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METHOD FOR PRODUCING HIGH STRENGTH
AND CRIMPED STAPLE FIBERS FROM
VISCOSE

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This invention relates to a method for produc-
ing high strength and crimped staple fibers from
viscose.

It is an object of the invention to produce high
strength staple fibers by means of a combined
bath and stretching process which is adapted to
impart a desirable and permanent crimped effect
to the fibers.

We have found that artificial silk filaments
produced from viscose with the aid of acid, iron-
containing baths can be stretched to an excep-
tionally great degree when in freely spun condi-
tion. The amount of stretch attainable in-
creases, up to a certain degree, with the tem-
perature of the spin bath employed and the
method provides a suitable means for obtain-
ing high strength artificial silk filaments. In
practicing the invention we employ preferably a
primary or spin bath of moderate temperature,
e. g. 40 to 60° C., such as is commonly used in
the viscose industry and a feature of the present
invention is that this must contain iron com-
pounds, either deliberately added thereto or
present therein at the outset. This is followed
by a second bath having a higher temperature,
e. g. 60 to 100° C. in which the stretching of the
filaments is carried to the extent desired, the
amount of stretch produced being considerable.
Ordinary so-called Müller baths of the viscose
industry may be used as the primary or spin
baths and which in addition to sulphuric acid,
sodium sulphate, and the like, also contain ap-
preciable quantities of a soluble iron salt or salts,
such, for example, as iron sulphate, to the ex-
tent, for example, of 10 to 100 grams per liter.
In accordance with U. S. Patent No. 836,452 to
Müller, of November 20, 1906, the so-called
Müller baths contain between 5 percent and 40
per cent of sulphuric acid, i. e., about 50 to 400
grams of sulphuric acid per liter of water. A
serviceable spin bath may be of the following
composition which is given as a non-limiting
embodiment of such a bath:

	Grams
H ₂ SO ₄ -----	125
Na ₂ SO ₄ -----	250
FeSO ₄ -----	50

The second bath used may consist of a diluted
spin bath or one that is automatically or spon-
taneously formed by the carrying over of bath
fluid from the primary bath. Care need only be
taken that the concentration of acid and salts
does not rise to an excessive extent, especially
when ripened viscose of a grade of 12° Hotten-

roth or even older is used. The composition of
the stretching bath, in which no iron content is
necessary, can be conveniently adjusted by the
addition of water. The temperature of the
stretching bath may lie between 60 to 100° C.,
and for practical purposes is preferably chosen
between 70 to 90° C., and the amount of stretch
effected in this bath may be easily carried to
beyond 50 to 100% and more without causing
the filaments to tear. Filaments spun and
stretched in this fashion constitute material
possessing the essential prerequisites for the
production of a high strength and well crimped
staple fiber. However, in order to obtain such
a product commercially with success, a number
of definite operative prerequisites and factors
are involved. In this connection it is to be
noted that additional spinning processes adapted
for producing fibers capable of providing raw
material suitable for the stretching and crimp-
ing process of the present application are dis-
closed in our co-pending application Serial No.
279,360 filed June 15, 1939, for "Process of spin-
ning viscose." In this application we disclosed
the following:

We are well aware that it has, heretofore, been
proposed to spin viscose solutions into primary
spin baths containing soluble zinc salts, such as,
for example, zinc sulphate, and to stretch the
coagulated products in a secondary bath main-
tained at a temperature of at least 60° C. in
order to obtain artificial silk having especially
favorable physical characteristics. (Vide British
Patent #467,500 to Courtaulds Ltd.) However,
such zinc-bearing baths are deficient in that
they rapidly cause clogging of the orifices of
noble metal spinnerets, especially when they
contain a large amount of zinc, for example 50
grams of zinc sulphate per liter of water. This
deficiency, however, cannot be overcome by
simply reducing the amount of zinc in the spin
bath for the reason that large amounts of zinc
salt are necessary to impart to regenerated cel-
lulose a high strength in dry and wet condition.

We have, unexpectedly, found by experimenta-
tion that the aforementioned deficiency of zinc
baths can be overcome by using a primary spin
bath containing soluble iron salts, such as iron
sulphate, etc. The amount of iron in the pri-
mary bath may be varied within wide limits,
although we have found that about 15 to 50
grams of an iron salt, for example, iron sulphate,
per liter of bath are generally sufficient to give
the desired result. However we do not wish to
be limited to this amount since it may be lowered

or raised in order to vary the physical characteristics of the artificial silk spun in the bath. In this manner it is possible to obtain viscose yarns, staple fibers, etc., from conventional viscose solutions having a dry strength of about 30 to 340 grams per 100 den., and a wet strength of about 190 to 200 grams per 100 den. The strength of the regenerated cellulose may naturally be modified by using special viscose solutions. Such special viscose solutions may contain lower or higher amounts of cellulose and/or be less ripened than conventional viscose solutions.

