic soda solution, for example of 18-40 per cent strength at room temperature.

(10) The mode of operation is as in the preceding examples, except that the fabric is previously mercerized according to any known mercerizing process.

In the foregoing examples, if temperatures below -8° C. are used in cooling, the caustic soda solution usually crystallizes or freezes slightly or more pronouncedly, especially if it is not strong, for instance of 5-12 per cent strength. This phenomenon in no wise impairs the result; in some cases, particularly when using dilute alkali solutions, it is even advantageous thereto.

The finished products may be calendered, schreinered, beetled or the like.

Examples for producing pattern effects by the reserve method are not required as the methods are well known.

The term "cotton" in the following claims comprises cotton in the form of pure cotton fabrics or mixed fabrics, or in the form of yarns, for example, in hanks or cops or warps.

The expression "mercerizing agent" in the following claims comprises alkali solutions or strong mineral acids.

The expression "cellulosic body" is intended to cover: cellulose itself, mercerized cellulose, or a cellulose conversion product, for example cellulose hydrate.

I claim:

1. The process of improving cotton which comprises treating it with carbon bisulphide in the presence of caustic alkali at a temperature which does not substantially exceed 5° C.

2. The process of improving cotton which comprises treating it with carbon bisulphide in the presence of caustic alkali at a temperature of below 0° C.

3. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises treating cotton with carbon bisulphide in presence of caustic alkali solution first above 0° C. and thereafter below 0° C.

4. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises treating cotton with carbon bisulphide in presence of caustic alkali solution first above room temperature and thereafter below 0° C.

5. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises treating cotton with carbon bisulphide in presence of caustic alkali solution at a temperature ex-

(6) Stretched unbleached or bleached cotton cloth is treated with a caustic soda solution of 5-15 per cent strength (for example of 10 per cent strength) containing 0.2-3 per cent of carbon bisulphide, at room temperature for a few seconds up to 10 minutes, or at +30° to +50° C. for a few seconds up to 5 minutes. The alkali solution is then cooled or the cloth is transferred to a previously cooled alkali solution, as in the preceding examples. The result is approximately as in Example (1).

(7) Unbleached or bleached cotton fabric is treated with caustic soda solution of 15-18 per cent strength, the excess of alkali solution is squeezed out and the cotton fabric, either directly or after it has been kept for 1 to 3 days at room temperature, is subjected to the action of carbon bisulphide vapours or of liquid carbon bisulphide alone or in admixture with a suitable diluent (for example a carbon bisulphide solution of 1/2 to 10 per cent strength in benzene), for a few seconds up to 10 minutes. The material is then immersed in a caustic soda solution of 5-15 per cent strength (for example of 10 per cent strength) which is cooled to 0° to -12° C. either from the first or after 10 minutes to one hour. Completion of the operation is as in the foregoing examples.

In this example the fabric may be stretched before or after the carbon bisulphide treatment.

The result is a silky lustre or finish or both according to the duration of the sulphidizing and accordingly as the material was or was not stretched. When stretching is not employed substantially no silky lustre is produced, but instead the cotton fabric or yarn assumes a wool-like appearance and feel respectively.

(8) The mode of operation is as in the preceding examples, except that to the alkali solution in which the freezing action occurs 0.2 to 1 per cent of cellulose (for example sulphite-cellulose or cotton, preferably in finely subdivided form, or of cellulose previously ground with water or of mercerized cellulose in finely subdivided form) is added.

(9) The mode of operation is as in the preceding examples, except that the fabric is treated with a strong sulphuric acid before it is subjected to the process. When a sulphuric acid of 49-50° Baumé specific gravity is used for this purpose, the treatment therewith, at room temperature, may occupy some seconds up to a few minutes or even longer. If a stronger sulphuric acid is used, for example one of 52-54° Baumé specific gravity, the treatment should occupy only a few seconds, if the sulphuric acid has room temperature. It may, however, occupy a

ceeding 0° C. and subjecting the cotton thus treated to the action of caustic alkali solution at a temperature below 0° C.

5 6. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises treating cotton with carbon bisulphide in presence of caustic alkali solution at a temperature exceeding room temperature and subjecting the cotton thus treated to the action of caustic alkali solution at a temperature below 0° C.

7. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises first treating cotton with carbon bisulphide, secondly with caustic alkali solution at a temperature above 0° C. and, finally subjecting it, in presence of caustic alkali solution to the action of a temperature below 0° C.

20 8. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises first treating cotton with carbon bisulphide, secondly with caustic alkali solution at a temperature above room temperature and finally subjecting it, in presence of caustic alkali solution, to the action of a temperature below 0° C.

9. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises first treating cotton with carbon bisulphide and then with caustic alkali solution at a temperature which does not substantially exceed 5° C.

35 10. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises first treating cotton with carbon bisulphide and then with caustic alkali solution at a temperature below 0° C.

40 11. The process for improving cotton by treatment with caustic alkali and carbon bisulphide which process comprises treating cotton with a mixture of caustic alkali solution and carbon bisulphide, first at a temperature above 0° C. and thereafter at a temperature below 0° C.

12. The process for improving cotton by treatment with caustic alkali and carbon bisulphide which process comprises treating cotton with a mixture of caustic alkali solution and carbon bisulphide, first at a temperature above room temperature and thereafter at a temperature below 0° C.

13. The process of improving cotton which comprises treating it with carbon bisulphide in the presence of caustic alkali at a temperature below 0° C., and adding to the caustic alkali with which the cotton is treated a cellulosic body.

85 14. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises first treating cotton with carbon bisulphide diluted with a diluent, secondly with caustic alkali solution at a temperature above 0° C. and finally sub-

jecting it, in presence of caustic alkali solution, to the action of a temperature below 0° C.

15. Process for improving cotton by treatment with caustic alkali and carbon bisulphide, which process comprises first treating cotton with carbon bisulphide diluted with a diluent and then with caustic alkali solution at a temperature below 0° C.

16. The process of improving cotton which comprises treating it with carbon bisulphide in the presence of caustic alkali at a temperature below 0° C., said cotton having previously been treated with a mercerizing agent.

17. The process of improving cotton which comprises treating it with carbon bisulphide in the presence of caustic alkali solution at a temperature below 0° C., and continuing the cold treatment at least up to incipient freezing of the alkali solution.

18. The process of treating cellulosic textile material by subjecting it to the combined action of carbon bisulphide and caustic alkali solution at a temperature which does not exceed 5° C. and for a period of time insufficient to cause complete dissolution of the material.

19. The process of treating cellulosic textile material by subjecting it to the combined action of carbon bisulphide and caustic alkali solution at a temperature below 0° C. and for a period of time insufficient to cause complete dissolution of the material.

20. The process of treating cellulosic textile material by subjecting it to the combined action of carbon bisulphide and caustic alkali solution at a temperature at one time above 0° C. and at another time at a temperature below 0° C., and for a period of time insufficient to cause complete dissolution of the material.

In testimony whereof I affix my signature.  
DR. LEON LILIENFELD.

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