

## UNITED STATES PATENT OFFICE

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## DELUSTERING AND WEIGHTING TEXTILE MATERIALS

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This invention relates to improvements in the treatment of fibres, yarns or fabrics, particularly for delustering natural silk, artificial silk of all types including cellulose esters or ethers or regenerated cellulose such as viscose silk, and has for its object to provide a new or improved process for treating such material or for weighting and/or delustering natural silk, linen or other materials.

Further objects of the invention are to avoid waste of reagents, to improve control, to allow higher concentrations of reagents to be employed and to obtain pattern effects of a very varied character.

The invention is a continuation in part of our application No. 93,232.

According to the present invention, the textile material is treated with the ingredients in two phases or stages, i. e., with some of the ingredients in one fluid, either liquid or gaseous, and with the remaining ingredients in another fluid. Illustrative of a desirable two phase procedure, two different liquid baths may be employed for successive treatment of the textile material. One bath may contain a solution of two of the ingredients and a second bath the third, but if desired the first bath may contain some proportion of all the ingredients and the second bath the requisite proportion of one or more of them to effect rapid precipitation.

The main advantages are as follows:

(a) If, for instance, the cloth or yarn is first passed through a bath containing urea, then there is obviously no waste of urea as the bath can be exhausted. In the case of the one-bath process there will always be a certain amount of methylene urea precipitate which cannot be used. Our experience has been that it is not possible to exhaust the reagents in a one-bath process, whether it be composed of the simple solution which we employ or the more complicated solutions obtained by dissolving solid partial condensates in acid.

(b) Another advantage of the two-bath process lies in the greater control which may be exercised. Once the methylene urea starts forming in the one-bath process there is very little which can be done to hinder it and the deposition takes place fairly rapidly. In the two-bath process greater control is possible as it is only in the second bath that any precipitation is

likely to occur and this second bath, usually of formaldehyde, may be kept to such dimensions that renewal of the liquor is necessary before precipitation occurs in the bath.

The use of two baths enables many variations to be made in the concentrations of the reagents in the baths and so permits of a greater range of delustering effects.

The precipitation of methylene urea is to some extent a time reaction and this is capable of more exact control in the case of the two-bath process.

(c) For certain effects, mainly localised effects where contrast is essential, the amount of urea is critical in many cases and this is another point in favour of the two-bath process. For instance, in some effects it is necessary to have 14% of urea in the cloth and we have not found a satisfactory means of achieving this by any one-bath process.

(d) Where a heavy matt effect is necessary, as against a mere opalescence, it is practically impossible to obtain this by a one-bath process on a commercial scale; this is important where strong contrasts are required. Similar considerations apply where methylene urea is used for increasing the weight and bulk of the cloth, here again the two-bath process is preferable.

(e) Lastly, the range of pattern effects (coloured, lustrous pattern on a white matt ground and many variations on this idea) is obviously very limited in the one-bath process. In the two-bath process it is possible to impregnate with the urea say and then apply the printing technique, plain resists, coloured resists, etc., before treating in the second bath which produces the local matt effect where desired. This offers much more scope for the contrast of matt and lustre effect combined with plain and coloured effects.

A preferred insoluble condensation product in particulate form is obtained from urea and formaldehyde. The insoluble compounds formed may be methylene ureas. It is also possible to use thio-urea. Other suitable non-resinous condensation products may be obtained by condensation to a limited extent of components which, if further condensed, would yield synthetic resins.

Thus, impregnation can be accomplished by treating the material with a solution of soluble intermediate condensation products or with the

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components thereof and then bringing about precipitation of an insoluble product from the solution. The material is impregnated with the solution containing a precipitant in sufficient quantity to form on the textile material, after sufficient time of contact, a precipitate substantially fast to washing. The material impregnated with the solution may alternatively be brought into contact with a precipitant such as an acid. Alternatively the material may be first treated with a precipitant such as an acid and then impregnated with the urea-formaldehyde solution.

The textile material may be treated with the components of a synthetic condensation product, separately and in any desired sequence, but it is preferred to impregnate with urea and formaldehyde simultaneously.

When yarns or fibres are treated, the impregnation is performed without materially distorting the materials.

Where weighting is required, it is desirable to bring about the precipitation on or in the material of a large amount of insoluble condensation product. For delustring alone, smaller quantities will suffice.

Fibres, yarns or fabrics may be treated by the process and the material so treated will withstand washing without regaining its lustre. The treated materials can be dyed. Cellulosic materials treated by the present process will withstand the mercerisation operation, and can also be dyed with acid dyes.

#### Example 1

Urea -----gms-- 60  
Formaldehyde (neutral 40% solution) ---cc-- 100  
Water -----gms-- 100

are mixed together without any heating. The fabric, yarn or fibres in the form of sliver or roving, is immersed in this solution until thoroughly wetted out, squeezed until it contains about its own weight of liquor and then immersed in 2% HCl until the delustring has taken place, when the fabric, yarn or fibre is well washed with soap and soda ash (sodium carbonate) solution together with an oil, then washed in warm water, squeezed and dried at ordinary drying temperatures, i. e., not above 100°-105° C.

