

UNITED STATES PATENT OFFICE

2,566,457

MANUFACTURE OF VISCOSE PRODUCTS

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No Drawing. Application December 21, 1950, Se-
rial No. 202,150. In the Netherlands January
11, 1950

4 Claims. (Cl. 18-54)

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This invention relates to a continuous process for the manufacture of viscose products, and more particularly to a process for the continuous aftertreatment of freshly formed viscose threads traveling at high speeds in a substantially linear direction through successive treating baths, wherein the decomposition of cellulose xanthate is carried out in a more economical manner.

In the continuous manufacture of viscose products such as threads, fibers, films, and the like, designated generically as threads, the freshly spun threads are drawn from an acid coagulating bath, passed through various liquid treating stages, and then either dried and collected or collected wet.

One method of passing the threads through the various liquid treating zones is that of the so-called "sheet" method, as described in U. S. Patent No. 2,334,325, assigned to the same assignee as this present application, in which a plurality of closely spaced parallel threads, in the form of a moving warp or sheet are conducted through a succession of trough-shaped receptacles containing the appropriate treating liquids. In this process the problem arises of effecting the necessary decomposition of the cellulose xanthate still remaining in the freshly spun threads to such an extent that subsequent desulfuring of the threads may be accomplished properly. This xanthate decomposition is ordinarily a slow process, but it has been found possible to reduce the reaction time considerably through the use of special treating baths as described in detail in U. S. application Serial No. 81,844, filed March 16, 1949, assigned to the same assignee as this application.

In the process according to this application, a thread that has been freshly spun in a normal spinbath is exposed to a decomposing bath at a temperature in the range of 60° to 95° C., containing both H_2SO_4 and Na_2SO_4 in about the same molecular ratio as they exist in the spinning bath (i. e., the molecular ratio of H_2SO_4 to neutral Na_2SO_4 less than 1). In this bath the xanthate ratio is reduced to 0.02. (The xanthate ratio, i. e., the ratio of the number of residual xanthate groups to the total number of cellulose groups ($\text{C}_6\text{H}_{10}\text{O}_5$) is determined by any of the well known chemical methods. One suitable determinative method is described in Leroy H. Smith, Synthetic Fiber Developments in Germany 1946, pages 201-204.)

After washing to remove the salts from the thread, a second treatment can then be carried out in a dilute H_2SO_4 bath which contains

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Na_2SO_4 substantially below the "bisulfate ratio," i. e., for every mol of H_2SO_4 , less than 1 mol of sodium sulfate is used. At the end of this second treatment, the xanthate ratio is reduced to less than 0.003, and preferably under 0.002. After these two treatments, desulfuring can take place very satisfactorily with the result that there is not enough residual sulfur in the finished thread to give rise to any undesirable yellow discolorations, which would be detrimental in the finished product.

The above values of xanthate ratio occur when the time of thread travel through each bath is 3 or 4 seconds, and the bath lengths are 3 to 5 meters for thread speeds of 60 to 100 meters per minute. Furthermore, the baths, especially the second, are at high temperatures and are exposed to the atmosphere, and accordingly there is a high rate of heat loss from the surface of the baths which incurs the disadvantage of high rate of steam consumption.

It is therefore an object of this invention to reduce this high steam consumption by providing an improved process for the continuous manufacture of viscose threads by passing a sheet of parallel threads at high speed in a substantially straight direction through a succession of treating baths whereby the decomposition of the cellulose xanthate is accomplished in a more economical manner than heretofore has been possible.

It is contemplated, according to this invention to first decompose the freshly spun threads to a xanthate ratio of 0.10, second, to load the threads with substantially pure dilute H_2SSO_4 , and third to subject the loaded threads to the action of steam until the xanthate ratio is below 0.003.

Other advantages and objects of this invention will become apparent from the following description.

In this present process, the first treatment of the freshly spun threads is similar to that described in the above mentioned application Serial No. 81,844 and comprises passing the threads through a decomposing bath containing both H_2SO_4 and Na_2SO_4 in approximately the same molecular ratio that they are contained in the spinning bath, i. e., with the molecular ratio of neutral Na_2SO_4 to H_2SO_4 greater than 1:1. The acid concentrations in this first decomposing bath lie between 0.5% and 6.0%, while the corresponding Na_2SO_4 concentrations lie between 1.0% and 10%. This bath is kept at a temperature between 60° and 95° C., which is the

same temperature range as for the earlier process. The threads are exposed to the action of this decomposing bath only for the time necessary (less than one second) to reduce the xanthate ratio to a value of 0.10, as contrasted to the value of 0.02 required in the earlier process. This permits the use of a treating bath which is markedly shorter in length. The sodium sulfate entrained on the threads is then washed out by means of a salt-free solution and this step is parallel to that of the earlier process.

