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PROCESS OF SPINNING RAYON AND THE BATH USED

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This invention relates to a process of spinning rayon, the coagulating bath used in that process, and the yarn resulting therefrom, and more particularly when making the viscose type of rayon.

The main object of the invention is to produce a yarn having improved tensile strength, toughness and elasticity. It is characteristic of the new bath that it contains zinc sulphate and nickel sulphate in certain specific quantities, and it is characteristic of the process that the yarn can be given an abnormally high stretch while spinning.

In producing a filament of regenerated cellulose, such as a viscose filament, it is believed that when the viscose solution is extruded into the spinning bath, a film of regenerated cellulose is formed around each tiny cylinder of viscose. This takes place very close to the face of the jet. Through this film by means of dialysis the remainder of the caustic soda within the viscose cylinder is neutralized by the acid of the bath and eventually the sodium cellulose xanthate is decomposed, producing a filament of regenerated cellulose.

The incorporation of zinc sulphate in the spinning bath produces yarn of increased tenacity and toughness. Apparently the presence of zinc sulphate greatly speeds up the neutralization of the caustic soda in the spinning solution. If too much zinc sulphate is present this action takes place so rapidly that it is impossible to draw the viscose into filaments. Hence the desirable qualities of increased tenacity and toughness attendant upon such percentages of zinc sulphate could not heretofore be obtained.

It is believed that in the presence of zinc sulphate, when the neutralization reaction is greatly speeded up, water is actually withdrawn from the cylinder of viscose surrounded by the skin of regenerated cellulose, neutralization of the caustic soda thereby taking place on the outside of this membrane. So rapid is this dehydration and neutralization that the membrane has no opportunity to shrink and consequently forms itself into folds, giving the serrated cross-section characteristic of yarns spun into precipitating baths containing zinc sulphate.

If the amount of zinc is excessive the rate of shrinkage is so great as to break off the filaments at the jet face, the jet becoming clogged, and producing faulty yarn. The shorter the age of the viscose, the less the amount of zinc sulphate that can be used and still produce a commercial yarn.

The dehydration and neutralization which take place in producing the filament of regenerated cellulose result in an extremely dry and quite plastic yarn, but owing to the toughness of the freshly spun thread there is a limit to the amount of stretch that can be given the yarn. A well known procedure to apply a stretch to the freshly spun thread is by carrying it over two successive revolving wheels, the second of which has a peripheral speed greater than the first by 25 to 40%.

I have discovered that the incorporation in the spinning bath of from .1 to .5% of nickel sulphate produces a twofold result. In the first place the presence of the nickel sulphate seems to overcome the bad influences of the zinc sulphate without interfering with the good effects thereof. This occurs to such an extent that spinning is possible with zinc sulphate concentrations which otherwise could not be used, and the yarn so produced has the characteristic properties as to tenacity and toughness described above as attendant upon the use of such percentages of zinc sulphate as were heretofore prohibitive. In the second place, the amount of stretch applied to the freshly spun yarn as above described can be increased to as much as 50%.

I give below the preferred composition of the spinning or coagulating bath and also the limits which can be used and still get the desired result. The specific gravity data are given at 40° C. and the composition data as per cent by weight.

Spinning bath	Preferred	Limits
Specific gravity	1.300	1.260 to 1.350
H_2SO_4	8.2%	6.5% to 9.0%
$ZnSO_4$	6.2%	4.0% to 8.0%
$NiSO_4$	0.3%	0.1% to 0.5%
$NaSO_4$	18.0%	14.0% to 18.0%

The balance of the spinning bath is H_2O. In this spinning bath it is also possible to use cobalt sulphate instead of nickel sulphate.

To use more sodium sulphate in the spinning or coagulating bath would result in the formation of crystals of sodium sulphate on the machine. The concentration of sodium sulphate can be reduced to as low as 14% if a small amount of glucose is added to the bath.

Although the above process is applicable to the usual type of viscose spinning solution or dope, employing wood pulp as a base, threads of considerably higher tenacity and toughness are produced if cotton pulp is substituted in part or in whole for the wood pulp. Similar advantage is obtained if a wood pulp which has been purified to a high alpha-cellulose content is used. The cellulose content of the viscose solution may conform to the usual figure of about 7%, and the sodium hydroxide concentration may be approximately that of the cellulose, or even higher, such as 8½ or 9%.

It has been found that a yarn made from such a cotton viscose solution or dope, spun in the above-mentioned high zinc sulphate bath protected by nickel sulphate, can be given a stretch in the spinning in excess of 40%—preferably 50%.

The result is that the tensile strength of the yarn is increased considerably without detracting from the yarn's toughness and elasticity. Thus, such a stretched yarn has a dry strength of about 3 grams per denier with an extensibility of from 25 to 30%. The wet strength is about 1½ grams per denier. If a 50% stretch were tried with the normal viscose and normal bath the dry tenacity would be much less than 3 grams per denier, and the extensibility would be too low to be practical.

