## UNITED STATES PATENT OFFICE.

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## MANUFACTURE OF FILAMENTS AND FILMS FROM VISCOSE.

No. 849,823.

Specification of Letters Patent.

Patented April 9, 1907.

Application filed May 3, 1904. Serial No. 206,174.

To all whom it may concern:

Be it known that I, Charles N. Waite, of Lansdowne, in the county of Delaware and State of Pennsylvania, have invented certain new and useful Improvements in the Manufacture of Filaments and Films from Viscose, whereof the following is a specification.

My invention is especially valuable in the manufacture of the filaments commercially known as "artificial silk," and I would refer to Letters Patent of the United States No. 622,087, dated March 28, 1889, and No. 716,778, dated December 23, 1902, as containing descriptions of typical and convenient methods of obtaining such filaments.

In processes of the character contemplated viscose (cellulose sulfocarbonate) is forced through spinnerets into a bath capable of gelatinizing the viscose, whereby the filaments thus produced are set in the form

given to them by the spinnerets.

Heretofore either an ammonium salt—such as ammonium chlorid or ammonium sulfid or sulfuric acid—has been employed as the efficient agent of the setting-bath. I

have found that when either ammonium salt aforesaid is used ammonium sulfid is liberated in large quantities and sulfur effloresces on the filaments, impairing their luster and rendering their color objectionable. I have also found that if sulfuric acid is used in aqueous solution hydrogen sulfid is liberated, with the disadvantages which are due to the presence of that poisonous gas, and

sulfur is also deposited in the filaments. The usual method of finally treating such products intended to eliminate any remaining sulfur and sodium hydrate is to immerse them in a saline solution and then to immerse them in a dilute acid. However, I find that notwithstanding such treatment the products are liable to retain sodium hydrate in quantity sufficient to partially redissolve the regenerated viscose filaments,

dissolve the regenerated viscose filaments, and thus impair their luster, and the residual sulfur effloresces on such filaments and impairs their color.

My invention is intended to avoid the objectionable features aforesaid, and to that

end I proceed as follows:

I employ for the setting solution a bath consisting of sodium bisulfite, (NaHSO<sub>3</sub>,) into which the filaments or films are pro-

jected, with the result that the sodium hy- 55 drate in the viscose is neutralized and the sulfur is converted into a modification soluble in water, viz: sodium hyposulfite.

For the purpose of dehydrating the filaments and rendering them firmer and less 60 liable to abrasion during the subsequent finishing process than if treated solely with the setting-bath of sodium bisulfite aforesaid I find it advantageous to add a saline solution to said bath. For instance, I make a bath 65 of saturated aqueous solution of acid sodium sulfite containing from eleven and onehalf to twelve per cent. of available sulfurous acid and to this add ten per cent. of its weight of a saturated solution of ammonium 70 sulfate sodium chlorid or other similar dehydrating agent. The setting-bath aforesaid should preferably be heated to about 60° centigrade, and after treatment therein I subject said products to the action of steam 75 in the presence of the residual sulfurous acid, the steaming process being continued for from one to two hours, according to the thickness of the products. The products may advantageously be placed upon tension 80 during the steaming process, and after steaming it is only necessary to wash said products and dry them.

The reactions due to the treatment above described may be assumed to be as follows: 85 The viscose compound is decomposed by heat. The sulfur contained therein is transformed into sodium hyposulfite, and the sodium hydrate resulting from the decomposition of the cellulose sodium xanthate is neu-90 tralized by the sulfurous acid. The ammonium sulfate or sodium chlorid added as aforesaid has also a similar neutralizing action.

The process above described is particularly valuable in the production of very fine 95 filaments of lustra-cellulose or artificial silk, where a number of such filaments are to be spun together to form composite threads. In this case any residual sodium hydrate tends to redissolve the surfaces of the individual roo filaments and agglomerate them, impairing the luster, softness, and pliability of the composite threads.

Having thus described my invention, I claim—

1. The hereinbefore-described process of making viscose products, which consists in subjecting liquid viscose to the action of a so-

lution comprising sodium bisulfite; and, then eliminating such residual constituents as sulfur and sodium hydrate, by steaming said products in the presence of sulfurous acid,

5 substantially as set forth.

2. The hereinbefore-described process of making viscose products, which consists in subjecting liquid viscose to the action of a solution comprising sodium bisulfite; then 10 eliminating such residual constituents as sulfur and sodium hydrate, by steaming said Pennsylvania, this 2d day of May, 1904. products in the presence of sulfurous acid; and, subjecting said products to tension during the steaming process, substantally as set 15 forth.

3. The hereinbefore-described process of

making viscose products which consists in initially subjecting liquid viscose to the action of a coagulating solution comprising sodium bisulfite; and, then eliminating such 20 residual constituents as sulfur and sodium hydrate, by steaming said products in the presence of sulfurous acid, substantially as set forth.

In witness whereof I have hereunto signed 25 my name, at Philadelphia, in the State of

CHARLES N. WAITE.

Witnesses:

ARTHUR E. PAIGE, ANNA F. GETZFREAD.