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2,728,631

**PROCESS FOR THE PRODUCTION OF CRINKLED
POLYACRYLONITRILE YARNS**

Nicolas Drisch and Paul Herrbach, Paris, France, assignors to Textile & Chemical Research Company Limited, a corporation of Guernsey, Channel Islands

No Drawing. Application February 20, 1953,
Serial No. 338,142

Claims priority, application France November 5, 1952

4 Claims. (Cl. 18—54)

This invention relates to the production of crinkled polyacrylonitrile yarns and particularly to the production of crinkled yarns of polymeric acrylonitrile or of copolymers of acrylonitrile with other ethylenic compounds containing at least 85% of acrylonitrile. Such polymers and copolymers will be hereinafter referred to for brevity simply as acrylonitrile polymers.

By the term "crinkled" yarn is meant a yarn which has a continuous curl of fine dimensions along its length.

According to the present invention a process for the production of crinkled yarns comprises spinning through spinnerets a solution of an acrylonitrile polymer (as above defined), into a spinning bath at 15° to 40° C. the said bath consisting of an aqueous solution of a solvent for the polymers and containing a salt having a highly hydrated ion, and thereafter, with or without stretching the filaments continuously with their production, washing and drying them without tension.

In this way there are obtained yarns having a very fine and stable crinkle. The spinning process is simple to carry out and the filaments produced, which have a relatively low swelling power, can readily be stretched in a hot aqueous medium after they leave the spinning bath. The stretched filaments (or yarns) which have been washed and dried without tension, have a pleasant silky feel and an excellent covering power. They have good serimetric characteristics and are readily dyed. They exhibit little tendency to accumulate static electrical charges.

The invention is of particular importance where the solvent used in the polymer solution and in the spinning bath is either dimethylformamide or diethylformamide and where the salt used is calcium chloride and the invention will therefore be described with particular reference to the use of these materials.

The spinning bath may contain from 30% to 60% of solvent for the polymer, from 10% to 30% of a salt such as calcium chloride, and from 10% to 60% of water. Preferably, the said bath consists of about 40% of solvent for the polymer, 20% of calcium chloride and 40% of water.

The temperature of the bath, as stated above, must be within the limits 15° to 40° C. but spinning is preferably carried out at 20° to 25° C.

The crinkle is obtained with unitary filaments of a wide range of denier, e. g. from 1 to 10 denier, but it is found to be best obtained with filaments of 2 to 5 or 6 denier.

The yarn may be washed on bobbins or in cakes. The washing may also be carried out continuously, for example on rollers or 2-cage reels or on any other device for continuous treatment. In continuous treatment processes, it is important to ensure that there is a sufficient length of travel of the yarn during such treatment to enable adequate washing to be effected.

The yarn may be washed with a dilute salt solution, for example a 1% solution, and then with water, or it may be washed with water alone, the water being heated, for

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example, to 30° to 80° C. The washing path may be shorter if hotter washing baths are employed. Generally speaking, the length of travel in the washing bath should be at least 5 m.

By circulating the washing solution in counter-current, washing solutions having a high solvent content can readily be recovered.

The polymer solution may be prepared, preferably immediately before spinning, by rapidly heating a dispersion of polymer in a solvent therefor and then cooling it before delivering it into the bath. The heating operation may be effected in a coil immersed in an oil bath, and the subsequent cooling of the hot solution may be effected by passing the solution into a swan-neck provided with a cooling jacket, the swan-neck having a spinning nozzle at its end. This method of preparation of the spinning solution enables clear, stable solutions having very little coloration to be obtained.

The following examples will serve to illustrate the invention but are not to be regarded as limiting it in any way.

Example I

200 g. of polyacrylonitrile having a molecular weight of approximately 60,000 are dispersed in 800 g. of dimethylformamide and this dispersion is heated to about 120° C. and then immediately cooled to 40° to 50° C. The dispersion is delivered into the thermal treatment apparatus at the rate of 8 g. per min.

The solution is spun through a nozzle having 100 holes of 0.1 mm. diameter into a bath containing 40% of dimethylformamide, 20% of CaCl₂ and 40% of water, at a temperature of 20° C.

The distance of travel of the spun filaments through the bath is 50 cm. the initial speed being 10 m./min.

