Our present invention relates to the manufacture of artificial silk from viscose and more particularly to the after-treatment and desulphurization.

One of its objects is to devise an improved process of physically desulphurizing artificial silk from viscose. Further objects will be seen from the detailed specification following hereafter.

In the manufacture of viscose artificial silk, for the purpose of removing sulphur and sulphur compounds present in the products of cellulose xanthate, it is necessary to subject the raw silk to a desulphurizing operation. It has hitherto been the custom in the industry to use, almost exclusively, aqueous solutions of sodium sulphide or sodium sulphite, that is to say substances capable of combining chemically with the sulphur. It has also been proposed to use instead of these substances, free alkali, salts of alkaline reaction, such as sodium carbonate, or substances which, like soap, are capable of yielding free alkali by hydrolysis. In the processes of the latter type, the desulphurizing action is due mainly to the fact that the free alkali or the alkali liberated by hydrolysis first of all converts a small part of the sulphur into sodium sulphide and this in its turn has a solvent action on sulphur.

This invention relates to a process of desulphurization in which sulphur is substantially completely removed, not by chemical means but by purely physical means by treating the viscose artificial silk with aqueous solutions or colloidal solutions of capillary active substances having an emulsifying action, that is to say, substances which have the capacity of reducing the surface tension of an aqueous solution. As the active solutions of these substances may have a neutral or even an acid reaction, the desulphurizing action is evidently due to a lowering of the surface tension of the water in respect of sulphur, which follows the addition of the said substances to such an extent that the material is thoroughly wetted, thus providing the preliminary conditions for an emulsification and detachment of the sulphur from the fiber. The substances which have been so far found to be useful are all those which are produced in the industry of textile assistants as well as by chemical means and are usually employed as such in the textile industry. Insofar as their chemical constitution is known approximately the following classes of substances may be mentioned as active substances in accordance with the present invention:

1. Acid esters of fatty acids with polybasic acids, for instance phthalic esters.

2. Higher fatty amines and their derivatives, for instance stearic amines, stearic pyridinium chloride, hydroxyethanolamines.

3. Fatty amines of aromatic aminosulphonated acids, for instance, laurylamphosphonic acid.

4. Alkylated and alkoxyated aromatic and hydroaromatic aminosulphonated acids, for instance, benzyl-naphthalenesulphonated acid, dissopropylbenzenesulphonated acid, or dialkylnaphthalenesulphonated acid, dodecyl-naphthalenesulphonated acid.

5. Fatty acid derivatives of the degradation products of albumen, such as lysobinonic acid and protaibinic acid.

6. True sulphonated fatty acids, for instance sulphonated palmitic acid.

7. Condensation products of fatty glicerides with naphthale sulphonated acids.

8. Cholic acid, glycocholic acid and naphthenic acid as well as derivatives and salts of these acids.

9. Lignine derivatives, such as sulphonated acids.

10. Sulphonated mineral oils, abietin sulphonates.

11. Semi-ethers of glycol and polyglycol with fatty alcohols.

12. Amides, anildes, alkylamides, naphthenilides of hydroxy fatty acids such as ricinoleic acid.

13. Ethanolamines, for instance, fatty acid ethanolamides and ethenolamines glycolized with ethylene oxide.

14. Esters of glicerine and polyglycine, or of glycol and polyglycol.

15. Lecithines.

The substances may be such which do not dissociate when dissolved in water or they may be salt-like compounds. According to their solubility they may be used in neutral, alkaline or even acid solutions. In order that the desulphurization may be rapid and uniform it has been found to be advantageous to use a concentration of these wetting and washing agents of 5 to 30 grams per liter, and to cause the solution to act upon the artificial silk preferably at a raised temperature, between 60 and 100°. The form in which the artificial silk is used is immaterial in this operation, it may be already dried or still quite fresh, that is to say only washed neutral; it may be subjected to this desulphurization in the form of a bank, a spool, a centrifugal cake, a continuous thread, a running band, or in the form of a finished textile material. Artificial products such as small bands and foils may also be desulphurized in this manner. As already stated, the efficacy of these substances is not confined to an alkaline reaction or to a pH value of more than 7, but the desulphuriza-
tion may be carried out under absolutely neutral conditions and also under weakly acid conditions, and if the weak alkaline reaction of certain classes of substances, such as, for example, fatty amines, is disturbing or is not desired, the reaction may be pushed as far as or beyond the neutral point by the addition of acid without loss of the desulphurizing action. On the other hand, however, there is no objection to the addition of these substances to the hitherto usual desulphurizing baths having a chemical action, for the purpose of improvement or acceleration. However, this possibility will not generally be considered because thereby the great technical advantage offered by the new process would remain unrealized. For instance, the wetting and washing agents mainly used for desulphurization will be those which, when the artificial silk is dried, can remain on the fiber and there serve as softening or preparing agents. In this manner the working-up process of the viscose artificial silk in this stage is shortened to two operations; namely to washing until neutral and removing sulphur, instead of the following sequence of operations hitherto used:—washing until neutral, desulphurization, washing, acidifying, washing until neutral and preparing. Consequently, according to the new process, operation under alkaline conditions comes in question practically only when, for certain purposes of preparation, it is advantageous to use a volatile alkali, such as ammonia, or a soap, such as Marseilles soap. If it is desired to enhance the effect of the preparation, it may be appropriate to incorporate with the treatment baths oils and fats, such as olive oil, paraffin oil and the like. It may be desirable to use a number of sulphur-removing agents simultaneously and in admixture with other substances; for example organic solvents or bleaching agents, such as hydrogen peroxide. In order to prevent a concentration of the sulphur products in the treatment bath, two baths may be used, the first bath being a repeatedly used circulating bath, while the second bath is clean and serves at the same time as a supplement and a refresher for the first bath. The following examples illustrate the invention:

