

# ALIEN PROPERTY CUSTODIAN

## PROCESS FOR THE PRODUCTION OF PRODUCTS OBTAINED FROM SUPERPOLYAMIDES

Franz Emich, Heppenheim, Germany; vested in the Alien Property Custodian

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The present invention concerns a process for the production of products obtained from superpolyamides.

It is known that superpolyamides are suitable for the production of a great variety of shaped products, as for example films, artificial threads and such like. Superpolyamides represent high molecular, polypeptide-like condensation products of for example  $\omega$ - $\omega$ -diamines and  $\omega$ - $\omega$ -dicarboxylic acids or  $\omega$ -aminocarboxylic acids or mixtures of these.

The superpolyamides known in the trade represent grains or lamellas containing about 7% of water. Pulverized superpolyamides have not yet been heard of, which fact probably is due to their extraordinary elasticity. It turned out to be exceedingly difficult to pulverize them by help of the usual methods.

The superpolyamides obtainable in commerce are worked up according to the following method: The lamellas are heated to melting temperature whereupon the melt, for example by die casting or press casting is given the desired shape. The production of a homogenous melt is very difficult. One nuisance consists in a formation of gas taking place. In the case of slight overheating considerable decompositions are observed which cause discoloration. In many cases the shaped products prepared from the melt have a porous surface and contain cavities. These impurities contained in the products known in the trade are transferred to the products with which they are to be worked up.

Thorough experiments have shown that working up of superpolyamides in order to obtain various products may be improved in every respect by admixing more water than is usually contained in air-dried superpolyamides. The superpolyamides usually contain about 7% of water. According to my invention objects, such as shaped products, powder and such like are produced from superpolyamides by first converting them into intermediate products rich in water, such as melts or pastes, whereupon these are subjected to a subsequent treatment. The water may be incorporated by heating the superpolyamides to suitable temperatures in the presence of water, preferably under water. The process is performed under pressure. In carrying out this process according to my invention I proceed as follows: The superpolyamides obtainable in the trade in lamella-form or perhaps in larger pieces are heated in pressure-vessels under water to melting temperatures or temperatures, which are lying above the melting point of the initial products.

Of course such temperatures are to be avoided which will cause damages, for example decompositions. I may, however, proceed in such manner that the superpolyamides are heated to only lower temperatures, which will just suffice for softening the superpolyamides and enable them to absorb water. This heating process is continued with until all of the desired amount of water is homogeneously taken up by the superpolyamides. According to the kind of superpolyamide employed, the temperatures and the duration of heating smaller or larger amounts of water may be incorporated with the superpolyamide, whereby the qualities of the melts or of the products prepared from the melts resp. may be influenced more or less. The heating of the superpolyamide may, for example, be interrupted after the softening of the melts has taken place, whereupon the heating may be carried on with for a shorter or longer time, in order to cause the superpolyamide to go on absorbing as much water as is desired.

It became evident that during the working process by which shaped products are to be obtained, the increase in the amount of water contained in the air-dry starting material is of great advantage. This content of water, which amounts to 7%, may be increased by a few percent only, for example 2-4%. With some starting materials the best results were obtained, if the increase in water amounted to 4-6%. For the preparation of powdery products it has proved to be advantageous to introduce considerably larger quantities of water into the intermediate products. Heating of the initial products under water according to the present invention is advantageous insofar as impurities of the starting products will be taken up by the water. Consequently purer final products are obtained. In some cases it has proved to be useful to add to the water as well other products, which may be mixed with water, such as glycerine and/or glycol.

The melts or pastes according to the invention to which water has been added, may be worked up in various manners. They may, for example, be shaped by casting, die casting or press casting. Another method consists in converting the melt to which water has been added, first, by lowering of the temperature, into a plastic kneadable mass, whereupon this mass is further worked up, for example by giving it shape and drying it. Another method consists in converting the intermediate products, to which water has been added, into powdery superpolyamides, which again may be worked up.