The yarn is washed in water at 50° C. on a reel, the yarn making several turns round the reel and being continuously traversed, the total travel on this device being about 10 m. The yarn is then stretched by means of stretch rolls to 8 times its initial length while passing through a boiling water bath. Finally, the yarn is lubricated with a product obtained by the reaction of fatty alcohols with ethylene oxide, and is wound on skein winders. The skeins are dried without tension.

The very silky, highly crinkled yarn thus obtained has the following properties:

	Tenacity, g./d.	Elongation, percent
Dry-----	3.1	20
Wet-----	2.8	21

Example II

A 25% dispersion of a polymer as in Example I is spun under the same conditions at a rate of 64 g. per min., by means of a nozzle having 1000 holes of 0.1 mm. diameter into a bath containing 35% of dimethylformamide, 22% of CaCl₂ and 43% of water at 22° C. the filaments having a travel of 40 cm. in the bath and the initial speed being 10 m. per min.

The yarn is then washed continuously with a 10% aqueous solution of CaCl₂ at 30° C. on a reel as in Example I and is afterwards washed with water at 20° C. on another device of the same type.

The yarn is stretched by means of rolls to 7.5 times its initial length while it passes through boiling water, is lubricated, cut into 60 mm. lengths, and then dried.

The fibre obtained is soft, has a silky lustre and woolly appearance and has a very fine crinkle which gives to the yarn a high covering power. It can be dyed in rich and bright shades, for example with dyes known for use on cellulose acetate.

The serimetric characteristics of the fibre are:

	Tenacity, g./d.	Elongation, percent
Dry.....	2.8	30
Wet.....	2.6	32

An important advantage of the present invention is that yarns are obtained which can be stretched immediately following their spinning, whereas in the majority of the known processes for spinning acrylonitrile polymers only a very fragile yarn can be obtained, which must subsequently be stretched in a separate operation. This advantage may derive from the fact that the filaments produced according to the present invention are in a highly de-swollen condition when they leave the nozzle, so that the yarn is immediately given substantial strength.

The yarns and fibres obtained have good textile properties and are easily worked on conventional textile machinery, notably by reason of their low tendency to accumulate static charges. Their resistance to friction and fatigue is materially greater than that of the known polyacrylonitrile yarns.

The crinkle is stable, and although it is reduced when the yarns are stretched, it is restored by conditioning in a humid atmosphere.

Acrylonitrile polymers which can be used in accordance with the present invention include polyacrylonitrile itself and copolymers of acrylonitrile with, for example, vinyl chloride, vinylidene chloride, vinyl pyridene, butadiene compounds, vinyl acetate or acetals, alkyl methacrylates and the like.

We claim:

1. A process for the production of crinkled yarns which comprises spinning through spinnerets a solution in a solvent selected from the class consisting of dimethyl and diethylformamides of an acrylonitrile polymer containing at least 85% of acrylonitrile into a spinning bath at 15° to 40° C. the said bath consisting of 30-60% by weight of a said solvent for the said polymer, 10-30%

by weight of calcium chloride and from 10-60% by weight of water, stretching the yarn continuously with its production and thereafter washing the yarn obtained and drying it without tension.

2. A process for the production of crinkled yarns which comprises spinning through spinnerets a solution in a solvent selected from the class consisting of dimethyl and diethylformamides of an acrylonitrile polymer containing at least 85% of acrylonitrile into a spinning bath at 15° to 40° C. the said bath consisting of about 40% of the said solvent, about 20% of calcium chloride and about 40% of water, stretching the yarn continuously with its production and thereafter washing the yarn obtained and drying it without tension.

3. A process for the production of crinkled yarns which comprises spinning through spinnerets a solution in a solvent selected from the class consisting of dimethyl and diethylformamides of an acrylonitrile polymer containing at least 85% of acrylonitrile into a spinning bath at 15° to 40° C. the said bath consisting of about 40% of the said solvent, about 20% of calcium chloride and about 40% of water stretching the yarn produced while it is passing through a hot aqueous medium, continuously with its production, washing the yarn and drying it without tension.

4. A process for the production of crinkled yarns which comprises spinning through spinnerets a solution in a solvent selected from the class consisting of dimethyl and diethylformamides of an acrylonitrile polymer containing at least 85% of acrylonitrile into a spinning bath at 15° to 40° C. the said bath consisting of about 40% of the said solvent, about 20% of calcium chloride and about 40% of water stretching the yarn produced while it is passing through a hot aqueous solution of calcium chloride, continuously with its production, washing the yarn and drying it without tension.

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