The salt-free threads are next passed very briefly through a so-called "loading" bath, containing substantially pure dilute acid, which is at a temperature between room temperature and about 60° C. The distance of travel through this loading bath can be as short as 50 cm., even when thread speeds of 100 meters per minute and higher are employed, thus giving a treatment time of less than one second. The concentration of the acid is determined by the total thread denier, the thread filament denier, and the retention of acid per unit length of thread. For example, if 100 denier threads of 40 filaments are being treated, a 1% solution is sufficient. Sodium sulfate may be present in this loading bath, but only if its molecular concentration is less than that of the acid. It is a prerequisite that the Na_2SO_4 molecular concentration be less than that of the acid in order for the process to be commercially satisfactory.

The "loaded" threads are then withdrawn from this loading bath and conducted through a tunnel or chamber where they are subjected to the action of saturated steam for the time necessary to reduce the xanthate ratio to 0.003 or lower. A time of three seconds for normal textile threads, and slightly longer for the heavier threads (1100 denier and higher) is generally sufficient. At thread speeds of 60 to 100 meters per minute, this necessitates a steam chamber of 3 to 5 meters in length as a minimum, and at most 4 to 6 meters in length.

Following the steam treatment, the threads can be washed with water only, which gives threads containing 0.04% sulfur, after which normal bleaching and finishing operations can be carried out. For this operation, the temperature of the water should be over 40° C. For many purposes, this percentage of sulfur is not troublesome in the final product. For lower values of sulfur, a desulfuring treatment with any of the well known desulfuring solutions (i. e., sodium sulfide) can be given prior to the bleaching and finishing, in which case it is possible to use a shorter bath travel through the desulfurant after the various liquid treatments, the thread can be collected wet in package form, or else dried as a running thread by any suitable means.

This process is especially suitable for the treatment of a moving sheet, or warp of a plurality of closely spaced parallel threads, which can be passed in succession through the various treating baths and then through slit-shaped steam chamber, the dimensions of which are adapted to the outer dimensions of the sheet of threads. In this manner, the individual threads of the moving sheet receive a very even and uniform heating effect in the steam chamber.

It can be seen that one of the most important advantages in the process of this invention resides in the saving of steam. Since the first decomposing bath is shorter than normal, less steam is required. Furthermore, the second loading bath may be kept at room temperature and may

be as short as 50 cm., which contrasts with the long second decomposing bath at an elevated temperature of earlier processes and represents a substantial steam saving. Further, since most of the sulfur can be removed from the thread by water, a shorter desulfuring bath can be used. Since the desulfuring bath is heated by steam, this results in a still further steam economy.

Offsetting this steam economy is the steam required in the steaming chamber, but when this chamber is properly designed so that a plurality of threads can enter and leave through narrow slits, an overall steam saving, as compared to the former method, is obtained which amounts to 30% to 60%.

A further advantage of this invention is the improvement obtained in the desulfuring and bleaching resulting from the effective decomposition of the cellulose xanthate.

One embodiment of the process of this invention can be illustrated by the following example:

Example

With the aid of a spinning machine, for example such as described in U. S. application Serial No. 182,534, filed August 31, 1950, and assigned to the same assignee as this application, 60 threads of 100 denier and 40 single filaments per thread were spun in a normal coagulating bath. Thereupon these threads were drawn off as a sheet of threads, with a mutual thread distance of 7 mm. The sheet of threads, withdrawn at a speed of 60 meters per minute, was guided through a decomposing bath at 80° C., which contained 4% H_2SO_4 and in which the ratio between acid and salts were equal to that of the spinning bath. The sheet of threads was stretched in this bath about 50% by means of a draw-off roller, which had a peripheral speed of about 90 meters per minute. The stretched sheet of threads was guided through a water bath of 20° C. with a speed of about 90 meters per minute and thereupon through a substantially pure 1% H_2SO_4 solution at 25° C.

