

UNITED STATES PATENT OFFICE.

BENNO BORZYKOWSKI, OF CLEVELAND, OHIO.

PROCESS FOR THE PRODUCTION OF ARTICLES FROM VISCOSE.

1,260,508.

Specification of Letters Patent.

Patented Mar. 26, 1918.

No Drawing.

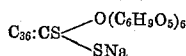
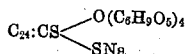
Application filed May 29, 1917. Serial No. 171,793.

To all whom it may concern:

Be it known that I, BENNO BORZYKOWSKI, a citizen of Russia, temporarily residing at Cleveland, in the county of Cuyahoga and State of Ohio, have invented a certain new and useful Improvement in Processes for the Production of Articles from Viscose, of which the following is a specification.

My present invention pertains to an improved process for the production of articles from viscose, and has for its object the production of articles directly from viscose in the fresh, unmaturred and unpurified state.

According to the literature and patents relating to this art (*e. g.* German Patent No. 187,369) the production of artificial articles from viscose requires a viscose solution which, by having been stored for some time, or by heating, or the like, has reached a certain state of maturity, or which has passed over into the xanthogenate



respectively, and it also requires conversion of the crude viscose into a so-called "purified" viscose before being transformed into such articles.

The difficulty which is encountered in connection with maturing or purifying and in the subsequent treatment of viscose which has undergone such preliminary treatment, is well known from practical experience to everyone skilled in the art, and it is also known that variable results are attained in the final product by the process as now carried out.

The subject of this invention pertains to a process for directly converting fresh (unmaturred or only slightly maturred) and unpurified viscose in a simple and inexpensive manner into artificial articles, such as lustrous and supple threads, and the invention is based on the observation that even wholly unmaturred viscose masses (hence immediately after solution and filtration of the same) may be converted into most excellent cellulose hydrate or cellulose xanthogenate products by using for the former very dilute acids, and for the latter highly saturated salt baths, but in both cases with the longest possible distance for precipitation.

This observation is surprising, inasmuch as it has hitherto been assumed (see, for

example, U. S. Patent No. 725,016, or British Patent No. 2529 of 1902) that only purified viscose can be precipitated by weak acids, while crude viscose is not precipitated by an excess of, *e. g.*, acetic acid, and according to U. S. Patent No. 716,778 viscose must be maturred for seven days at 15.5° C., in order to form threads from the same by precipitation in salt baths.

It is furthermore surprising, and in this respect of great value from the technical standpoint, that by the present process serviceable results are attained, although only slightly acidified water is used for the setting bath, while the material can be forced out at the highest possible rate of speed. For instance, while a normally prepared fresh viscose mass, in a 5% sulfuric acid precipitating bath and with a rate of take-up speed of 40 meters per minute, yields even with the shortest practicable distance for precipitation, 3 cm., only turbid threads which become brittle when dried, this same mass, in a sulfuric acid precipitating bath of only 2.5 to 3% strength and a distance for precipitation of 25 to 30 cm., at a rate of take-up speed of 40 meters a minute, yields excellent threads, clear as glass, even in the wet state are relatively firm and remain soft and supple after drying. In such a bath, containing less than 5% of sulfuric acid, a distance of at least 10 cm. is required for continuous coagulation. In this manner it is possible to obtain directly cellulose hydrate threads with high power of production and with cheap precipitants.

