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PROCESS OF PRODUCING VISCOSE RAYON

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This invention relates to the production of viscose rayon of especially high tensile strength. It has been proposed to produce threads of high tensile strength in the normal spinning baths, for instance in the so-called Müller baths, which may contain zinc sulfate or other bivalent metal salts, by strongly stretching the freshly spun threads in hot neutral or acid baths.

It has further been proposed previously to coagulate viscose in precipitating baths of low sulfuric acid content, for instance in baths containing no more than 7 per cent sulfuric acid. The coagulation leads to cellulose xanthate threads which are subsequently stretched by 25 and more, for instance 100 per cent, in a second bath which contains an alkaline swelling agent.

It is an object of the present invention to provide a process which is simpler and more easy to carry into effect, and which moreover allows to recover the valuable metal salts.

Further objects of the invention will appear from the following detailed description.

We have found that unexpectedly, better results are obtained by using as a spinning bath the normal Müller bath, which may contain sulfuric acid and salts, leading the freshly spun threads into alkaline solutions containing zinc or aluminum, for instance solutions of zinc hydroxide in ammonia or of aluminum hydroxide in caustic soda lye and subjecting the threads thus treated to strong stretching. The alkaline treating baths containing zinc may be produced by adding to a solution of zinc sulfate in water enough ammonia to redissolve the zinc hydroxide which precipitates at first. The zinc hydroxide precipitated from a solution of a zinc salt may also be separated from the solution and dissolved in dilute ammonia. In the same manner alkaline solutions of aluminum hydroxide may be produced from aluminum salt solutions by means of caustic alkalies. The acid concentration of the spinning baths may be kept under or above 7 per cent sulfuric acid. Before the thread enters the alkaline treating bath the adhering acid is removed as well as possible. If desired a short intermediate treatment with a washing bath, for instance a sodium sulfate solution may be interposed between the spinning bath and the zinc bath.

The swelling action of the zinc or aluminum bath enables the thread to be easily stretched to 50 and even 100 per cent and more in excess of the original length of the thread coagulated in the acid precipitating bath. The stretching can be effected in the alkaline zinc or aluminum bath itself or after leaving this bath during the passage of the thread through air, or in a third bath consisting of hot water. Instead of the hot water bath steam may be applied of about 100° C. In producing endless

threads the stretched threads are collected on suitable receiving devices for instance reels or spools or spinning pots and simultaneously treated with dilute acids to finally fix them. The final acidification may also be applied to the running thread which latter measure is especially useful in producing staple fibers.

The threads obtained according to the process of the present invention possess mechanical properties which equal those of cotton. The tensile strength in the dry state of more than 3 or 3.5 g. per denier and in the wet state of more than 2.2 and even 3 g. per denier combined with an extensibility of 15 to 20 per cent can be easily obtained.

The above described method for the production of these threads can be carried out on a technical scale without any difficulties. The process is superior to those hitherto known of producing threads of like mechanical constants by its simplicity.

The invention is illustrated by the following examples.

Example 1

A viscose produced from alkali cellulose ripened 60 hours at 20° C. which viscose has a content of 8 per cent cellulose and 7.5 per cent alkali and has a spinning ripeness of 8.3 common salt number and a viscosity of 27.5 poises is spun at 40° C. into a bath containing 30 per cent Na_2SO_4 and 12 per cent H_2SO_4 . A spinneret of 600 pores of a diameter of 0.07 mm. is employed. The feed corresponds to a titer of 1.5 denier. The spinning speed being 50 m. of the stretched thread per minute. The length of bath-travel in the precipitating baths is 18 ccm. Immediately after leaving the precipitating bath the thread is led through an ammoniacal zinc bath containing 6 per cent zinc and 20 per cent ammonia and kept at 20° C. The thread emerging from the alkaline bath is subsequently stretched in a hot water bath or in an atmosphere of steam by about 100 per cent of its original length. The stretched thread is finally acidified with sulfuric acid of 4 per cent acid content in stretched condition and wound on spools. The dilute acid can also be used as an additional bath in which the spool turns. Even if the thread wound on spools is cut and after-treated as staple fiber the constants are very satisfactory:

Dry tenacity	3.7 g. per denier
Wet tenacity	2.7 g. per denier
Extensibility	15 to 18 per cent

Example 2

Alkali cellulose produced from a high grade sulfite pulp and ripened at 20° C. for 20 hours is dissolved to yield a viscose containing 6.5 per cent cellulose and 8.5 per cent alkali. The vis-

cose which has a common salt number of 7 to 8 and a viscosity of 191 poises is spun as in Example 1 in a Müller bath. After leaving the precipitating bath the thread is led by means of rolls through an alkaline aluminum bath containing 2 per cent aluminum and 6 per cent sodium hydroxide (produced from aluminum sulfate solution and caustic soda lye). The thread is subsequently stretched as strongly as possible in a steam atmosphere at 100° C. and is led onto a reel on which it is acidified with sulfuric acid of 3 per cent strength.

Dry tenacity----- 3.5 g per denier
Wet tenacity----- 2.7 g per denier
Extensibility----- 15 per cent

Example 3

Alkali cellulose produced from a high grade beechwood-pulp is ripened at 20° C. for 20 hours and a viscose is prepared therefrom containing 8 per cent cellulose and 7.5 per cent alkali. The viscose is spun at 45° in a bath containing 10 per cent sulfuric acid, 7 per cent zinc sulfate and 18 per cent sodium sulfate, and the thread is subsequently led through an alkaline zinc bath which was obtained by adding ammonia to a zinc sulfate solution and which contains 6 per cent zinc and 20 per cent ammonia. The thread is subsequently stretched in a steam atmosphere at 100° C. by about 100 per cent of its original length and a great number of such threads is collected into a strong fiber bundle. After acidifying with sulfuric acid of 5 per cent strength at room temperature the fiber bundle is led through the usual after-treating baths and finally continuously cut to staple lengths.

Dry tenacity----- 3.8 g per denier
Wet tenacity----- 2.6 g per denier
Extensibility----- 16 per cent

We claim:

1. A process of producing viscose rayon of high tensile strength which comprises spinning threads in a bath containing sulfuric acid and an alkali metal sulfate, leading the threads thus formed through an alkaline bath containing in solution a metal hydroxide selected from the group consisting of zinc and aluminum hydroxides, said bath having a swelling action on said threads and stretching said threads to exceed the original length of the threads leaving the spinning bath by at least 25 per cent.

2. A process of producing viscose rayon of high tensile strength which comprises spinning threads in a bath containing sulfuric acid and an alkali metal sulfate, leading the threads thus formed through an alkaline bath containing zinc hydroxide in solution, said bath having a swelling action on said threads, and stretching the swollen threads in the swelling bath to exceed their original length by at least 25%.

3. A process of producing viscose rayon of high tensile strength which comprises spinning threads in a bath containing sulfuric acid and an alkali metal sulfate, leading the threads thus formed through an alkaline bath containing zinc hydroxide in solution, said bath having a swelling action on said threads and stretching the swollen threads in hot water to exceed their original length by at least 25%.

4. A process of producing viscose rayon of high tensile strength which comprises spinning threads in a bath containing sulfuric acid and an alkali metal sulfate leading the threads thus formed through an alkaline bath containing zinc hydroxide in solution, said bath having a swelling action on said threads, and stretching the swollen threads in hot steam to exceed their original length by at least 25%.

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