I believe that I am the first to discover the effectiveness of small amounts of nickel sulphate to modify the excessive effects of high zinc sulphate baths.

I have also discovered that if during the process of applying stretch to the thread thus produced, heat is applied to the thread either by means of direct steam or by immersing it in hot water substantially at the boiling point, the amount of the stretch can be increased to as much as 100%. Such increase from the 50% previously described produces threads having a much higher tenacity than heretofore, without excessive reduction in extensibility.

It can also be seen that by applying such stretching process to the thread obtained from the spinning bath previously described, it would be possible to spin a thread of say 200 denier, and by heating and stretching reduce it to 100 denier. Thus, the effect of high percentage of zinc sulphate becomes much less drastic inasmuch as the draw off speed from the face of the jet is much less and the size of the individual filament is much greater than would be the case with the lower stretches normally used.

Consequently, if it seemed desirable, the concentration of zinc sulphate can be still further increased with further improvement in tenacity and toughness without encountering the spinning difficulties heretofore mentioned. It is also characteristic of this process that much higher percentages of the other coagulating bath constituents can be used, particularly if hot water is used as the plasticizing medium during the stretching operation, as the water removes the excess bath before tendering or crystallization can take place.

It has been found that the best results are obtained when the thread is subjected to the action of water at or near the boiling point over a distance of about 18" between two successive godet wheels, the second of which is revolving at a peripheral speed substantially 80% greater than that of the first. If the water thus used is recirculated, appreciable amounts of precipitating bath constituents will be accumulated therein, and according to the amount of fresh water added, may approach the concentrations present in the precipitating bath as a limit.

The following are the limits of operation for both the spinning bath and the plasticizing agent used in stretching, also the preferred conditions. The specific gravity data are given at 40° C. and the composition data as per cent by weight.

	Preferred	Limits
Specific gravity	1.300	1.260 to 1.400
Sulphuric acid	8.2%	6.5% to 12.0%
Zinc sulphate	6.2%	4.0% to 10.0%
Nickel sulphate	0.3%	0.1% to 1.0%
Sodium sulphate	18%	14% to 26%
Temp. of plasticizing water	100° C.	60° C. to 100° C.
Percent stretch	80%	40% to 100%

Yarn spun into the above preferred bath from viscose made of 100% cotton pulp hereinbefore described and given a stretch of 80% while subjected to the action of water at a temperature of 100° C. will have a dry strength in excess of 3½ grams per denier, a dry extensibility of 20% and a wet strength of 2½ grams per denier. Thus it will be seen that while the use of a sulphate of the nickel and cobalt group has important advantages, as above described, without the hot stretching, further advantages accrue from their cooperative use with hot stretching.

I claim:

1. A viscose spinning bath having a specific gravity of from 1.260 to 1.350 at 40° C. and containing by weight from 6.5% to 9% sulphuric acid, from 14% to 18% sodium sulphate, from 4.0% to 8.0% zinc sulphate, and from 0.1% to 0.5% of a sulphate of a metal of the group consisting of nickel and cobalt to counteract the objectionable effects of the zinc sulphate.

2. A viscose spinning bath having a specific gravity of from 1.260 to 1.350 at 40° C. and containing by weight from 6.5% to 9% sulphuric acid, from 14% to 18% sodium sulphate, from 4.0% to 8.0% zinc sulphate, and from 0.1% to 0.5% nickel sulphate to counteract the objectionable effects of the zinc sulphate.

3. A viscose spinning bath having a specific gravity of from 1.260 to 1.400 at 40° C. and containing by weight from 6.5% to 12% sulphuric acid, from 14% to 26% sodium sulphate, from 4% to 10% zinc sulphate, and from 0.1% to 1.0% of a sulphate of the group consisting of nickel and cobalt to modify the excessive effects of the zinc sulphate.

4. A viscose spining bath having a specific gravity of from 1.260 to 1.400 at 40° C. and containing by weight from 6.5% to 12% sulphuric acid, from 14% to 26% sodium sulphate, from 4% to 10% zinc sulphate, and from 0.1% to 1.0% nickel sulphate to modify the excessive effects of the zinc sulphate.

5. A viscose spinning bath containing by weight about 8.2% sulphuric acid, about 18% sodium sulphate, about 6.2% zinc sulphate, and about 0.3% nickel sulphate.

6. In a method of spinning viscose rayon, the step of projecting the spinning solution into a coagulating bath containing by weight from 6.5% to 9% sulphuric acid, from 14% to 18% sodium sulphate, from 4% to 8% zinc sulphate, and from 0.1% to 0.5% of a sulphate of the group consisting of nickel and cobalt to counteract the objectionable effects of the zinc sulphate.

7. In a method of spinning viscose rayon, the step of projecting the spinning solution into a coagulating bath containing by weight from 6.5% to 9% sulphuric acid, from 14% to 18% sodium sulphate, from 4% to 8% zinc sulphate, and from 0.1% to 0.5% nickel sulphate.

