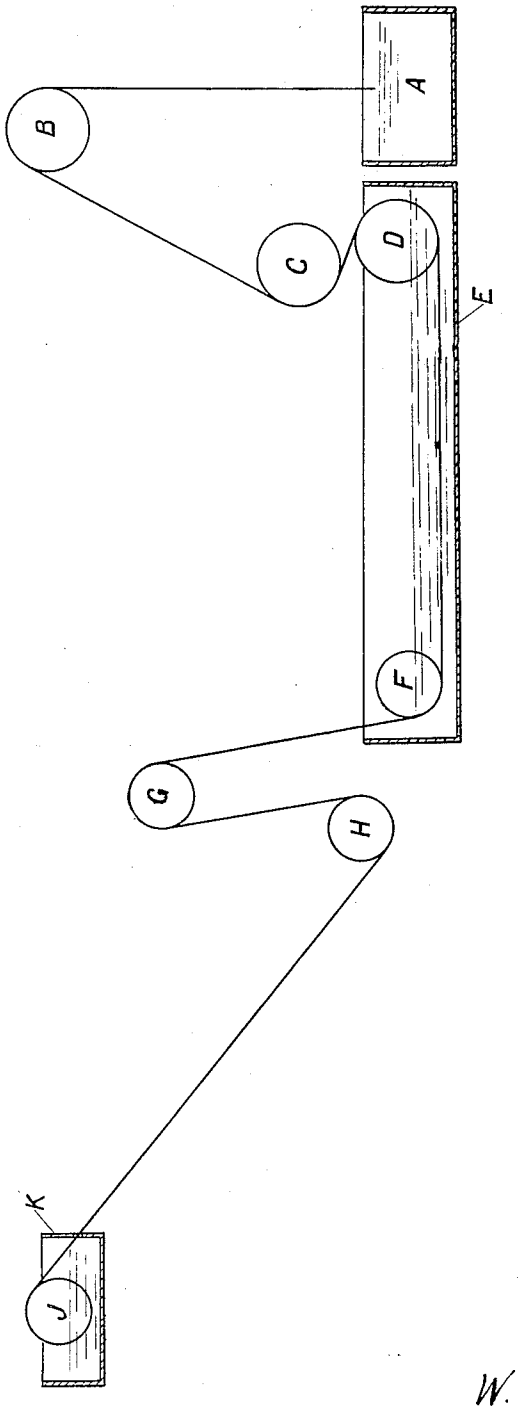
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MANUFACTURE OF ARTIFICIAL FILAMENTS

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MANUFACTURE OF ARTIFICIAL FILAMENTS

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It is known to spin viscose by what is known as the stretch spinning process according to which the formed threads are drawn away from the jet orifices at a greater velocity than that at which the viscose solution issues from the orifices.

It is also known to produce artificial silk of higher strength than usual, that is to say, higher strength than is obtained by a simple process of stretching, e. g., 2 grams per denier, by the use of a swelling agent for cellulose such as strong acids of hydrion concentration greater than that of a 45% solution of sulphuric acid, zinc chloride and calcium thiocyanate, the thread being stretched while in a plastic state occasioned by the use of a swelling agent immediately after (one bath process) or some time after (two bath process) the setting or decomposition operation during which cellulose is formed from the viscose. In other words, the cellulose formed in the setting process is caused to swell by the application of a cellulose swelling agent or agents and while thus swollen the threads are stretched considerably and the swelling agent removed.

The object of this invention is to produce strong threads from viscose without the use of such swelling agents and without the use of exceptionally strong acids, that is to say, without using acids of hydrion concentration corresponding to that of a 45% solution of sulphuric acid, the acids if used being advantageously of considerably lower concentration.

I attain my object by the application of tension and extension in a manner and under conditions hereinafter described which have not hitherto been applied in the spinning of viscose silk.

