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CHEMICAL PROCESS AND COMPOSITION

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This invention relates to the preparation of textile treating agents and to processes for the treatment of textiles. More particularly, it relates to the finishing of textile materials including filaments, fibers, threads, skeins, yarns and fabrics. Still more particularly, it relates to the finishing and softening of textile fibers, threads and fabrics. The invention also relates to treating solutions.

10 This invention has for an object to improve the art of finishing and softening textile materials. A further object is to produce new and effective textile treating agents. A further object is to treat natural and synthetic fibers, particularly cellulosic fibers, including the chemically modified cellulose fibers so that they will be soft and smooth to the touch. A still further object is to prepare soft, smooth, cellulosic fibers and fabrics which are free from objectionable odor and do not become rancid. A still further object is to soften and finish cellulosic fibers in such a manner that their strength will not be impaired. A still further object is to soften and finish synthetic and natural textile fibers and fabrics in such a manner that their solubility characteristics are not increased. Other objects will appear hereinafter.

The above and other objects are accomplished by the preparation and use of certain salts of the products prepared by reacting a non-gaseous saturated hydrocarbon with gaseous sulfur dioxide and chlorine and hydrolyzing the resulting products.

The hydrolysis is preferably carried out under such conditions that neutralization of the acidic groups is effected. While the compositions and compounds having free acid groups have surface active properties and are useful in textile treating operations, the water soluble amine salts are much more superior in such relations and in some instances give new and unexpected results.

An important embodiment of the invention resides in an efficient method of making amine salts, which are new compounds and compositions. This method comprises dissolving the initial reaction product resulting from treatment with sulfur dioxide and chlorine which is a complex mixture containing hydrocarbon sulfonyl chlorides, chlorhydrocarbon sulfonyl chlorides, hydrocarbon polysulfonyl chlorides, and chlorhydrocarbon polysulfonyl chlorides in a water-soluble organic solvent, especially an aliphatic monohydric or polyhydric alcohol and bubbling a gaseous or readily volatile amine,

e. g. mono-, di- and trimethyl, ethyl and ethanolamines therethrough at a temperature below 60° C. The reaction is continued until the hydrolysis and neutralization has gone to completion or to the desired extent. A continuous procedure may be resorted to if desired by drawing off hydrolyzed and neutralized products and adding fresh material.

While the products prepared by using concentrated alkali metal solutions have good finishing and softening properties for fibers and fabrics, it has been found that the salts from organic nitrogenous bases particularly lower aliphatic amines have greatly enhanced finishing and softening properties. The amine salts represent the preferred embodiment of this invention.

It has been found that when fibers, yarns, fabrics, etc., either synthetic or cellulosic are treated with aqueous solutions containing small amounts of the above compounds, they become soft and smooth to the touch, free from odor and color and their strength is not impaired. In the case of the amine salts the resulting fibers are softened to a marked degree. They are not only smooth and soft, but are fuller, more pliable and in general more pleasing to the touch.

The compounds are usually employed in the form of a treating solution which is preferably aqueous. They may be employed over a wide range of concentration. From 0.001% to 5.0% by weight of the solution represents a practical range. The amine salts even when used in concentrations on the order of 0.001% to 0.5% by weight of the aqueous liquid give excellent softening and finishing results.

The invention will be further illustrated, but is not intended to be limited by the following examples, in which the parts stated are parts by weight unless otherwise indicated.

Example I

Five hundred grams Asiatic wax melting point 58-60° C. was placed in a three liter flask and a mixture of sulfur dioxide and chlorine passed in under the influence of light and with stirring. The initial temperature was 128° due to heating the wax to melt it. The temperature gradually dropped to 95° C. where it stayed due to the heat of reaction. After seven hours, the gain in weight was 450 g. The product is a very thick water-white syrup, containing 25.8% chlorines and 11.11% sulfur.

One hundred grams of the above reaction product was mixed with 200 cc. (95%) alcohol and dimethylamine gas was passed in keeping the

temperature below 50° C. by the use of an ice bath. After the reaction product, which was initially insoluble in the alcohol, had become completely soluble the flow of dimethylamine gas was interrupted and the reaction mass was stirred for ten minutes, then heated on the steam bath under vacuum to remove the alcohol and dimethylamine. The residue weighed 144 g. and was water soluble.

