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PROCESS FOR TREATING NITRATED MATERIAL

Brian Porter Sutherland, Geoffrey Thorp Shaw,
and Darcy Drummond Morris, Trail, British
Columbia, Canada, assignors to The Consoli-
dated Mining & Smelting Company of Canada,
Limited, Montreal, Quebec, Canada, a corpora-
tion of Canada

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Our invention relates to a protective treatment for nitrated cellulose fibre in the form of thread or cloth, which is to be exposed to the action of acid solutions.

5 The use of nitrated cellulose cloth for filtering acid solutions is well known. The filter cloth may be nitrated after it is woven or may be woven originally from spun nitro-cellulose fibre. It is also well known that when this type of material is exposed to the action of acid solutions, it shows a gradual loss of tensile strength, which loss is caused by the progressive disintegration of the material, until finally the cloth becomes completely disintegrated, with the result that replacement is necessary. The rate of disintegration of the fibre varies, of course, with the type, the concentration and the temperature of the acid solution to which the material is exposed.

10 A principal feature of the present invention resides in the method of treating the nitrated cellulose material in the form of fibre, thread or cloth, whereby the resistance of such costly material to disintegration on exposure to attack by acid solutions is greatly increased and its rate of loss of tensile strength under such conditions is greatly retarded.

15 A further feature of our invention resides in its application to the treatment of nitrated cellulose cloth of an inferior quality, particularly where such cloth is otherwise practically useless for filtering acid solutions. The resistance of such inferior cloth to attack by acid solutions is increased by our treatment to a degree of resistance only slightly less than that shown by a good grade of cloth after the latter has been given our protective treatment.

20 In the development of our invention, we have found that the resistance of the nitrated cellulose material to attack by acid solutions is greatly increased when the material is treated with certain organic compounds. The most suitable compounds are those we have found to be included in the group identified as the aromatic amine group. This general classification includes compounds which may have no beneficial results and others which have a definitely deleterious action on the fibre. For example, we have found that the resistance of nitrated cellulose material to attack by acid solutions, such as phosphoric acid, is decreased after the material has been treated with certain aromatic amine compounds such as orthotolidine, aniline, and benzidine, while compounds such as paratolidine have no apparent effect. In general, however, we have found that 25 the compounds included in the secondary amine

group in which the nitrogen atom is linked directly to two aromatic nuclei, such as phenyl-alpha-naphthylamine and diphenylamine are the most satisfactory while certain other aromatic amines and derivatives of aromatic amines, such as meta-nitroaniline, alphanaphthylamine and diphenylnitrosamine are fairly satisfactory.

As a preferred embodiment of our invention, we dissolve the organic chemical, such as phenyl-alpha-naphthylamine, in a suitable solvent. We prefer to use methanol or gasoline, and where there is danger of fire, carbon tetrachloride. Reasonable care, however, must be taken in selecting the solvent to ensure that it does not have a deleterious action on the nitrated material. For example, we have found that methanol is suitable in many instances, but some cloths are partially dissolved by methanol, probably because of their tri-nitrate content, whereas other cloths, more fully nitrated, are not so attacked.

20 The nitrated material is usually stored under water, while awaiting use, and it is generally preferable to dry the material prior to immersing it in the protecting solution. The degree of drying depends on the type of solvent used. For example, in using methanol, which is miscible with water, satisfactory results may be obtained by removing sufficient water, such as by draining, to prevent dilution of the methanol solution and to avoid precipitation of the protective agent prior to its action upon the fibres of the cloth. In instances where gasoline is used as a solvent, it is usually preferable to have the cloth substantially dry prior to its immersion in the solution.

25 After the drying step, the cloth is immersed in the protective solution for a period of time depending on the weight of the cloth, the concentration of the solution and other variants, but usually not less than from two to three hours. A longer time is no disadvantage if an inert solvent is used. After the immersion period, the cloth is drained and preferably air dried prior to using or returning to water storage.

