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PRODUCTION OF SHAPED OBJECTS FROM VISCOSE

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4 Claims. (Cl. 106—165)

This invention relates to a process for the production from viscose of artificial threads and other shaped objects, including films, having greatly improved properties as regards their resilience and their smaller degree of swelling. The invention also relates to the resulting products.

This application is a division of U.S. application Serial No. 541,095, filed October 17, 1955.

One of the principal objects of the present invention is to provide a new and improved method for spinning artificial products such as threads and other shaped objects from viscose in such manner as to confer upon them greatly improved properties as regards their strength, elasticity and abrasion-resistance. A further object of the invention is to provide a new and improved method for producing products of the kind just indicated having unusually strong and well-defined peripheral zones. A still further object of the invention is to provide improved products of the kind indicated.

The manner in which these and other objects and features of the invention are attained will appear more fully from the following description thereof, in which reference is made to typical and preferred procedures in order to indicate more fully the nature of the invention, but without intending to limit the invention thereby.

Very great value has been attached for a number of years in the viscose artificial fiber industry to the production of threads having a strong and well-defined peripheral zone because of their better properties as regards resilience and their smaller degree of swelling. For the production of such fibers, use has been made of unripened viscoses and spinning baths which contain zinc sulphate having comparatively low acid contents. The zinc sulphate content has in fact been considered to be an essential condition for the production of these fibers.

Processes are also known by which such threads may be obtained by supplying additives to the viscose or to the spinning bath. Such additives are alkali-soluble monoamines, which must also be soluble in the spinning baths which are used and which contain 3–25% of zinc sulphate. In accordance with the prior known processes, the additions of such amines should not amount to more than 4 millimols/100 g. of viscose.

When using such prior processes, difficulties were encountered from the fact that only very small fluctuations are permissible both as regards the amounts of amines added to the viscose and as regards the concentrations of acid and zinc sulphate to be maintained in the spinning bath if the expected technical effect is to be produced.

According to the present invention, it has now been discovered that threads having improved strength and elasticity values may be obtained if, in contrast to the

hitherto known processes, complex compounds are added to the viscose having the following formula:

$$[Me(X)_6]An$$

in which Me represents iron, zinc, chromium, cadmium, tin; X represents alkyl amine, alkanol amine or aryl amine; and An represents chloride, sulphate or nitrate.

The process may be carried out in a particularly advantageous manner by using

$$[Fe[(C_2H_4OH)_3N]_6]Cl_3$$

$$[Fe[(CH_3\text{-}CHOH\text{-}CH_2]_2NH)_6]Cl_3,$$

or

These compounds are added just prior to the spinning operation in amounts of 0.06–0.4% by weight, and preferably 0.12% by weight. Viscoses having a conventional composition of 7 to 8% of cellulose and 5 to 7% of NaOH, with a gamma value of at least 42, are spun at temperatures of 45–70° C. in spinning baths having the following composition: 40–65 g./l. of H_2SO_4, 160–260 g./l. of Na_2SO_4 and 30–50 g./l. of $ZnSO_4$. It is expedient to operate with thread withdrawal speeds of 40–60 m./min. The withdrawal speed may be increased, however, if care is taken with the aid of known means to impart to the spinning bath, at least in the first section of its travel after the spinning nozzle, a velocity which is substantially equal to the thread velocity so that the friction between the thread and the spinning bath is thereby substantially reduced. The length of travel in the bath is to be at least 30 cm. In a second bath, which contains approximately 2–10 g./l. of H_2SO_4, the thread is subjected to a drawing of 80–120%, preferably 95–100%, at temperatures higher than 90° C. The thread may then be deposited onto a bobbin, into a centrifuge or onto a perforated web, as desired.

The threads spun by the process according to the present invention have strength values of 380–400 g./100 den., which may be increased by subsequent drawing to 420–440 g./100 den. The finally drawn threads show a very high degree of shrinkage, namely of more than 8%, and have a low degree of swelling.

In order to indicate still more fully the nature of the present invention, the following examples of typical procedure are set forth, it being understood that this description is presented by way of illustration only, and not as limiting the scope of the invention.