10 Cotton broadcloth of 132/60 construction is padded through a bath containing .25% by weight of the above dimethylamine salt of Asiatic wax sulfonic acid and dried in hot air. It has a very smooth surface and is more pliable than before the treatment. Other cotton fabrics such as muslins, sheetings, percales, twills, drills, etc., may be processed in a similar manner with baths containing from 0.1 to 0.5% of the softening agent to give them an enhanced "feel" when handled. Similarly, the product may be used in baths containing stiffening and weighting agents, such as starches, dextrans, gelatin, talc, china clay, etc., for sizing textile fabrics, wherein the product serves to lubricate and soften the effect of the stiffening and weighting agents giving the fabric a more desirable finish. The product may also be applied to textile fibers in the staple form before spinning to improve their processing characteristics and to yarns of staple and continuous filament fibers to lubricate them and improve their processing characteristics.

Example II

35 Five hundred grams of paraffin wax (commercial "parawax") was melted and a gaseous mixture of chlorine and sulfur dioxide in equimolecular proportions was passed in at 85 to 100° with an incandescent light under the flask. After 7 hours the gain in weight was 409 g. and on blowing with air, 4 g. of dissolved gases was blown out. The total chlorine passed through the reaction mass was 911 g. and the total sulfur dioxide was 1483 g. One hundred grams of the resulting product containing 23.03% Cl and 40 11.19% S was suspended in 200 cc. (95%) alcohol and trimethylamine passed in. When complete solution had occurred, the mass was allowed to stand for 1 hour and then the alcohol evaporated under vacuum on a steam bath. The yield was 140 g. When evaluated as a softening agent in a bath containing from 0.1 to 0.5% of the same, on viscose yarn, it showed softening action and on cotton broadcloth it also showed surface lubricating properties.

55 In place of the specific amines disclosed in the preceding examples may be substituted any aliphatic, aromatic, heterocyclic, aryl substituted aliphatic or mixed amines. As further examples of suitable amines, mention is made of 2-methyl piperidine, 1-ethyl piperidine, 1-n-butyl piperidine, n-butylamine, di-n-butylamine, cyclohexylamine, n-amylamine, di-isobutylamine, di-isopropylamine, piperidine, etc.

65 The initial compounds or compositions set forth in the above examples are only representative of many other compounds or compositions which could be used. Thus, the hydrolysis and neutralization products obtained by using a gaseous mixture of sulfur dioxide and chlorine according to the aforescribed process with any non-gaseous hydrocarbon, preferably saturated hydrocarbon, e. g. pentane, octane, nonane, decane, dodecane, octadecane, tetracosane, paraffin wax, scale wax, petrolatum wax, etc., can be resorted to. The neutralized products or salts

having a short chain length, e. g. 5 to 10 carbon atoms, are particularly useful as printing, mercerizing, etc., assistants. The products which have a chain length of 12 to 18 carbon atoms are particularly useful in conjunction with enzymes for starch removal, whereas those of longer chains such as the products obtained from wax distillates, match wax, paraffin wax, scale wax, petrolatum, petrolatum wax, etc., are particularly useful as textile softening, lubricating, 10 fulling, etc. agents.

In order to insure against the preparation of sulfonamides, some water must be present in the reaction zone. This may be accomplished by the use of ordinary alcohol containing 4% or more of water. Even traces of water cause some salt to be formed.

15 The temperatures are preferably kept below the boiling point of the reaction medium. High temperatures may be used, however, but they tend to promote amide formation.

Water-miscible organic solvents may be used during the hydrolysis or neutralization step to promote contact between the reaction mass and the hydrolyzing agents. As examples of such solvents or diluents may be mentioned ethyl, propyl, methyl, etc., alcohols, dioxane, glycol, and its ethers and esters, e. g. ethylene glycol diethyl ether, ethylene glycol dimethyl ethers, etc. The hydroxy compounds mentioned are particularly useful when amines are used as the hydrolyzing and neutralizing agents.

This invention possesses the advantage that fibers, etc. which are so treated are more acceptable to the trade. They are softer than untreated fibers and smooth to the touch and fuller. When white or bleached fabrics are so treated no discoloration obtains. The shade and light fastness of dyed materials is unchanged, which is surprising. The treatment also reduces static charges. This is more apparent at higher concentrations of the condensation product, but is noticeable with lesser amounts. These advantages are especially noticeable with viscose rayon both of the lustrous and delustered type.

45 As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope thereof, it is to be understood that we do not limit ourselves to the specific embodiments thereof except as defined in the following claims.