The aforementioned effect of iron salts in the primary spin bath, however, is only obtained when the initial products formed therein are stretched in a hot, secondary spin bath. In other words, it is not possible to eliminate the iron salt from the primary bath and to stretch the yarn, etc., to the same degree in the hot secondary spin bath. When a viscose solution is projected into a primary spin bath devoid of iron to form an "initial" thread and this thread is then given the maximum stretch in a hot, secondary bath, the "final" thread will have less strength than threads of regenerated cellulose obtained in accordance with our novel process. The beneficial effect of iron in the primary bath is bound to the stretching in the hot, secondary bath. This effect becomes noticeable at a temperature of about 60° C. and pronounced at a temperature of about 70° C. However, the secondary bath may have a temperature of 80°, 90°, and attain even the boiling point of water.

We are well aware that it has, heretofore, been recommended to add soluble iron salts, such as iron sulphate, iron nitrate, iron nitrite, ferrous ammonium sulphate, ferric ammonium sulphate, etc., to spin baths in order to somewhat eliminate hydrogen sulphide generated during the spinning of viscose. However, it could not be foreseen by those skilled in the art that iron in the primary bath has the property of promoting the stretching in a hot, secondary bath, this stretching resulting in especially strong end products. Furthermore, it could not be foreseen that spin baths containing soluble iron salts, especially iron sulphate, reduce the clogging of spinneret orifices in comparison with zinc-bearing spin baths. This reduction in clogging of spinneret orifices results in a reduction of spinneret change during the production of viscose products. When conventional, acid spin baths containing about 50 grams of zinc sulphate per liter are used, a considerable clogging of the orifices of noble metal spinnerets takes place after six hours, and the cross section of each orifice is reduced about 25 to 30% by the formation of so-called "zinc rings." When the novel spin baths, containing for example 50 grams of iron sulphate and no zinc sulphate are used the spinneret orifices are not clogged after six hours of spinning. There is no formation of corresponding "iron rings."

Example

A conventional viscose solution, containing about 7.2% cellulose and 6.5% alkali and matured to 12.5° (H), is spun into a primary spin bath at a temperature of about 50° C. the bath travel is about 20 cm. The primary bath contains about 125 g. of sulphuric acid, 260 g. of sodium sulphate and 50 g. of iron sulphate per liter of water. The thread is continuously stretched about 80% by means of driven rollers in a secondary bath maintained at a temperature of

about 85°. The first roller has a speed of about 33.5 meters per minute and the second one a speed of about 60 meters per minute. The secondary bath may consist of pure water or it may contain small amounts of the ingredients of the primary bath. These ingredients may be carried over by the thread from the primary to the secondary bath. The thread, thus, produced has the following physical characteristics:

	Grams
Dry strength per 100 den.....	300-340
Wet strength per 100 den.....	190-200

Although we prefer to use iron sulphate for reasons of economy in our spinning process, we wish to emphasize that it may be replaced by any iron salt sufficiently soluble and stable in spin baths conventionally used in the viscose art.

In practicing the method of the present invention, it is preferable after stretching, first to assemble the numerous filaments coming from the various spinnerets into a thick bundle having a denier value of about 50,000 and more. The bundle is then maintained (in continuous working phase) at the increased length imparted to said filaments by the stretching operation, and for a limited period, one or more minutes, to cause them to retain their stretch and care must be taken that no shortening occurs during this period. This step, we have found, is important if especially high strength goods are to be produced. The filaments or rather the continuously produced bundles of filaments are thereupon cut into the desired staple lengths. At this point it may be found that the filaments become somewhat shortened as if a portion of the originally effected stretch amounted merely to a sort of temporary elastic extension. The degree of contraction or retraction of the material however is dependent on the use of iron compounds in the spin bath from which the filament were produced.

The bundles, which need not be opened up to any special extent, are then, in accordance with the present invention, either immediately or at least very soon placed in a limited amount of hot or boiling water or hot diluted spin bath or some other suitable hot liquid. This serves to drive out the carbon disulphide which escapes violently.

