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METHOD OF DESULPHURIZING ARTIFICIAL FILAMENTS OF VISCOSE

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For desulphurizing artificial filaments of viscose formerly one has generally employed sodium sulphide and caustic soda. These desulphurizing agents have the disadvantage that the fibre swells to a large extent and consequently can be readily attacked both chemically and more particularly mechanically. If the whole of the after-treatment is to be carried out upon the bobbins then when using the said alkalies as desulphurizing agents only ceramic bobbins or bobbins of acid-proof steel can be used since these are the sole substances which are attacked neither by the spinning bath nor by the said alkalies.

In more recent times it has been preferred to use, instead of these, the alkali salts of weak acids. These have the same desulphurizing action as the said alkalies since alkali is hydrolytically split off in their aqueous solution. Owing to the smaller alkalinity, however, the fibre does not swell so highly, so that chemical attack on the filament, and more particularly mechanical injury to the fibre, cannot take place so readily. In addition, bobbins, vats and so forth are less attacked than when sodium sulphide and soda lye are used, although in view of the high temperature of the desulphurizing bath, the chemical attack is still very considerable.

According to the present invention one of the following substances is used for desulphurizing filaments, more particularly artificial silk, the working procedure being as usual, that is to say 1 to 2 hours treatment at 80° to 90° C., 1.5% to 2% solution being used.

(1) The salts of the sulphuric acid esters of high molecular alcohols, (general formula

$$R_1—CR_2R_3—O—SO_3—M,$$

where R_1 is a polyatomic residue, R_2 and R_3 are any desired organic residues or hydrogen, and M is a metal) e. g. the sodium salt of the sulphuric acid ester of octadecanol:

$$C_{17}H_{33}—CH_2—O—SO_3Na$$

such as has been introduced into commerce under the name "Gardinol" by the firm H. Th. Böhme, Chemnitz of Germany and the National Aniline and Chemical Company in the United States of America, or the scrooping agent "Gazetan CP" of the firm G. Zimmerli, Aarburg (Switzerland).

(2) The fatty acid esters of the low aliphatic oxysulphonic acid salts (general formulae

$$C_nH_{2n-1}—CO—O—Alk—SO_3M$$

and

$$C_nH_{2n+1}—CO—O—Alk—SO_3M)$$

e. g. the oleic acid ester of the sodium salt of oxyethyl sulphonic acid:

$$C_{17}H_{33}—CO—O—CH_2—CH_2—SO_3Na$$

such as is sold under the name "Igepon A" by the firm I. G. Farbenindustrie of Germany.

(3) The fatty acid amides of the low aliphatic aminosulphonic acid salts (general formulae

$$C_nH_{2n-1}—CO—NH—Alk—SO_3M$$

and

$$C_nH_{2n+1}—CO—NH—Alk—SO_3M)$$

e. g. the oleic acid amide of the sodium salt of aminoethyl sulphonic acids:

$$C_{17}H_{33}—CO—NH—CH_2—CH_2—SO_3Na$$

such as is retailed in Germany under the name "Igepon T", similarly by the firm I. G. Farbenindustrie of Germany.

(4) The neutral salts of the fatty acid amides of unsymmetrical dialkyldiaminoethane (general formulae

$$C_nH_{2n-1}—CO—NH—CH_2—CH_2—N\ (Alk)_2HX$$

and

$$C_nH_{2n+1}—CO—NH—CH_2—CH_2—N\ (Alk)_2HX)$$

e. g. the hydrochloric salt (or acetic acid salt, lactic acid salt, and so forth) of the oleic acid amide of unsymmetrical diethyldiaminoethane:

$$C_{17}H_{33}—CO—NH—CH_2—CH_2—N(C_2H_5)_2.HCl(CH_3COOH,\ CH_3—CHOH—COOH)$$

such as has been introduced into commerce under the names "sapamin CH", "sapamin A", "sapamin MS" and so forth, by the firm Gesellschaft für chemische Industrie of Basle.

In contradistinction to desulphurization with the alkali salts of weak acids, desulphurization with the said substances has the advantage that the substances have a completely neutral reaction in aqueous solution, so that scarcely any attack on the bobbins, vats and so forth can take place.

It is true that it has already been proposed to desulphurize with neutral substances, e. g. with alcohol vapours and alcoholic solutions of glycerin, grape sugar and cane sugar (U. S. Patent No. 1,651,404 and French Patent No. 655,729). These desulphurizing agents, however, have never come into practical use, already for the reason that extensive recovery plant is necessary for recovering the vaporized alcohol at the high temperatures necessary for effecting the desulphurization; a further reason is because of the danger of fire and finally, there is the much

too high cost of the alcohol as a solvent as compared with water.