We claim:

1. A process which comprises treating a textile material with a solution of paraffin derivative soap obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous sulfur dioxide and chlorine, and hydrolyzing the resulting product with an organic nitrogenous base.

2. A process which comprises finishing and softening a textile material with a solution of a paraffin derivative soap obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous sulfur dioxide and chlorine, and hydrolyzing the resulting product with an organic amine base.

3. A process which comprises treating a cellulosic material with an aqueous solution of a paraffin derivative soap obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous sulfur dioxide and chlorine, and hydrolyzing the resulting product with a lower alkyl-amine base.

4. A process which comprises treating a textile material with a solution of a paraffin derivative soap obtainable by reacting a non-gaseous paraffin hydrocarbon with gaseous sulfur dioxide and chlorine, and hydrolyzing the resulting prod-

uct with a concentrated aqueous solution of a lower alkyl amine base.

5 5. The process of treating a textile material which comprises padding the same with a solution containing an organic amine salt obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous sulfur dioxide and gaseous chlorine and hydrolyzing the resulting product with an organic amine; and removing the excess solution.

10 6. A process as set forth in claim 5 wherein the non-gaseous paraffin hydrocarbon is paraffin wax.

15 7. The process of treating a textile material which comprises padding the same with a solution containing a lower alkyl amine salt obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous sulfur dioxide and gaseous chlorine and hydrolyzing the resulting product with a lower alkylamine in the presence of water and a lower alcohol; and removing the excess solution.

20 8. As new compositions the amine salts of the reaction products obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous chlorine and sulfur dioxide, hydrolyzing the resulting products and neutralizing them with an organic amine in the presence of an aliphatic alcohol.

30 9. As new compositions the lower alkylamine salts of the reaction products obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous chlorine and sulfur dioxide and chlorine and hydrolyzing the resulting products in the presence of a lower alkyl amine.

35 10. A textile treating solution for the softening and finishing of textile materials comprising an aqueous solution containing a paraffin derivative soap obtained by reacting a non-gaseous paraffin hydrocarbon with gaseous sulfur dioxide and chlorine and hydrolyzing the resulting product with an organic amine base.

40 11. A textile treating solution for the softening and finishing of textile materials comprising an aqueous solution containing a paraffin derivative soap obtained by reacting a non-gaseous paraffin with sulfur dioxide and chlorine and hydrolyzing the resulting product with a concentrated aqueous solution of a lower alkyl amine.

50 12. A textile treating solution for the softening and finishing of textile materials comprising an aqueous solution containing a paraffin derivative soap obtained by reacting a non-gaseous paraffin with sulfur dioxide and chlorine and hydrolyzing the resulting product with a concentrated aqueous solution of a lower alkyl amine.

ing and finishing of textile materials comprising an aqueous solution containing a paraffin derivative soap obtained by reacting a non-gaseous paraffin with sulfur dioxide and chlorine and hydrolyzing the resulting product with a concentrated aqueous solution of an organic amine.

13. A textile treating solution for the softening and finishing of textile materials comprising an aqueous solution containing a paraffin derivative soap obtained by reacting a non-gaseous paraffin with sulfur dioxide and chlorine and hydrolyzing the resulting product with a concentrated aqueous solution of a lower alkyl amine.

14. The process which comprises reacting a non-gaseous hydrocarbon with gaseous sulfur dioxide and chlorine and hydrolyzing the resulting product in the presence of an organic amine.

15. The process which comprises reacting a non-gaseous hydrocarbon with gaseous sulfur dioxide and chlorine and hydrolyzing the resulting product in the presence of a lower alkyl amine.

16. The process which comprises reacting a non-gaseous saturated hydrocarbon with gaseous sulfur dioxide and chlorine, adding a water soluble saturated aliphatic alcohol and passing into the mixture a readily volatile aliphatic amine at a temperature below 60° C.

17. The process which comprises reacting a non-gaseous saturated hydrocarbon with gaseous sulfur dioxide and chlorine, adding a water soluble saturated aliphatic alcohol and passing into the mixture a methyl amine at a temperature below 60° C.

18. The process which comprises reacting a non-gaseous saturated hydrocarbon with gaseous sulfur dioxide and chlorine, adding a water soluble saturated aliphatic alcohol and passing into the mixture dimethyl amine at a temperature below 60° C.

19. The products produced by the process of claim 16.

20. The products produced by the process of claim 17.

21. The products produced by the process of claim 18.

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