#### Example 2

Urea -----gms-- 60  
Formaldehyde (neutral 40% solution) ---cc-- 100  
Water -----gms-- 100

are mixed together without any heating, and the fabric, yarn or fibre in the form of sliver or roving well wetted with a solution, squeezed and then subjected to acid vapour until the delustring has occurred, then well washed with soap and soda ash solution together with an oil, and finally washed and dried as in Example 1.

#### Example 3

Urea -----gms-- 100  
Formaldehyde (neutral 40% solution) ---cc-- 200

are refluxed with 3%  $\text{NH}_4\text{OH}$  (sp. gr. .880) for 3 mins. at the boil, cooled, 300 cc. water added and acidified with  $\frac{1}{2}$  to 1% tartaric acid. Fabric, yarns or fibres are immersed in this solution passed through a mangle and allowed to stand until the delustring has taken place, and finally washed and dried as in Example 1.

#### Example 4

60 gms. urea are dissolved in 100 ccs. form-

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aldehyde (solution 40%). A natural silk fabric is run through this solution and squeezed through rollers to ensure even penetration. The cloth is then passed quickly through 4% hydrochloric acid (spec. grav. 1.02) and the reaction allowed to proceed until precipitation takes place. The amount of weighting obtained will depend on the amount of liquor left on the cloth, on the nature of the cloth to be treated, on the time for which precipitation is allowed to take place and on the nature of the catalyst. After precipitation has taken place the fabric is well washed with soap and soda ash and oil, then washed in water and finally dried.

In order to obtain further increase in weight, the whole process can then be repeatedly performed until the requisite increase in weight is obtained.

It is also possible to obtain plain (undyed) or dyed pattern effects by applying the process to selected areas of the yarn or fabric in any of the ways well known for producing patterns, e. g., the suitably thickened urea-formaldehyde solution may be applied by direct printing followed by precipitation; selected areas may be treated with a suitable resist (e. g., with the ethanolamines and other bases or with known resists and then the delustring process applied, or selected areas of the delustred material may have the lustre restored by discharging (printing with a thickened acid) and steaming, followed by washing). The patterned fabric may be dyed or dyes may be incorporated in the resist.

#### Example 5

The fabric is impregnated with a solution of urea and dried at the ordinary temperatures of drying textile materials. It is then printed with a vat colour in the ordinary way using sufficient caustic soda to withstand the subsequent acid treatment and after standing the cloth is run through a mixture of acidified formaldehyde and squeezed and allowed to stand in the damp condition until the delustring has taken place on the unprinted portions of the fabric. Bright coloured effects can thus be obtained on a matt ground.

#### Example 6.—All-over delustred effect

25 lbs. of urea was dissolved in 10 gals. of water and 3 qts. of glycerine added. An all-viscose satin cloth was impregnated with this solution and excess liquid squeezed off by passing it between the bowls of a two-bowl mangle adjusted so as to leave a quantity of liquor on the cloth equivalent to about 80% of its own weight.

The cloth was then dried at a moderate temperature and then impregnated with a solution of 60 parts by volume of commercial 40% formaldehyde and 4 parts by volume of sulphuric acid (specific gravity 1.72) in 40 parts by volume of water. The cloth was then allowed to stand exposed to the air for 6-7 mins. to allow the delustring effect to develop, after which time the cloth was washed first in water, then in soap solution and finally rinsed in water and dried.

#### Example 7.—Delustred coloured design with an undelustred background

The requisite amount of vat dye ester, for example, Soledon or Indigosol dyestuffs, is dissolved in one part by volume of water or other solvent. This mixture is added to 9 parts by volume of a paste made by mixing 100 lbs. urea, 8 lbs. sodium chlorate, 6 lbs. ammonium chloride, 3 ozs.

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ammonium vanadate, 4 gals. glycerine, 23 gals. gum dragon or water to dissolve the above chemicals, 1 qt. ammonia, and the whole then made up to 36 gallons.

A cotton viscose mixture fabric was printed with this colour, dried at a moderate temperature and passed through an ager for 4-5 mins. to develop the colour. The fabric was then impregnated with a cold solution containing 65 parts by volume of commercial 40% formaldehyde, 5 parts by volume of sulphuric acid (specific gravity 1.72) and 30 parts by volume of water and then passed face downwards through a two-bowl mangle.

The cloth was then allowed to stand exposed to the air for 6-7 mins. during which time the delustering effect developed. The fabric was then washed in water and again in dilute soda ash solution and finally washed in boiling soap solution. The fabric was then rinsed in water and finished in the usual way.