Example 1

Alkali cellulose consisting of linters or wood cellulose is sulphidized for 5 hours with 42% of CS_2 and the xanthate obtained is dissolved to form a viscose with 7.3% of cellulose and 5.5% of NaOH. The viscose is de-aerated, filtered and finally ripened in the usual manner and is mixed 5 hours before being spun with 0.1% by weight of a compound having the formula

$$[Fe[(CH_2CH_2OH)_3N)_6]Cl_3$$

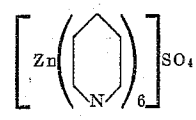
The viscose is spun with a gamma value of 45 from a spinneret with 1000 orifices to provide a thread of 1650 den., this taking place in a bath with 49 g./l. of H_2SO_4, 200 g./l. of Na_2SO_4 and 39 g./l. of $ZnSO_4$. The thread then travels through a distance of 70 cm. in the spinning bath and is withdrawn by a first roller at 23 m./min. The thread then travels through a second bath with 6 g./l. H_2SO_4 at a temperature of 90° C. and is withdrawn by a second roller at 46 m./min.; that is to say, it is stretched by 100%. The thread travels from

the second roller onto a bobbin and is collected thereon. The thread is then rewound, washed, finished and dried. It has the following textile data:

Strength, g./den.		Elongation, percent	
dry	wet	dry	wet
4.2	2.8	14	26

Example 2

0.15% by weight of



is added to a viscose with 7.3% of cellulose and 5.5% of NaOH about 10 minutes before spinning, whereupon it is spun in a bath containing 55 g./l. of H_2SO_4, 170 g./l. of Na_2SO_4 and 45 g./l. of $ZnSO_4$, the withdrawal conditions and the stretching being the same as in Example 1. The washed and finished threads are subjected to a final stretching by 8% and are thereafter dried. They have the following textile data:

Strength, g./den.		Elongation, Percent	
dry	wet	dry	wet
4.1	2.8	10	24

Example 3

0.1% by weight of

$$[Fe[(CH_3CH(OH)CH_2)_2NH]_6]Cl_3$$

is added to viscose with 7.3% of cellulose and 6% of NaOH about 1 hour before spinning. It is then spun in a spinning bath which contains 60 g./l. of H_2SO_4, 220 g./l. of Na_2SO_4 and 50 g./l. of $ZnSO_4$. The bath has a length of 70 cm. It is operated with a thread withdrawal velocity of 23 m./min. and the thread is stretched by 100% by being withdrawn at a velocity of 46 m./min. in a second dilute acid bath at a temperature of 90° C. From the second roller, the thread runs into a centrifuge operating at 6,000 r.p.m. and is given a twist of 120 turns per meter. The thread is then washed and finished and dried after final stretching of 8%. In this way,

there are obtained threads with the following textile data:

Strength, g./den.		Elongation, percent	
dry	wet	dry	wet
4.3	3.0	12	23

While specific examples of preferred methods and products embodying the present invention have been described above, it will be apparent that many changes and modifications may be made in the methods of procedure and the products without departing from the spirit of the invention. It will therefore be understood that the examples cited and the methods and procedures set forth above are intended to be illustrative only and are not intended to limit the invention.

What is claimed is:

1. A viscose spinning solution containing 0.06–0.4% by weight based on the viscose of a complex compound having the formula:

$$[Me(X)_6]An$$

wherein Me is selected from the class consisting of Fe, Zn, Cr, Cd and Sn; X is selected from the class consisting of alkyl amine, alkanol amine, and aryl amine; and An is selected from the class consisting of Cl, SO_4 and NO_3.

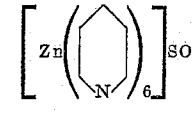
2. A viscose spinning solution according to claim 1, wherein the complex compound has the formula:

$$[Fe[(CH_2CH_2OH)_3N]_6]Cl_3$$

3. A viscose spinning solution according to claim 1, wherein the complex compound has the formula:

$$[Fe[(CH_3CHOHCH_2)_2NH]_6]Cl_3$$

4. A viscose spinning solution according to claim 1, wherein the complex compound has the formula:



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