

## UNITED STATES PATENT OFFICE

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## METHOD OF DELUSTERING

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The present invention relates to a method and agent for processing regenerated cellulose. More particularly, it relates to a process for delustering regenerated cellulose textile materials.

Regenerated cellulose can be dulled, as is known, with titanium dioxide or the like. In this case satisfactory effects are particularly produced when the dulling material is incorporated with the spinning solution. These effects sufficiently fulfil the usual requirements. As is known, the mechanical working up of rayon thus pre-treated involves, however, considerable difficulties. A material after-dulled by means of titanium dioxide or another inorganic pigment was, however, hitherto not sufficiently resistant to washing; furthermore a detrimental effect was produced by the fact that the water-repellency always considerably decreased when for instance titanium dioxide was combined with the products usually applied in commerce for the production of a water-repellent processing.

Now we have found that pigment which do no longer show the drawbacks described above may be obtained in a simple manner by treating titanium dioxide, zinc sulfide or mineral earths, such as china clay, kaolin or the like, in a manner known as such, with aqueous sols of alumina or zirconia. Pigments of this kind may be applied to the material on the padding machine either in the form of an aqueous suspension or together with other impregnating agents and finishing agents. The pigments may also be applied together with suitable adjuvants, for instance those named in the co-pending U. S. application Serial No. 193,850, filed March 4, 1938, now issued as U. S. Patent 2,185,427 dated January 2, 1940, in a highly diluted solution, for instance by treating the material on the winch. The resistance of the dulled goods to washing which as such is already very high may be further increased by simultaneously using basic salts of zircon, for instance zircon oxychloride. The simultaneous use of a small quantity of an agent capable of precipitating salts of aluminium or zircon in the rinsing bath, for instance ammonia, is likewise of advantage.

The following examples serves to illustrate the invention, but they are not intended to limit it thereto:

(1) A solution of 1 part of hydroxy-ethylated stearyl-biguanidine in 500 parts of water is intimately mixed with 500 parts of titanium dioxide, 50 parts of sol of alumina (50 per cent of  $\text{Al}_2\text{O}_3$ ) dissolved in 200 parts of water is then caused to run in and the whole is stirred until

the mass which temporarily has become solid liquefies again. With the same success there may be used a sol of zirconia instead of a sol of alumina.

10 parts of the paste thus obtained are diluted with three times to five times its volume of water of about  $35^\circ\text{C}$ . and the suspension thus produced is added to an impregnation liquor prepared in the usual manner. If, for instance, tissue of artificial silk staple fiber is treated with this suspension, the fabric is delusted, but the water-repellent effect produced by the impregnating agent is not impaired thereby. The dulled goods are resistant to several mild soap scourings.

(2) 170 parts of the paste obtained as described in the first paragraph of Example 1 are mixed with 6 parts of the emulsifying agent named in said paragraph and 3 parts of an ester of a monocarboxylic acid such as coconut oil fatty acids, castor oil fatty acids and the like with polyglycols or analogues thereof such as are described in German Patent No. 575,911. The product thus obtained may be used with excellent success for dulling rayon on the winch. The pigment is slowly and uniformly adsorbed. By the addition of weak alkalies, such as ammonia, to the rinsing bath the resistance of the dulled goods to washing is further improved.

(3) 50 parts of sol of alumina (50 per cent of  $\text{Al}_2\text{O}_3$ ) in 200 parts of water are gradually caused to run, while stirring, into 500 parts of zinc sulfide which have been intimately mixed, as described in Example 1 with hydroxyethylated stearylguanidine. The zinc sulfide thus treated possesses an excellent floating power and in combination with the known filling, finishing and impregnating agents it is an excellent dulling agent for textile materials.

(4) 25 parts of china clay are intimately kneaded with 2.5 parts of sol of alumina (50 per cent of  $\text{Al}_2\text{O}_3$ ) and 50 parts of water. Into the suspension thus obtained there is caused to run, while well stirring, a mixture heated to  $60^\circ\text{C}$ . of 16 parts of stearic acid polyglycerinester, 2 parts of oleyl-polyglycol ether and 8 parts of urea and the whole is stirred until the mass has cooled. By the addition of a small quantity of acetic acid the stability of the liquors to be applied to hard water is considerably increased. Solutions containing 100 to 200 grams per liter of the afore-named mixture yield on the padding machine a good, non-dusting dulled material having an agreeable and soft handle.

The invention is of course not limited to the

specific details described, for obvious modifications may occur to a person skilled in the art.

We claim:

1. A method for delustering regenerated cellulose textile materials which comprises treating regenerated cellulose with solutions containing inorganic dulling pigments pretreated with sols of the group consisting of sol of alumina and sol of zirconia.

2. A method for delustering regenerated cel- 10

lulose textile material which comprises treating regenerated cellulose with solutions containing impregnating agents rendering the material water-repellent and inorganic dulling pigments pretreated with sols of the group consisting of sol of alumina and sol of zirconia.

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