Components of synthetic condensation products other than those indicated may be used but those which will react rapidly in presence of a catalyst such as an acid are particularly suitable. The process differs from that disclosed in German Patent 499,818 in that precipitation of an insoluble condensation product is designedly brought about to make resin formation difficult; it is not necessary to dry the material and heat it to a high temperature since the intermediate compound can be made insoluble while the fabric is in the wet state by the action of acid at room temperature. As the product is not deposited in a coherent impermeable layer but rather in particulate form, it is possible to treat a textile material to delustre it, and then to treat it further according to the process of German Patent 499,818 to improve its resistance to creasing, or to dye it, or to submit it to other suitable textile processes.

The treatment is effected in such manner as to cause a large reduction of lustre.

The lustre of a fabric is due to the specular reflection (i. e., reflection as from a mirror) of light from the fibres.

For the purpose of measurement it may be defined as the ratio of the intensity of the specularly reflected beam at a given angle to the diffusely reflected beam in any direction.

It is assumed that the diffusely reflected beam is of the same intensity in all directions.

As lustre defined in this way varies with the direction in which the fabric is viewed, we prefer to make all measurements in the direction of maximum lustre.

In the special example of an all-viscose satin we wish to define delustering as reducing the lustre at least to  $\frac{1}{2}$  the original lustre. With less lustrous fabrics than all-viscose satin, not so great a reduction is obtained with similar treatment; in treating other kinds of fabrics the treatment should be such as if applied to an all-viscose satin, its lustre would be reduced at least to  $\frac{1}{2}$  the original lustre.

The treatment should not be such as to deposit a coherent resinous layer. The time of treatment must be sufficiently prolonged to allow of adequate precipitation, though this varies with acidity, dilution, temperature and ratio of formaldehyde to urea.

Moreover after the precipitation in situ on the textile material of the non-resinous substance, such as methylene urea, from the aqueous solution, the wet fabric should be treated, prior to

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drying the same, to remove therefrom any substance or ingredient adapted to cause resin formation upon the drying of said material. Illustratively, this may be accomplished by washing the fabric with an aqueous solution pursuant to the various examples hereinabove disclosed.

We declare that what we claim is:

1. A process for delustering textile material in aqueous solution which comprises impregnating said material with a solution containing urea and contacting the so treated material in another phase with another solution containing an aldehyde and an acid, whereby an insoluble, non-resinous, synthetic condensation product in particulate form is precipitated in situ on the material to delustre the same, and washing said wet textile material having the non-resinous product precipitated thereon to remove any ingredient adapted to cause resin formation upon drying said material.

2. The method as in claim 1 wherein the aldehyde is formaldehyde and the acid is hydrochloric acid.

3. A process for delustering textile material which comprises impregnating said material with an aqueous solution containing urea, drying, printing with an acid-resisting agent, and contacting the so treated material in another phase with another aqueous solution containing an aldehyde and an acid, whereby an insoluble, non-resinous, synthetic condensation product in particulate form is precipitated in situ on the material to delustre the same, and washing the wet textile material with substances adapted to substantially completely remove any unreacted ingredients prior to drying the same.

4. The process of delustering textile material by the method of precipitating an insoluble non-resinous material by addition of an acid to a solution providing a source of amino and aldehyde compounds capable of forming an aminoplastic aldehyde resin on condensation and adapted to form a non-resinous compound in aqueous solution characterized by the feature that the textile material is contacted in at least two phases with at least two different ingredients including an amino compound, an aldehyde and an acid, each phase containing at least one and less than all of said reagents, the two phases jointly including all of said ingredients and water, and contacting said wet textile material having the non-resinous precipitate thereon with an aqueous liquid adapted to remove any substance which will cause resin formation upon drying said material.

5. The process of delustering textile material with methylene urea characterized by the feature that the material is treated in two different baths with two different solutions jointly containing urea, formaldehyde, water and sufficient acid so that on interaction of said solutions methylene urea is precipitated in situ on the textile material, and washing the wet textile material after said methylene urea formation with an alkaline solution, to thereby prevent resin formation upon drying said material.

6. The process of delustering textile material by the precipitation thereon of a water insoluble non-resinous substance comprising treating the textile material in two distinct phases which jointly include an aqueous solution containing as ingredients urea and formaldehyde together with an acid in quantity sufficient to cause the precipitation of methylene urea, each phase containing at least one and less than all of said in-

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gradients, thereby forming said methylene urea precipitate in situ on said textile material, and removing from said wet textile material having the methylene urea precipitate thereon any substance adapted to cause resin formation upon drying the said material.

7. The process of delustring textile material by the precipitation thereon of a water insoluble non-resinous substance comprising treating the textile material in two distinct phases which jointly include an aqueous solution containing as ingredients a source of amino and aldehyde compounds adapted to form a non-resinous precipitate in aqueous solution, together with acid in quantity sufficient to cause the precipitation of said non-resinous substance, each phase containing at least one and less than all of said ingredients, thereby forming said non-resinous precipitate in situ on said textile material, and removing from said wet textile material having said precipitate thereon any substance adapted to cause resin formation upon drying the said material.

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