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2,814,603

DIMETHYLFORMAMIDE-ALIPHATIC DIESTER SPINNING SOLUTIONS OF POLYACRYLONITRILES

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No Drawing. Application November 13, 1953, Serial No. 392,045

Claims priority, application Germany December 18, 1952

2 Claims. (Cl. 260—31.6)

This invention relates to the production of filaments from polyacrylonitrile or from mixed polymerisates containing polyacrylonitrile, dissolved in a solvent consisting of, or containing, dimethylformamide.

It is an object of this invention to provide a solvent for the polymerised compounds which is free from the tendency of allowing the matter dissolved therein to swell or form jellies and to thereby disturb the spinning operation.

It is a further object of this invention to provide a solvent for the materials mentioned above which does not lead to discoloration of the filaments.

It is a particularly important object of this invention to improve solutions in dimethylformamide, which is a particularly good solvent for the said materials, by doing away with any tendency of jelly-formation and discoloration.

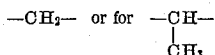
As is well known to those skilled in the art, the number of solvents suitable for practical use in the preparation of spinning solutions from polyacrylonitrile or mixed polymerisates containing this compound is particularly limited. It is equally known that dimethylformamide is on the whole a satisfactory solvent, but requires being improved in several directions. Thus, for instance, the dispersion which is first formed when trying to dissolve the polyacrylonitrile in dimethylformamide, causes a tendency of the nitrile to form a jelly and renders the spinning solution inhomogenous to the extent that the spinning process is disturbed and the filament breaks.

Apart from this, solutions of the polyacrylonitrile in dimethylformamide have hitherto suffered from slight discolorations of the spinning solution and of the filaments produced from them.

I have now found that altogether satisfactory spinning solutions containing dimethylformamide as predominant solvent can be obtained by adding to this solvent a certain quantity of an aliphatic diester constituted according to the formula



wherein R stands for



and n is an integer ranging from zero to two.

For instance, if R is replaced by the methylene group ($-\text{CH}_2-$), the compound to be added to the dimethylformamide is methyleneglycol-diacetate, methyleneglycol-dipropionate or methyleneglycol-dibutyrate.

If R is replaced by the ethylidene group



there are obtained the corresponding ethylidene compounds, viz. ethyleneglycol-diacetate, ethyleneglycol-dipropionate or ethyleneglycol-dibutyrate.

The mixtures of dimethylformamide with one of the

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substances aforesaid, for instance a mixture of dimethylformamide with methyleneglycol-diacetate, form with polyacrylonitrile or its mixtures with other polymerisates altogether satisfactory spinning solutions.

I have found that the best solvent is obtained if 5–20 percent by weight, and preferably 7–10 percent of the diester calculated on the weight of the dimethylformamide, are added to it.

When using solvent mixtures of the kind aforesaid in the preparation of spinning solutions from polyacrylonitrile or mixtures of the nitrile with other polymerisates, the tendency of the polymers for the formation of jellies is altogether done away with and the disturbances arising when operating with dimethylformamide alone are avoided altogether.

I have further found that the spinning solution is improved also in that neither the solutions nor the filament obtained with their aid do show any discoloration worth speaking of.

I have noticed also that the use of mixtures of the kind forming the object of this invention increases the stretching capacity of the filaments obtained from the solutions.

In operating this invention, I may, for instance, proceed as follows:

Example 1

22 parts by weight of polyacrylonitrile, having a K-value of 87, were added to 78 parts of a solvent mixture consisting of 85 percent by weight dimethylformamide and 15 percent methyleneglycol-diacetate.

The polymerisate was stirred according to well-known methods to form a dispersion which then dissolved in the solvent mixture under heating. The solution thus obtained yielded an unobjectionable liquid of high filament-forming capacity. The filaments spun from it had a particularly high stretching capacity and stretching velocities of 300–350 meters per minute could be employed. The filaments possessed particularly favorable mechanical properties and showed a resistance to breaking of 42 kilometers.

In comparison therewith, when using the same starting products, however, without the addition of methyleneglycol-diacetate the dispersion formed in the solvent showed an undesirable tendency of jelly-formation and the filaments obtained from the solution could be stretched only at the normal velocity of 200 meters per minute. Their resistance to breaking amounted to 38 kilometers.

Example 2

19 parts by weight of a co-polymer formed of 95 percent acrylonitrile and 5 percent vinylimidazol, having a K-value of about 92, was stirred to form a dispersion in 75 percent of a solvent mixture consisting of 80 percent by weight dimethylformamide and 20 percent ethylideneglycol-diacetate. After heating to fusion temperature a light-colored solution was obtained, which could be spun without any disturbances. The filaments from the solution could be stretched at a velocity of 250–300 meters per minute. The resistance to breaking extended to 40 kilometers.

Example 3

21 parts by weight of a co-polymer formed from 90 percent by weight acrylonitrile and 10 percent acrylic acid amide (K-value near 87) were dispersed in 79 parts of a solvent mixture formed of 82 percent dimethylformamide and 18 percent methyleneglycol-dipropionate.

In following the methods reported with reference to Example 1 a solution of high filament-forming capacity was obtained. The resistance of the filaments extended to about 42 kilometers.

Various changes may be made in the proportions and compositions of the materials and solvents reported in

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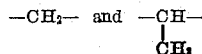
the foregoing specification without departing from the invention or sacrificing the advantages thereof.

I claim:

1. The process of preparing spinning solutions which comprises dissolving a member selected from the group consisting of polyacrylonitrile and a copolymer of acrylonitrile with another polymerizable unsaturated compound in minor proportion in a solvent comprising dimethylformamide in admixture with from 5 to 20 percent by weight of an aliphatic diester of the formula



wherein R is a member of the group consisting of



and n has the value zero to two inclusive.

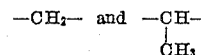
2. The process of preparing spinning solutions which

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comprises dissolving a member selected from the group consisting of polyacrylonitrile and a copolymer of acrylonitrile with another polymerizable unsaturated compound in minor proportion in a solvent comprising dimethylformamide in admixture with from 7 to 10 percent by weight of an aliphatic diester of the formula



wherein R is a member of the group consisting of



and n has the value zero to two inclusive.

References Cited in the file of this patent

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