According to one embodiment of my invention I use as coagulants for the viscose, preferably viscose made from unaged or only partially aged alkali cellulose, baths containing strong or mineral acids of hydrion concentration less than that of a 7% solution of sulphuric acid, down to even 0.5% or less, preferably in the presence of neutral salts, and thereby effect coagulation of the viscose and slow decomposition into cellulose. With such a coagulating bath the viscose after coagulation exists for a short time in an evanescent or an active plastic state, that is to say, a condition in which the viscose remains plastic but active in so far that evanescent cellulose formed by the decomposition process is present and combining or polymerizing, its duration being the shorter and its distribution throughout the filaments being the less homogeneous the greater is the hydrion concentration of the coagulating acid; for example, in a solution of hydrion concentration equal to that of a 0.5% solution of sulphuric acid the active plastic state exists for distinctly less than 30 seconds, with a filament of about 1 denier, and most probably less than 10 seconds. In a solution of 7 per cent sulphuric acid the existence of the active plastic state is terminated in distinctly less than 3 seconds and probably less than 1 second, and, superficially, considerably less than 1 second and between these concentrations one has sufficient time to complete the stretching process.

In order that the stretching process may be completed within the duration of existence of the active plastic state, as soon as the filaments have sufficient solidity to permit of contact with a solid surface I begin to apply tension which continues with increasing stress so that the threads are extended at least 25% or even 100% or more during the time the viscose is being decomposed, that is to say, during the existence of the active plastic state. The time of application of tension is such that decomposition of the viscose into cellulose is practically completed while the threads are being subjected to tension.

In practice the threads may remain under tension for a longer time than the duration of existence of the active plastic state, but the actual extension of the threads only proceeds while the viscose is in the plastic state.

According to a further modification I use as coagulants in a first bath any of the coagulants already referred to, but immediately or very soon after their formation I pass the threads into a bath of water, the second bath, to which have been added such substances as, while permitting or even assisting the swelling of the formed threads possibly by virtue of the presence of some active xanthogenate groupings, will not carry the swelling process far enough to cause dissolution of the threads or will prevent such dissolution; for example, neutral salts with addition of one or more organic substances such as for instance sugar or glucose up to such a concentration as by the osmotic effects will prevent the dissolution of the threads. In this way the evanescent or active plastic state is maintained for a longer time and thus permits more opportunity whenever desired to adjust the time at which stretching is applied. I may thus stretch the threads at least 25% or 100% or even more, either before entering the second

bath or while in the second bath or after leaving the second bath or with any combination of these conditions, in fact at any time before or so long as the threads remain impregnated with the solution forming the second bath providing that the true fluidity of the viscose has first been overcome, but in any case during the existence of the evanescent or active plastic state.

My process differs essentially from ordinary practice in which normal viscose is spun into normal spinning baths in which the tension applied is insufficient to produce an extension or more than 25% after formation of the filaments. My process also differs essentially from processes merely applying a simple process of extension after the formation of the threads from normal viscose by the use of a normal spinning bath of usual or greater length than usual with one or several friction devices in so far that in those processes the tension and chemical decompositions are not adjusted to one another and the evanescent or active plastic state is missed.

My process differs further essentially from prior processes in which tension is applied after the viscose has been set and decomposed and treated, during such decomposition or immediately or some time afterwards, with exceptionally strong acids or other swelling agent for cellulose, in so far as I apply tension to and effect extension of the threads while they are in an evanescent or active plastic state which by the selection of conditions I cause to exist before the viscose is completely set or decomposed into cellulose.

Essential to my invention is the feature that after the filaments have been formed in the coagulating bath, that is to say, after the drawing effect which takes place close to the jet orifices in the ordinary spinning process or after the viscose has passed out of the true fluid state but during the period the viscose remains in an evanescent or active plastic or active colloidal state, I subject the filaments to tension sufficient to produce an extension of at least 25% or even 100% or more, and, while maintaining the stretched or tensioned condition, I bring about practically complete decomposition of the viscose. By suitable selection of conditions it is not difficult to produce threads with a tenacity of 2.5 to 3.5 grams per denier and even more may be secured by careful adjustment of the several operative factors.

The duration of existence of the active plastic state and the degree of plasticity may be varied within wide limits by altering the conditions of manufacture.

Thus, the time during which such tension and extension are to be applied or the length of thread over which such tension and extension are to be applied is to be adjusted in dependence on the duration of existence of the evanescent or active plastic state, i. e., on the concentration of the viscose, the age of the viscose, the concentration of alkali in the viscose, the rate of delivery of viscose from the jet orifices, the size of the jet orifices, the composition and concentration of the coagulating bath or other bath applied later, and the temperature of such bath or baths. Such adjustment is to be made that the viscose is practically completely decomposed while the threads are being subjected to tension.