This violent driving out of the carbon disulphide has been found to be an additional, if not in fact an all sufficient prerequisite for the production of a satisfactory and lasting crimped effect. It is therefore desirable and important not to drive out the carbon disulphide until the moment that the filament or bundles are subjected to the hot bath treatment. For this reason it has even been found preferable not to prolong the heating in the hot stretching bath beyond the point needed for achieving the required stretch and furthermore the bundles must not be washed in either hot or cold liquids; in other words, the major portion of the carbon disulphide must be retained in the threads until the time that the desired violent evolution of the carbon disulphide is to be produced. If the uncut filaments or staple fiber bundles were to be even merely washed cold, a great deal of the crimped effect would become lost. The crimped effect is also decreased if the bundles are allowed to remain exposed to the air for a prolonged period, e. g. several hours, even at room temperature. But if an appreciable amount or rather the greater portion of the carbon di-

sulphide is still present in the bundles when they are hot treated then, when they are introduced into the hot medium, a violent evolution of carbon disulphide takes place and an exceptional and remarkably uniform crimped effect is produced. The masses of fibers may thereafter be washed by suitable known methods, desulphurized, bleached, brightened, and dried without producing any decrease in the crimped effect.

In order to produce a high strength well crimped staple fiber it is therefore essential to have a number of prerequisites and in any case two main desiderata: the freshly spun threads must be strongly stretched and extended and this is best effected in a hot bath, and especially in a second stretching bath. In order to effect an especially high degree of stretch, a spin bath of special properties must be used, the feature of which, in accordance with the present invention, resides in the fact that it contains an appreciable amount of iron, e. g. soluble iron salts such as iron sulphate. The filaments, maintained under tension and stretch for a suitable period after spinning and stretching, are then cut into staple fiber units and care must be taken that the carbon disulphide contained in the filaments and bundles remains therein down to the instant at which the bundles (in accordance with the invention) are introduced into the hot treating bath, the carbon disulphide being then caused to be suddenly and violently driven out of the filaments. The presence of iron in the spin bath used for producing the filaments not only controls the degree of the stretch subsequently obtained and the high strength associated therewith as a function thereof, but, considered as applied to the process as a whole, it also controls the elastic properties of the filament bundle at the time that the same is cut off into lengths from the tensioned or stretched bundle of filaments and therefore also controls the production of a satisfactory crimped effect.

We claim:

1. In the process of producing crimped, high-strength staple fibers, the steps in sequence which comprise projecting a viscose solution into a primary spin bath at a temperature of about 40 to 60° C. to form a multi-filament thread, stretching said thread in a secondary, aqueous bath maintained at a temperature of about 70 to 90° C., instantly cutting said stretched thread under tension into staple fibers and instantly immersing said staple fibers into a liquid maintained at a temperature sufficiently high to rapidly remove carbon bisulphide from said

fibers, said primary spin bath containing about 125 g. of sulphuric acid, 280 g. of sodium sulphate and about 10 to 100 g. of a soluble iron salt per liter of water.

2. In the process of producing crimped, high-strength staple fibers, the steps in sequence which comprise projecting a viscose solution into a primary, aqueous spin bath at a temperature of about 40 to 60° C. to form a multi-filament thread, stretching said thread in a secondary, aqueous bath maintained at a temperature of about 60 to 100° C. to form an elastic, stretched thread, instantly cutting said elastic, stretched thread under tension into staple fibers and instantly immersing said staple fibers into a liquid maintained at a temperature sufficiently high to rapidly remove carbon bisulphide from said fibers, said primary bath comprising about 50 to 400 grams of sulphuric acid and about 10 to 100 grams of dissolved iron salt per liter of water.

3. In the process of producing crimped, high-strength staple fibers, the steps in sequence which comprise projecting a viscose solution into a primary, aqueous spin bath at a temperature of about 40 to 60° C. to form a multi-filament thread, stretching said thread in a secondary, aqueous bath maintained at a temperature of about 60 to 100° C. to form an elastic, stretched thread, instantly cutting said elastic, stretched thread under tension into staple fibers and instantly immersing said staple fibers into a liquid maintained at a temperature sufficiently high to rapidly remove carbon bisulphide from said fibers, said primary bath comprising about 50 to 400 grams of sulphuric acid and about 10 to 100 grams of dissolved iron sulphate per liter of water.

4. In the process of producing crimped, high-strength staple fibers, the steps in sequence which comprise projecting a viscose solution into a primary spin bath at a temperature of about 40 to 60° C. to form a multifilament thread, stretching said thread in a secondary, aqueous bath maintained at a temperature of about 70 to 90° C., instantly cutting said stretched thread under tension into staple fibers and instantly immersing said staple fibers into a liquid maintained at a temperature sufficiently high to rapidly remove carbon bisulphide from said fibers, said primary spin bath containing about 125 g. of sulphuric acid, 280 grams of sodium sulphate and about 10 to 100 grams of dissolved iron sulphate per liter of water.

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