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2,978,439

PROCESS FOR THE PURIFICATION OF POLYAMIDES

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No Drawing. Filed Mar. 13, 1958, Ser. No. 721,108

Claims priority, application Italy Mar. 20, 1957

4 Claims. (Cl. 260—78)

This invention relates to a process for the purification of polyamides and more particularly to the removal of monomeric and low molecular weight compounds from polyamides by treatment with aqueous solutions of hydrazine.

Polyamides from ω -amino-carboxylic acids or their polyamide-forming derivatives, such as lactams, alone and from mixtures of these materials with other polyamide-forming materials, such as diamines and dicarboxylic acids, usually contain a certain amount of unchanged amino-carboxylic acids or low molecular weight condensation products which exude during storage or during working up, for example, during the spinning of melts to thin threads thereby giving the polyamides an undesirable appearance. This can have an extremely deleterious effect due to the formation of vapours, especially of lactam vapours, and in the case of rather high content of vapourizable materials, can make the working up practically impossible.

In order to reduce the content of these monomeric and low molecular weight products in the polyamides to a suitably low amount, the polyamides are usually converted into chip form and boiled in water or the chips are washed for several hours with a stream of boiling hot water. The water, in both cases, possibly contains a small amount of a reducing agent such as hydrogen sulphite or sulphur dioxide. Nevertheless, a yellow colouration or even a deterioration of the polyamide can easily occur which not only effects the appearance of the polyamide but also reduces the mechanical properties of objects produced therefrom.

I have now found that such colouration or deterioration of polyamides by their treatment with hot water can be prevented in a technically simple and advantageous manner if small amounts, such as for example, about 10 to about 100 milligrams per litre of hydrazine or hydrazine hydrate are added to the water before it comes into contact with the polyamide.

If cold water is initially used, then it is recommended that the water be heated up for a short time to temperatures of, for example, between about 50 and about 80° C. after the addition of the hydrazine but before it is used for treating the polyamides.

Since the hydrazine does not leave behind either solid

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materials such as possible salts in the case of hydrogen sulphite, or corrosive or saponifying materials such as sulphuric acid from the addition of sulphur dioxide, and finally, since it does not in any way cause recognisable alteration of the polyamide, the process according to the invention also permits the production of polyamides of the highest quality.

The following example is given by way of illustration:

Example

Chips of a polyamide which has been obtained in a known manner from caprolactam are placed in a container in which a stream of water heated to 60° C. can be introduced from below. The container is provided with a heating device which heats the water to boiling point while it flows through the container. The water then runs away through an overflow. In order to reduce the content of monomeric and low molecular weight products in the polyamide to less than about 1 percent, a period of treatment of about 20 to 30 hours is necessary. If in this treatment of the polyamide, water is used without a special additive then the polyamide chips become yellow during the course of the treatment. This is especially true in the case of those chips lying in the proximity of the point of entry of the stream of water. If there are added 40 milligrams of hydrazine hydrate per litre of water before its admission to the heater, then the polyamide chips remain completely white. The polyamide treated according to the invention can be much more easily spun to a thread than a corresponding polyamide which has been treated with water without the addition of hydrazine. Furthermore, fibres made from a polyamide treated according to the invention have improved properties.

I claim:

1. Process for the purification of chips of high molecular weight polyamides selected from the group consisting of poly-E-aminocaproic acid and mixed polyamides of E-aminocaproic acid with polyamide forming diamines and dicarboxylic acids, containing impurities selected from the group consisting of monomeric E-caprolactam and low molecular weight condensation products thereof, which comprises washing said chips with boiling aqueous solutions of hydrazine, the amount of hydrazine being from about 10–100 mg./l. solution.

2. A process according to claim 1 wherein the aqueous treatment solution contains 40 milligrams of hydrazine per litre.

3. A process according to claim 1 wherein the solution is heated to a temperature within the range of 50–80° C. before being used to treat the polyamide.

4. A process according to claim 1 wherein the polyamide is polymerised caprolactam.

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