30 As a specific example of our process, nitrated cotton cloth weighing about 20 ounces per square yard and containing about 12% nitrogen (the nitrogen content being principally tetra and penta nitrate, which are practically insoluble in methanol) was spread on racks and dried at a temperature of about 80° F. The cloth was then immersed in a solution of methanol containing about 0.7 pound of phenylalpha-naphthylamine per imperial gallon and left soaking for a period of sixteen hours after which it was removed from the solution, drained, air dried and returned to

water storage. There was a consumption of about one ounce of phenylalpha-naphthylamine and a loss by evaporation of about eight ounces of solvent per square yard of material treated. 5 The solvent could be recovered in known ways, if desired.

In laboratory tests, the untreated nitrated cloth showed a loss in tensile strength of more than 50% when exposed for a certain period to the action of crude phosphoric acid having a concentration of 40% P_2O_5 and maintained at a temperature of about 80° C. Untreated nitrated cloth of the same type, after treatment according to our invention as described above, showed an 10 inappreciable decrease in tensile strength after having been subjected to the same conditions for the same period of time.

In general commercial use, we have found that the useful life of the nitrated filter cloth is more than doubled when treated with phenylalpha-naphthylamine, as set out above, and exposed to the action of crude phosphoric acid having a concentration of 34% P_2O_5 and maintained at a temperature of 70° C. 20

Other results typical of those obtained from exposure of the treated filter cloth to the action of other acids are as follows: 25

30	Test conditions	Loss in tensile strength in percent	
		"Treated cloth"	"Untreated cloth"
35	30% H_2SO_4 at 60° C. for 72 hours	40	90
	30% HNO_3 at 60° C. for 24 hours	7	50
	10% HCl at 60° C. for 72 hours	50	100

The preceding results were obtained from tests carried out upon nitrated cloth which previously had been treated with diphenylamine dissolved in methanol. 40

We have found that our protective treatment when applied to spun nitrocellulose cloth increases its resistance to attack by acid solutions, although, on occasion, this increased resistance may be less pronounced than in the case where cloth, which has been nitrated after weaving, is treated by our process. 45

During the protective treatment, there is usually a certain amount of shrinkage in the nitrated cloth and particularly is this the case in the treatment of spun nitrocellulose. If a suitable solvent, such as described above, is used, there is no decrease in tensile strength, but rather an apparent increase due to the shrinkage. 50

We have found also that nitrated cloth, the 55

quality of which is inferior as a result of certain conditions prevailing during the nitration process and which is considered practically useless for such purposes as filtering acid solutions, may be satisfactorily protected by subjecting it to our protective treatment. The inferior cloth, after treatment, is found to show resistance to attack which is only slightly less than the resistance shown by a good grade of treated cloth. This feature is important in that it permits the operation of the nitration process under less rigidly controlled conditions and also serves to avoid the loss of cloth, which loss would otherwise result from rejection of the cloth spoiled in the nitration process. 10 15

It will be apparent, of course, that departures may be made from the preferred embodiment of our invention disclosed herein without departing from the scope of the appended claims.

What we claim as new and desire to protect by Letters Patent of the United States is: 20

1. A process for increasing the acid resisting properties of fibrous nitrated cellulosic material which comprises the step of immersing the fibrous material in a solution formed by dissolving, in a solvent in which said material is not soluble, an aromatic amine compound selected from the group consisting of diphenylamine, meta-nitroaniline, alphanaphthylamine, diphenylnitrosamine and phenylalphanaphthylamine. 25 30

2. Fibrous nitrated cellulosic material impregnated with an aromatic amine selected from the group consisting of diphenylamine, meta-nitroaniline, alphanaphthylamine, diphenylnitrosamine and phenylalphanaphthylamine. 35

3. Acid resistant nitrated cellulosic filter cloth impregnated with an aromatic amine selected from the group consisting of diphenylamine, meta-nitroaniline, alphanaphthylamine, diphenylnitrosamine and phenylalphanaphthylamine. 40

4. A process for increasing the acid resisting properties of fibrous nitrated cellulosic material which comprises the step of immersing the material in a solution formed by dissolving diphenylamine in a solvent in which said material is not soluble. 45

5. A process for increasing the acid resisting properties of fibrous nitrated cellulosic material which comprises the step of immersing the material in a solution formed by dissolving phenylalphanaphthylamine in a solvent in which said material is not soluble. 50

BRIAN PORTER SUTHERLAND. 55
GEOFFREY THORP SHAW.
DARCY DRUMMOND MORRIS.