United States Patent Office 2,833,738 Patented May 6, 1958

2,833,738

METHOD OF PRODUCING A SPINNING SOLUTION OF AN ACRYLONITRILE POLYMER

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No Drawing. Application June 16, 1954
Serial No. 437,285

Claims priority, application Germany June 25, 1953

3 Claims. (Cl. 260—31.6)

This invention relates to a process for producing a spinning solution from polyacrylonitrile or copolymers thereof.

Polyacrylonitrile and copolymers thereof which are soluble in the usual organic solvents only with difficulty, or not at all, heretofore were dissolved in solvents such as dimethylformamide or glycolic acid nitrile, and the solutions thus obtained were used in the production of threads or filaments. It was found, however, that the preparation of the spinning solutions by means of any of the known solvents was accompanied by serious drawbacks, more particularly a tendency of the solutions thus obtained to form jellies. Even when all possible precautions were taken this jelly-forming tendency persisted and could not be inhibited.

However, apart from the jelly-forming tendency, if glycolic acid nitrile was used as a solvent another very inconvenient drawback occurred and this was the tendency of glycolic acid nitrile to decompose at an elevated temperature.

It has been tried to get rid of the jellies formed in the solution by heating the solution to a more elevated temperature, but this requires heating to temperatures where the glycolic acid nitrile starts decomposing and this tendency to decompose creates other disturbances when the solution is used for spinning.

On the whole it proved impossible, according to the methods hitherto used, to obtain spinning solutions giving full satisfaction. Even the addition of other substances such as nitromethane to the glycolic acid nitrile did not counter-balance the drawbacks mentioned above.

I have now found that it is possible to produce fully satisfactory spinning solutions which do not display the tendency of forming jellies and from which high-grade threads or filaments can be obtained by spinning, if the polyacrylonitrile or copolymers thereof are dissolved in a well defined mixture of solvents. I have found that in order to obtain a fully satisfactory solvent, glycolic acid nitrile must be mixed with an adequate quantity of an aliphatic diester of the general formula

$$CH_3.(CH_2)_n.CO.O.CH_2.O.CO.(CH_2)_n.CH_3$$

in which n may be any numeral between 0 and 2.

Examples of the added subtances are methylene glycol diacetate, if in the formula cited hereabove n is 0; methylene glycol dipropionate if n is 1; and if n is 2, methylene glycol dibutyrate.

According to many tests I made the mixture to be used as a solvent has particularly favorable solvent properties if from 5 to 30 percent by weight and preferably from 10 to 15 percent by weight of the diester, calculated on the weight of the glycolic acid nitrile are added. If the solvent characterized hereabove is used in the production

of spinning solutions from polyacrylonitrile or copolymers thereof, the often observed tendency of the spinning solution to form a jelly and thereby creating undesirable disturbances will be found to have disappeared.

I have ascertained also that if this solvent mixture is used, the spinning solution itself as well as threads or filaments obtained therefrom are colorless and remain colorless.

In practicing my invention I may for instance proceed as follows.

Example 1

200 g of polyacrylonitrile having a K-value of 85 were stirred in 800 g of a solvent mixture consisting of 80 percent by weight of glycolic acid nitrile and 20 percent by weight of methylene glycol diacetate, at a temperature of about 15° C., to form a suspension. While the suspension was stirred, it was deaerated in a vacuum. After about one hour the deaerated mixture was heated gradually to a temperature ranging between 110° and 115° C. and stirring was continued for another hour at this temperature.

The spinning solution thus obtained was found to be satisfactory in every way and was then spun continuously according to the dry-spinning method.

The threads or filaments were stretched in hot condition in two steps, to from 8 to 10 times their original length. These threads or filaments were found to present a particularly uniform cross-section and to display a favorable strength of about 40 breaking km. at an elongation of between 7 and 8 percent.

Example 2

200 g of a copolymer consisting of 90 percent by weight of acrylonitrile and 10 percent of acrylic acid amide were stirred in cold condition together with 800 g of a solvent mixture containing 75 percent by weight of glycolic acid nitrile and 25 percent by weight of methylene glycol dibutyrate, to form a suspension which was then worked up, in accordance with Example 1. On being spun the solution thus obtained proved very satisfactory and the threads or filaments produced from it had a particularly favorable strength of about 45 breaking km. at an elongation of about 8 percent.

Various changes may be made in the proportions and compositions of the materials and solvents reported in the foregoing specification without departing from the invention or sacrificing the advantages thereof.

I claim:

1. The method of producing a spinning solution of a polymer of the group consisting of polyacrylonitrile and a copolymer of 90% by weight of acrylonitrile and 10% by weight of acrylic acid amide which comprises dissolving the said polymer in a solvent mixture consisting of glycolic acid nitrile and a diester of the general formula

$$CH_3.(CH_2)_n.CO.O.CH_2.O.CO.(CH_2)_n.CH_3$$

wherein n may be a numeral between 0 and 2.

2. The method of claim 1 in which the solvent mixture contains from 5 to 30% by weight of the diester calculated on the weight of the glycolic acid nitrile.

3. The method of claim 1 in which the solvent mixture contains from 10 to 15% by weight of the diester calculated on the weight of the glycolic acid nitrile.

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