**Example 1.** Undesulfurized viscose artificial silk in the form of bands is treated for 1/2 to 2 hours at 70 to 90° C. with a solution of 1 to 2 per cent. strength of the sodium salt of di-isopropylbenzene-Sulphonic acid. With this agent also a good desulphurization is obtained in neutral solution. If the pH-value is varied between 6.3 and 9 the result remains unchanged.

**Example 2.** In the solution referred to in Example 1, there is used a solution of approximately 2 per cent. strength of the sodium salt of di-isomethylbenzene-sulphonic acid. With this agent also a good desulfurization is obtained in neutral solution. If the pH-value is varied between 6.3 and 9 the result remains unchanged.

**Example 3.** Undesulfurized artificial silk is treated with a solution of 1 per cent. strength of an oxethylamide of a higher fatty acid, such as oleic acid, stearic acid or the like, for 2 hours at 80° C. The solution of this oxethylamide is, at neutral point, a stable emulsion; when the pH-value falls below the neutral point, as far as pH-value 5.1, oily separations occur, but the sulphur removal is not thereby impaired. Fatty acid oxethylamides which have been treated with ethylene oxide behave in a similar manner.

**Example 4.** Instead of the aforesaid products there is used an aqueous emulsion of 1.5 to 2 per cent. strength of a highly concentrated solution of an alkyl-substituted naphthalene-sulphonic acid in a terpene.

**Example 5.** Hanks of viscose artificial silk are first treated with a 2 per cent. solution of a polyglycol ether, for instance, oleyl polyglycol ether in accordance with Example 1 and then handled for a further 10 minutes in a solution of similar composition but with an addition of 0.05 per cent of H₂O₂ at 80° C. The fiber which is completely desulfurized after the first treatment becomes quite white after the second treatment. Instead of polyglycol ether there may also be used a polyglycol ester, for instance, polyglycol stearic acid ester.

**Example 6.** Hanks of artificial silk from viscose which have not yet been desulfurized are treated with a 1 to 2 per cent. solution of oleyl lecithine for about 2 hours at 70 to 90° C. After this treatment the hanks are completely desulfurized.

What we claim is:

1. A process of desulfurizing and after-treating viscose artificial silk, which comprises treating artificial silk from viscose with a 2 per cent. solution oleyl polyglycol ether for about 2 hours at about 70 to 90° C. and for about 10 minutes with a solution of similar composition but with an addition of 0.05 per cent of H₂O₂ at about 80° C.

2. A process of desulfurizing and after-treating viscose artificial silk, which comprises treating sulphur-containing artificial silk from viscose with a liquid dispersion of an alkylated aromatic sulphonic acid.

3. A process of desulfurizing and after-treating viscose artificial silk, which comprises treating sulphur-containing artificial silk from viscose with a liquid dispersion of a polyglycol ether.

4. A process of desulfurizing and after-treating viscose artificial silk, which comprises treating sulphur-containing artificial silk from viscose with a liquid dispersion of a polyglycol ether.

5. A process of desulfurizing and after-treating viscose artificial silk which comprises treating sulphur containing artificial silk from viscose with a slightly basic to neutral to acid aqueous dispersion of an organic substance having the property of reducing the surface tension of an aqueous solution which substance is capable of effecting removal of sulphur from the silk by a purely physical action.

6. A process of desulfurizing and after-treating viscose artificial silk which comprises treating sulphur containing artificial silk from viscose with a slightly basic to neutral to acid aqueous dispersion of an organic substance having the property of reducing the surface tension of an aqueous solution which substance is capable of effecting removal of sulphur from the silk by a purely physical action, the concentration of said substance in said dispersion being from about 5 to about 30 grams per liter.

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