I have also found that, contrary to the opinion advanced, (see U. S. Patent No. 716,778), it is possible to produce excellent cellulose xanthogenate articles from fresh unpurified viscose in the first stages of maturity. It is true, however, that with fresh viscose masses, even with a relatively slow discharge of 20 meters a minute, which can hardly be considered for practical purposes, it is not possible to produce artificial threads on a commercial scale with the known "mild" salt baths which, according to the art, must not have a specific gravity in excess of 18 or 19° Bé. But when for a fresh viscose mass there is used according to my process a salt bath of at least 20°, and preferably 23 or 25° Bé. and a distance of at least 25 cm. for precipitation, and preferably of 40 to 60 cm., it becomes possible to work continuously at a rate of discharge of 40 or more meters a minute. The products (*e. g.* threads) ob-

tained from such a setting bath do not have the yellowish-white appearance of the cellulose hydrate threads as obtained from precipitation in acids, but have the typical character of cellulose xanthogenate, are elastic and transparent in wet (coagulated) state, and after final treatment with acid, etc., and drying yield supple threads of high luster which crackle like silk. The woody feel and dull appearance of the commercial viscose threads are entirely obviated by the present process, the defect referred to being due chiefly to the fact that the viscose mass has heretofore been aged to such a degree that even the mildest precipitants (the weakest concentration of the baths, and shortest passage through the setting bath) have attacked the materials too much and rendered them brittle.

As setting baths for this process any of the known baths may be employed, and, generally speaking, any precipitants, singly or mixed together, which, with a specific gravity of at least 20° Bé. and above, are capable of being maintained in solution. The choice of the proper precipitants, and of the temperature depends entirely on the character and form of the article to be produced. For the production of artificial silk a bath may be used, which consists of one part (by weight) of sodium sulfate (Glauber salt) and one part (by weight) of ammonium sulfate diluted with such an amount of water that the solution will be at least of 22° Bé. It is advisable to add about 2 to 3% (computed on the whole amount of the bath) of hydrochloric acid or sulfuric acid in order to prevent neutralization of the setting bath by the alkali of the viscose mass. The quantity of acid used should in no case exceed 10% sulfuric acid, or its equivalent. Instead of the acid, a readily dissociating acid compound, *e. g.*, aluminum sulfate, may be employed.

The temperature of the bath may be between 35 and 50° C. The present process does not in the least exclude the use of other substances in the baths, such as alcohols, metallic salts or organic substances, more particularly the organic nitroso bases described in my U. S. Patent No. 1,143,569, whereby the deposition of free sulfur on the surface of the threads is prevented, and the capillary threads are prevented from sticking together.

Having thus described my invention, what I claim is:

1. The process of producing articles from

fresh viscose solutions, which consists in passing the same through an extended path in a precipitating bath.

2. The process of producing articles from fresh viscose solutions, which consists in passing the same through an extended path in a dilute acid precipitating bath.

3. The process of producing articles from fresh viscose solutions, which consists in passing the same through such a diluted acid precipitating bath that at least a path of 10 centimeters is required for a continuous coagulation.

4. The process of producing articles from fresh viscose solutions, which consists in passing the same through a path longer than 10 centimeters in an acid precipitating bath having a concentration less than 5% of sulfuric acid.

5. The process of producing articles from fresh viscose solutions, which consists in passing the same through a path of at least 20 centimeters in an acid precipitating bath containing from 2.5 to 3% of sulfuric acid.

6. The process of producing articles from fresh viscose solutions, which consists in passing the same through a precipitating bath of concentration greater than 18° Bé.

7. The process of producing articles from fresh viscose solutions, which consists in passing the same through a path longer than 25 centimeters in a precipitating solution.

8. The process of producing articles from fresh viscose solutions, which consists in passing the same through a path longer than 25 centimeters in a precipitating bath of concentration greater than 18° Bé.

9. The process of producing articles from fresh viscose solutions, which consists in passing the same through a path of 40 to 60 centimeters length in a precipitating solution of a concentration of from 20 to 30° Bé.

10. The process of producing articles from fresh viscose solutions, which consists in passing the same through an extended path in a solution having a concentration greater than 18° Bé. and containing less than 10% of sulfuric acid or the equivalent amount of other acids.

11. The process according to claim 1, having in the bath a substance which acts to prevent the deposition of sulfur on the surface of the threads, and also the sticking together of the fibers.

In testimony whereof I have signed my name to this specification.

BENNO BORZYKOWSKI.