8. In a method of spinning viscose rayon, the step of projecting the spinning solution into a coagulating bath containing by weight from 6.5% to 12% sulphuric acid, from 14% to 26% sodium sulphate, from 4% to 8% zinc sulphate, and from 0.1 to 1.0% of a sulphate of the group consisting of nickel and cobalt and removing the excess concentration of the bath carried by the filaments before it can harm the same.

9. In a method of spinning viscose rayon, the step of projecting the spinning solution into a coagulating bath containing by weight from 6.5% to 12% sulphuric acid, from 14% to 26% sodium sulphate, from 4% to 10% zinc sulphate, and from 0.1% to 1.0% of nickel sulphate, and remov-

ing the excess bath carried by the filaments before it can harm the same.

10. In a method of spinning viscose rayon, the step of projecting the spinning solution into a coagulating bath containing by weight about 8.2% sulphuric acid, about 18% sodium sulphate, about 6.2% zinc sulphate and about 0.3% nickel sulphate.

11. In a method of spinning viscose rayon, the step of projecting the spinning solution into a coagulating bath containing by weight from 6.5% to 9.0% sulphuric acid, from 14.0% to 18.0% sodium sulphate, from 4.0% to 8.0% zinc sulphate, and from 0.1% to 0.5% nickel sulphate, and then stretching the freshly spun yarn.

12. In a method of spinning viscose rayon, the steps of projecting the spinning solution into a coagulating bath containing by weight from 6.5% to 12% sulphuric acid, from 14% to 26% sodium sulphate, from 4% to 10% zinc sulphate, and from 0.1% to 1.0% of a sulphate of a metal of the group consisting of nickel and cobalt, reducing the concentration of the excess bath carried by the yarn, and then stretching the freshly spun yarn in excess of 40%.

13. In a method of spinning viscose rayon, the steps of projecting the spinning solution into a spinning bath containing from 6.5% to 12% sulphuric acid, from 4% to 10% zinc sulphate, from 14% to 26% sodium sulphate, and from 0.1% to 1.0% of a sulphate of a metal of the group consisting of nickel and cobalt, and then stretching the yarn in excess of 50% while subjecting the yarn being stretched to the action of an aqueous heating liquid at a temperature of from 60° to 100° C.

14. In a method of spinning viscose rayon the steps of projecting the spinning solution into a coagulating bath containing from 6.5% to 12% sulphuric acid, from 4% to 10% zinc sulphate, from 14% to 26% sodium sulphate, and from 0.1% to 1.0% of nickel sulphate, and then stretching the yarn in excess of 50% while subjecting the yarn being stretched to the action of an aqueous heating fluid at a temperature of from 60° to 100° C.

15. Method of producing viscose rayon having improved tensile strength, toughness and elasticity, which comprises projecting viscose spinning solution into a coagulating bath containing from 6.5% to 12% sulphuric acid, from 14% to 26% sodium sulphate, from 4% to 10% zinc sulphate, and from 0.1% to 1.0% of a sulphate of the group consisting of cobalt and nickel, which counteracts the excessive effects of the zinc sulphate, subjecting the yarn to aqueous medium and stretching the yarn in excess of 50% with the application of heat to a temperature of from 60° to 100° C.

16. Method of producing viscose rayon having improved tensile strength, toughness and elasticity which comprises projecting viscose spinning solution into an aqueous coagulating bath containing from 6.5% to 12% sulphuric acid, from 14% to 26% sodium sulphate, from 4% to 10% zinc sulphate and from 0.1% to 1.0% nickel sulphate, continuously withdrawing the coagulated filaments from said bath, wetting said filaments, and stretching them between godets in excess of 40% while heating the filaments to keep them plastic.

17. In a method of spinning viscose rayon, the steps of projecting the spinning solution into a spinning bath containing from 6.5% to 12% sulphuric acid, from 4% to 10% zinc sulphate, from 14% to 26% sodium sulphate, and from 0.1% to 1.0% of a sulphate of a metal of the group consisting of nickel and cobalt, and then stretching the yarn in excess of 50% while subjecting the yarn being stretched to the action of direct steam.

18. In a method of spinning viscose rayon, the steps of projecting the spinning solution into a spinning bath containing the following constituents: from 6.5% to 12% sulphuric acid, from 4% to 10% zinc sulphate, from 14% to 26% sodium sulphate, and from 0.1% to 1.0% of a sulphate of a metal of the group consisting of nickel and cobalt, then stretching the yarn in excess of 50% while subjecting the yarn being stretched to the action of an aqueous medium at a temperature from 60° to 100° C., circulating and recirculating the aqueous medium to accumulate the coagulating bath constituents therein, adding water to the circulating aqueous medium and controlling the amount of water added to maintain the concentration of the coagulating bath constituents in the circulating aqueous medium the same or less than the concentration of the coagulating bath.

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