### Examples

1.  $\frac{1}{2}$  kg. of superpolyamide of adipic acid hexamethylene-diamine and  $\omega$ -aminocaproic acid of melting point  $180^{\circ}$  C. containing 7% of water, is heated to melting in a pressure vessel, the capacity of which amounts to about 4.5 liter, and which contains 2 liter of water. The necessary temperature of about  $180^{\circ}$  C. being reached the amount of water contained in the melt has gone up to 12%. The melt's consistency corresponds to, say, that of thin honey. This melt may preferably be used for the production of shaped products, for example for casting in molds, die casting, press casting or other means. Generally the working process should be proceeded with before any disturbing cooling down, for example to temperatures below  $100^{\circ}$  C. has taken place.

When cooling down to temperatures of about  $50^{\circ}$  C. or less the melt is getting plastic and rather viscous and may then be shaped. This shaping of the mass may be carried out according to usual methods. Products of any desired shape may be obtained. For example films may be prepared from the melt as well as the plastic mass, for example in such manner that first a rather thick film is prepared, whereupon this is treated between rollers or with other means until the desired thickness is obtained. I have found that the products prepared from the melt or the plastic mass practically did not show any disturbing shrinkage. The strength of the shaped products may be considerably increased by suitable after-treatment, for example by putting them into water of suitable temperature, for example of about  $60^{\circ}$  C.

Pasty intermediate products with an increased content of water may advantageously be worked up in such manner that they are kneaded or treated between rollers together with other substances, such as artificial substances, for example artificial rubber, based on butadiene, filling material, as for example carbon black. If desired several substances of various kind may be employed.

According to one embodiment of the invention the contents of the pressure vessel, after having been heated to its melting temperature of about  $180^{\circ}$  C. is kept on heating, whereby the same temperature is maintained. This method allows more water to be introduced. After about  $2\frac{1}{2}$  hours' heating the content of water of the now thinly liquid melt amounts to 33%. During cooling down this melt rich in water is turning into a moist product, which essentially consists of powdery particles which are sticking together. These may exceptionally easily be converted into powder by means such as crushing, pulverizing or grinding. If the mass is cooling down in a pressure vessel under water while it is being stirred a powdery product may be obtained immediately.

The obtained moist powder loses the superfluous water very easily. It may be converted

into a superpolyamide containing the normal amount of water, of for example about 7%, by keeping it at the air or by drying it at moderate temperatures, for example such below  $60^{\circ}$  C., if desired in vacuo. The physical qualities of the powdery products may be influenced by variation of the process as described as above with respect to temperature, duration of heating and amount of water to be introduced. The obtained powders are much better suited for various purposes than the lamella-formed or granulated products as are obtained in the trade. The powder may, for example, directly be shaped by only applying heat and pressure, if desired together with other substances. Further on it is much easier to convert powder into homogenous melts than lamellas or the like.

The moist powder, which is obtained at first, may advantageously be made use of as well. For example I found out that this powder may easily be worked up together with admixtures, for example artificial substances or filling material, as for example carbon black, zinc white or the like, or with a plurality of substances, for example artificial rubber based on butadiene, and filling material, by kneading, treating between rollers or the like, whereby homogenous products are obtained, which have excellent qualities.

During the production of such mixed products the quantitative relation may range in between wide limits. One may, for example, prepare products of artificial rubber, based on butadiene and superpolyamides, which obtain up to 90% and, if desired, even more superpolyamides, and which as well contain filling material of the usual kind. The amount of filling material may be varied as much as is wanted.

2.  $\frac{1}{2}$  kg. of a superpolyamide of adipic acid hexamethylene-diamine is heated to melting temperature, i. e. in this case to  $250^{\circ}$  C., in a pressure vessel under water under conditions as described in Example 1. When having reached melting temperature the melt, which is somewhat more viscous than the melt according to Example 1, contains about  $12\frac{1}{2}\%$  of water. The melt may be worked up according to Example 1 in order to obtain shaped products and the like. If the contents of the autoclave is kept on the same temperature of  $250^{\circ}$  C. the melt may take up larger quantities of water. After about 3 hours' heating the content of water of the melt amounts to 100%. The melt rich in water may, according to Example 1, be worked up in such manner that powdery products are obtained.

3.  $\frac{1}{2}$  kg. of the superpolyamide according to Example 2 is heated in a pressure vessel until it is soft and represents a viscous paste. After the paste is taken out of the pressure vessel it is immediately pressed into a mold, for example a wooden mold, whereby the product takes on the desired shape.

FRANZ EMICH.