Example I

Viscose prepared by any of the ordinary ways containing about 7% cellulose and about 6½% caustic soda and of about 10 ammonium chloride degrees of age is forced through a jet containing 25 orifices so that 1.2 grams of viscose are forced through per minute into a coagulating bath at 40 to 50° C., consisting of 20% sodium sulphate 2.5% sulphuric acid 14% ammonium sulphate.

The threads are passed as soon as can be conveniently arranged on to a flat, cylindrical or conical surface moving at say 25 metres per minute and after passing over any means for applying tension gradually are collected at say 40 to 50 metres per minute on bobbins or drawn over a godet or godets at this speed and finally collected in a centrifugal pot. The stretching operation is to be completed within one second from the time any particular quantity of the viscose leaves the jet to the time the thread passes to the collecting device or godet.

Example II

As soon as can be conveniently arranged the threads are passed out of the coagulating bath into a solution of Glauber salt containing 5 to 10% of sodium sulphate with or without addition of organic compounds such as alcohol or glucose of any strength provided that it does not cause separation of the inorganic salts from solution, or of alkalies such as caustic soda of less than 8% strength or ammonia of any strength and finally collected at 40 to 50 metres per minute. The stretching operation may be applied at any time after the viscose has lost its fluidity to a sufficient extent to permit of contact with a solid surface without disruption of the elongated gels or filaments.

Example III

As either of the preceding examples excepting that the speed of the collecting device is reduced to 30, 20 or even 10 metres per minute or less, as desired, always provided that the threads are stretched above 25% before passing on to the collecting device.

Example IV

As any of the preceding examples excepting that the amount of extension is increased to 150 or even 200% or to the maximum amount which by virtue of the magnitude of the plasticity remaining in the threads is possible without breaking the threads.

Example V

As any of the preceding examples excepting that in place of ordinary viscose there is used another alkali soluble salt or derivative of viscose such as zinc viscose which, like ordinary viscose, is decomposed into cellulose on treatment with a mineral acid or in other ways.

Example VI

As Example II excepting that, in the second bath, in place of Glauber salt, there is used a solution of another salt or of an organic compound such as alcohol or glucose whose osmotic activity is sufficient to prevent dissolution of the elongated gels but insufficient to cause appreciable dehydration or decomposition of the elongated gels.

Example VII

As any of Examples I to VI excepting that

a jet with many orifices, say a 150 hole jet is used.

Permissible variants and details unnecessary to describe at length include the employment of viscose made in a particular way, and the use of mechanical devices of any form in the performance of the invention.

The figure of the accompanying drawing illustrates diagrammatically apparatus preferred for performing the process.

In the drawing A denotes a spinning bath into which viscose is delivered through a jet. B denotes a pulley over which the filaments formed in the bath A are led from the bath. In the particular apparatus illustrated, the pulley B, assumed to have a circumference of 38.6 centimeters, rotates at the speed of twenty revolutions in seventy-two seconds. From the pulley B the filaments are led to the pulley C rotating at the same speed as the pulley B. From the pulley C the filaments pass to the pulley D, of the same circumference as the pulley B and performing twenty revolutions in fifty-four seconds. The pulley D dips into the bath E, i.e., the second bath. From the pulley D the filaments submerged in the bath E pass to the pulley F rotating at the same speed as the pulley D. From the pulley F the filaments pass to the pulley G, thence to the pulley H and thence to the spool J, which dips into an acid bath K.

In the particular apparatus illustrated, the spool J, having a circumference of 29.8 centimeters, performs sixty revolutions in sixty-six seconds.

I claim:—

1. The improvement in the manufacture of artificial filaments from viscose, consisting in coagulating the viscose in the form of filaments in acids of a hydrion concentration less than that of 7% H_2SO_4, causing the viscose to enter an evanescent active plastic state, then passing the filaments into a bath of water to which have been added substances which permit the swelling of the formed filaments by the action of the water, without bringing about dissolution of the filaments, and stretching the filaments before complete decomposition of the viscose, substantially as described.

2. The improvement in the manufacture of artificial filaments from viscose, consisting in effecting coagulation of the viscose, in the form of filaments, in acids of hydrion concentration less than that of 7% H_2SO_4, whereby the filaments are caused to enter an evanescent active plastic state, and gradually applying tension to the filaments to effect extension of the filaments commencing with solidification of the filaments and continuing while the filaments are in said evanescent active plastic state, whereby the filaments are extended at least 25% before complete decomposition of the viscose.

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