The sheet of threads, which had been thereby partially decomposed, washed and reloaded with pure acid, was guided through a slit-shaped tunnel 5 meters in length and during the passage through the tunnel, the sheet was exposed to the action of saturated steam. The sheet was then washed with water. At this point the sulfur content of the thread amounted to 0.04%. The product was finally bleached and finished. As an alternative, the sheet of threads was treated to remove a portion of the water, for example, by means of an expressing operation between squeezing rollers, and then dried. Finally, the individual threads of the sheet were collected by winding.

To give a final product with lower sulfur content, a treatment in a normal desulfuring bath was carried out before bleaching. As a variation, the product was squeezed out, for example, by means of squeezing rollers. After this it was dried and finally the individual threads of the sheet were wound in the twisted state.

The invention is not limited to normal rayon such as 2.5 and 5 filament denier, but is also applicable to the preparation of thicker products made from viscose such as horsehair, ribbons, and films, and when the expression "threads" is used in the claims it is intended to cover these other said products.

Obviously, various modifications may be made

without departing from the spirit and scope of the invention.

What is claimed is:

1. A process for the continuous manufacture of viscose threads which comprises extruding a multiplicity of threads through spinnerets into a coagulating bath containing sulphuric acid and salts thereof, passing the threads in a parallel relationship linearly through a second bath of such length that the speed at which the thread passes through the bath limits the time of passage to less than one second, thereby lowering the xanthate ratio to about 0.10, which is insufficient to permit subsequent complete desulphurization, said second bath being maintained at a temperature in the range of 60° C.-95° C. and containing a lower concentration of acid and salt than that of the first bath, washing the threads free of salts, treating the threads for less than one second with a dilute sulphuric acid solution maintained between room temperature and 60° C., decomposing the remaining xanthate in the threads to a xanthate ratio below 0.003 by exposing them to the action of steam, and finally removing all of the sulphur except about 0.04% by washing the threads in water above 40° C.

2. A process for the continuous manufacture of viscose threads which comprises extruding a multiplicity of threads through spinnerets into a coagulating bath containing sulphuric acid and salts thereof, passing the threads in a parallel relationship linearly through a second bath of such length that the speed at which the thread passes through the bath limits the time of passage to less than one second, thereby lowering the xanthate ratio to about 0.10, which is insufficient to permit subsequent complete desulphurization, said second bath being maintained at a temperature of about 80° C. and containing 4% sulphuric acid, washing the threads free of salts, treating the threads for less than one second with a 1% sulphuric acid solution maintained at about 25° C., decomposing the remaining xanthate in the threads to a xanthate ratio below 0.003 by exposing them to the action of saturated steam, and finally removing all of the sulphur except about 0.04% by washing the threads in water above 40° C.

3. A process for the continuous manufacture of viscose threads which comprises extruding a multiplicity of threads through spinnerets into a coagulating bath containing sulphuric acid and salts thereof, passing the threads in a parallel relationship linearly through a second bath of such length that the speed at which the thread passes through the bath limits the time of passage to less than one second, thereby lowering the xanthate ratio to about 0.10, which is insufficient to permit subsequent complete desulphurization, said second bath being maintained at a temperature in the range of 60° C.-95° C., and containing a lower concentration of acid and salt than that of the first bath, washing the threads free of salts, treating the threads for less than one second with a dilute sulphuric acid solution maintained between room temperature and 60° C., decomposing the remaining xanthate in the threads to a xanthate ratio below 0.003 by exposing them to the action of steam, again washing the threads and effecting substantially complete desulphurization.

4. A process for the continuous manufacture of viscose threads which comprises extruding a multiplicity of threads through spinnerets into a coagulating bath containing sulphuric acid and salts thereof, passing the threads in a parallel relationship linearly through a second bath of such length that the speed at which the thread passes through the bath limits the time of passage to less than one second, thereby lowering the xanthate ratio to about 0.10, which is insufficient to permit subsequent complete desulphurization, said second bath being maintained at a temperature of about 80° C. and containing 4% sulphuric acid, washing the threads free of salts, treating the threads for less than one second with a 1% sulphuric acid solution maintained at about 25° C., decomposing the remaining xanthate in the threads to a xanthate ratio below 0.003 by exposing them to the action of saturated steam, again washing the threads and effecting substantially complete desulphurization.

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