

# ALIEN PROPERTY CUSTODIAN

## TREATMENT OF TEXTILE MATERIAL

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This invention contemplates a method for treating textile material with a cuprammonium cellulose solution. More particularly the invention refers to a method of incorporating cellulose to fibrous material and aims at a permanent loading of the material, or a sizing insofar as warp threads are concerned, or a better grip insofar as fabrics are concerned. The solution used in the process is a so-called saturated solution that is a solution which is saturated with cellulose and is practically free from an excess of copper oxide ammonia. Therefor it has practically no etching effect on cellulose.

In using cellulose solutions of this kind, it is a characteristic feature of my invention that the original textile material to be treated is the same as the loading or sizing or finishing substance, namely cellulose, since I found that a permanent effect cannot be secured but by homogeneousness; therefor the fibrous material to be treated must be cellulose, either in the natural form or in a regenerated form (artificial threads or fibres obtained by the viscose or cuprammonium-process).

Hitherto the textile material has been impregnated with a cuprammonium cellulose solution and then treated with suitable means to precipitate and decopperise the cellulose previously dissolved within the impregnating solution.

This after-treatment was hitherto performed by a plurality of steps, the first of which consisted in the use of an alkaline bath for instance soda lye to coagulate the cellulose, while in the final step the copper in the coagulated cellulose was dissolved by suitable acid solutions for instance diluted sulphuric acid.

Another method of after-treatment consisted in precipitating the cellulose and decopperising same by a single treatment for instance with diluted acid.

According to my invention the textile material is impregnated with a cuprammonium cellulose solution and then, preferably after the excess of the impregnating solution has been removed, treated with suitable means to evaporate the ammonia. There results a precipitation of cellulose in and on the fibrous material which still contains the copper in the form of hydroxide, but is substantially free from ammonia. The textile material is then treated with a solution of acid reaction to decopperise the precipitate. After a neutralisation the textile material is washed and dried.

The evaporation of the ammonia can be performed in different ways. Suction can be used with advantage. I prefer, however, the method

of evaporating the ammonia by heat. The impregnated material is either guided along a heated surface for instance a heated rotated drum, or it is brought into contact with a hot gaseous means, for instance heated air which may be stagnant or moved preferably in countercurrent to the direction of movement of the textile material. Temperatures up to about 105° C. are useful, while higher temperatures may cause a modification of the precipitated copper which is difficultly soluble in the decopperising liquids.

A kind of hot gaseous means is steam which acts as heat source and carries away the evaporated ammonia.

A plurality of these means may be used in combination.

Together with the ammonia the water of the cellulose solution may be evaporated either in part or totally.

The dry or wet gaseous ammonia which is removed from the textile material may be collected either by increased pressure or by suitable washing liquids for instance water or sulphuric acid, or by adsorbents for instance activated carbon. The ammonia can easily be reused as condensate or aqueous solution, or by heating the adsorbate, or, in the case of acids as washing liquids, by decomposing the formed ammonia salt by suitable substances for instance caustic alkali or alkaline earths.

After the ammonia has been removed from the textile material, the decopperising step takes place. Inorganic or organic acids or acid salts of low concentration can be used, for instance sulphuric, hydrochloric, formic or acetic acid. Since the textile material is practically free from ammonia, aqueous solutions from 1 to 2 percent of acid are sufficient to dissolve the copper. After exhaustion the decopperising liquid may be treated to recover the copper. The dilute liquid may be concentrated preferably after neutralisation, and a copper salt is obtained by crystallization. Further methods consist in recovering the copper in metallic form either by electrolysis or by cementation (addition of metallic iron).

The preferred method, however, consists in adding alkaline substances for instance caustic or carbonic alkali or both in a sufficient amount to precipitate the copper as basic salt or hydroxide, which readily can be reused for the preparation of a new bulk of cellulose solution.

The textile material freed from copper is neutralised, washed and finally dried.

The effect is, in the case of fabrics, a better grip, in the case of yarns a perfect size, and par-

ticularly in the case of fibre flocks a considerable increase in weight which amounts up to 14 per cent.