The substances set forth by the applicant are well known to those skilled in the art as wetting and scrooping agents. Although it has already been proposed to use other scrooping agents, such as soap, "Turkey red oil" and "Marseilles soap" for desulphurizing purposes, no success could be obtained with these substances when operating on a practical scale. It is true that the chemical attack of these substances on the bobbins, vats and so forth is no greater than in the case of other alkali salts of weak acids; the dissolved metals, however, here form insoluble salts with the fatty acids and fatty acid derivatives which deposit on the filament in the form of smeary marks.

In contradistinction thereto, when desulphurizing with the said neutral salts there is practically no attack at all on the bobbins, vats and so forth, and inasmuch as traces of metal still occur in the desulphurizing agent they form soluble compounds with these which cannot contaminate the filament.

The advantages just referred to of the substances in question already appear, it is true, when they are used as scrooping agents so that it could perhaps be said that there is not much advance in making use of these advantages in desulphurization operations, once it has already been proposed to desulphurize with scrooping agents. It was not in any way to be foreseen, however, that it is just these modern scrooping agents which would be so suitable for desulphurization purposes. On the contrary, the expert would have to assume that they are unsuited for effecting desulphurization since, contrary to soap, "Turkey red oil" and "Marseilles soap," they are not hydrolytically split and therefore the known desulphurization by alkali action cannot be exerted at all. That these substances do not split off alkali in aqueous solution is well known to the expert and has been particularly emphasized by Bertsch, amongst others, in the "Zeitschrift für angewandte Chemie", 1931, No. 23, page 488; this property depends upon the fact that alkali salts of somewhat stronger acids, namely sulphonic acids, come into question.

Surprisingly enough it has been found that, nevertheless, the said substances act as desulphurizing agents. In the case of the substances in classes 1 to 3, the desulphurizing action is a property of the free sulphonic acids; in the case of substances coming in class 4, it is, for purposes of desulphurization, immaterial whether acid is present in the solution at all, whether just the quantity of acid necessary for forming salt is present, or whether there is excess of acid, and which acid is used for forming the salt. In order, however, to avoid attack on the bobbins, the vats and so forth, according to this invention, neutral desulphurizing baths are used in every case.

The invention also consists in this that after desulphurization with the said substances the washing out of the desulphurizing agent and the scrooping hitherto following thereon are omitted so that, therefore, the desulphurization agent, at the same time, assumes the task of the scrooping agent.

Instead therefore of the desulphurizing bath hitherto used, the two water baths following thereon and the scrooping bath, a single bath is employed which serves simultaneously for desulphurizing and scrooping purposes. In this way the operative procedure is substantially simplified and the water consumption, which, as is well known, is very big in the manufacture of artificial filaments, is considerably diminished, and also economy in plant costs is effected.

Over and above all this the fibre is subjected to a minimum of impairing influences on account of the small number of baths.

The known advantages of the said substances as scrooping agents come particularly to the fore when the substances are simultaneously used for desulphurizing purposes. More particularly the simultaneous desulphurization and scrooping with the same treatment baths is advantageous when the whole of the after-treatment is carried out on the bobbin or in the pot, since in such cases the purity of the treatment liquids is a matter of primary importance. The artificial filament after-treated according to the invention, is characterized not only by its cleanness, but also to an outstanding and pre-eminent extent by its softness and flexibility.

What I claim is:

1. In the after-treatment of artificial filaments of viscose the step of desulphurizing the filament by treatment with a neutral aqueous solution of at least one substance selected from the following group of neutral water-soluble wetting and scrooping agents of fatty character: salts of sulphuric acid esters of high molecular alcohols and the fatty acid esters of low aliphatic oxy-sulphonic acid salts.

2. In the after-treatment of artificial filaments of viscose the step of simultaneously desulphurizing and scrooping the filament by treatment with a neutral aqueous solution of at least one substance selected from the following group of neutral water-soluble wetting and scrooping agents of fatty character: salts of sulphuric acid esters of high molecular alcohols and the fatty acid esters of low aliphatic oxy-sulphonic acid salts.

3. In the after-treatment of artificial filaments of viscose the step of simultaneously desulphurizing and scrooping the filaments by treatment with a neutral aqueous solution of an agent of fatty character.

4. In the after treatment of artificial filaments of viscose, the step of desulphurizing the filaments by treatment with a neutral agent of the type of a fatty acid amid, which combines wetting and scrooping properties.

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