According to the invention a perfect and separate recovery of the chemical substances used in the process is secured. The ammonia can be recovered without losses and in a form which can readily be reused, and the decopperising liquid remains free from ammonia. The copper can easily be regained from the decopperising liquid as basic salt or hydroxide which are suitable for the preparation of the cellulose solution. A further advantage consists in that the decopperising liquid can be weaker than before since the textile material to be decopperised is substantially free from ammonia, which otherwise consumes a part of the acid. This is a distinct advantage particularly in the case of a continuous process of sizing warp threads or finishing fabrics since the decopperising liquid remains efficacious for a longer period.

The cuprammonium cellulose solution is, as previously mentioned, a saturated one, and is preferably of low viscosity, approximately like machine oil. This viscosity can be obtained by an additional amount of ammonia in comparison to that amount which is commonly used in spinning solutions. Further methods for decreasing the viscosity consist in reducing the cellulose concentration to about 2 to 6 percent, calculated to air-dry cellulose, or by avoiding addition of neutral salts to the solution or formation of same in the solution, or by employing a plurality or all of these means.

In applying the cellulose solution to the textile material it is advisable to remove the excess solution from the impregnated material by mechanical means for instance soaking, centrifuging, squeezing or the like. This treatment is of particular importance in the case of flocks which should not stick together after the treatment.

The removal of the copper must not necessarily be carried out in the same form of textile material which previously had been impregnated with the solution.

In certain cases it may be advantageous to carry out a textile operation with the impregnated textile material after the same has been freed from ammonia and dried but before the removal of the copper, and to decopperise the material after that textile operation. Thus, for instance, dry copper-containing blue threads may be woven to a fabric which is afterwards decopperised. The blue copper-containing threads are particularly suitable for textile operations, and can be stored as long as desired. Their elasticity is excellent.

#### Example 1

A suitable vessel provided with stirring or kneading arms is filled with cotton flock and with an easily fluid copperoxide ammonia solution having a content of about 3% cellulose. The

amount of the solution is about twenty times as much as the weight of the fibre. The content of the vessel is thoroughly agitated during half an hour. The content of the vessel is then centrifuged to remove the excess of the solution, and afterwards treated with steam until the ammonia is substantially evaporated. Instead of this treatment, the fibrous mass after being freed from the excess of the cellulose solution is brought in another closed vessel and subjected therein to suction.

The blue fibrous mass is then decopperised by sulphuric acid of a concentration of 1 to 2 percent, neutralised, washed and finally dried; the flock is now ready for use in the carding-process.

When flocks of artificial fibres consisting of regenerated cellulose are treated in the same manner, the fibres get rid of their limp touch, furthermore their spinning qualities are considerably improved.

#### Example 2

A large number of warp threads consisting of natural or of regenerated cellulose or of both are drawn off from a warp beam or a number of such beams and impregnated or coated by a cuprammonium cellulose solution, preferably of somewhat higher viscosity than that according to Example 1. The excess of the solution is removed by a wiper or scraper made of rubber; the threads travel along to a heat source for instance around a steamheated rotatable drum or through a chamber which may be provided with heating elements or through which passes a heated gas or air. The threads freed from ammonia are then guided through a bath consisting of dilute sulphuric acid of three percent, squeezed, neutralised, washed and finally dried on a heated drum or in another drying chamber of the same kind as mentioned before. Finally, the dried threads are wound on a warp beam. The process is carried out continuously.

According to a modification of this method the action of the heat on the freshly impregnated threads is continued until they are dry, and the blue threads are immediately wound on the warp beam. The decopperisation takes place in the woven fabric.

#### Example 3

A fabric made from cotton yarn or from artificial fibres of regenerated cellulose or from both is treated in a similar manner as the warp threads according to Example 2. The effect of the treatment consists in a better appearance and a better grip.

Instead of evaporating the ammonia by heat, steam may be employed in a closed chamber to evaporate the ammonia and carry it away to a receptacle in which aqueous ammonia is condensed either by cold or by pressure or by both.

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