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PROCESS FOR THE PRODUCTION OF OXALKYLATION PRODUCTS

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The present invention relates to a process for the production of oxalkylation products.

We have found that valuable oxalkylation products are obtained by causing polyamides or superpolyamides (under which term we comprise polycondensation products of compounds capable of yielding acid amides) to react with alkylene oxides.

As polyamides and superpolyamides suitable for the purposes of the present invention we mention those which are obtainable from $\omega.\omega'$-diamines and dicarboxylic acids or from aminocarboxylic acids. For example the polyamides or superpolyamides from tetramethylenediamine, pentamethylenediamine, hexamethylenediamine or aliphatic diamines of a still higher molecular weight, or mixtures of these diamines and succinic acid, glutaric acid, adipic acid, pimelic acid or dicarboxylic acids of a still higher molecular weight or mixtures of such dicarboxylic acids are suitable. Polyamides and superpolyamides obtainable by the polycondensation of glycocoll and higher aminocarboxylic acids, such as aminocaproic acid, or their lactams, or mixed condensation products are also well adapted for being reacted with alkylene oxides.

The reaction can be practiced by heating the polyamides or superpolyamides together with one or several alkylene oxides, for example with ethylene oxide or propylene oxide, preferably while using the alkylene oxides in an excess quantity, in a closed vessel, or by introducing the alkylene oxides into the molten polyamides or superpolyamides or their solutions in organic solvents. The reaction generally takes place already at comparatively low temperatures, for example at between 80 and 150°C. In the case of melts higher temperatures must be used, depending on the melting point of the initial materials.

By the treatment with alkylene oxides polyalkylene-glykol radicles are introduced into the polyamide or superpolyamide molecules. Depending on the quantity and the type of the alkylene oxide and on the nature of the initial material used products of varying properties are obtained. Generally speaking, the oxalkylation will lower the melting point and increase the solubility in water.

The products obtained by the oxalkylation of polyamides and superpolyamides are adapted for quite a variety of applications, for example as softeners for plastics, especially those obtained from polyamides and superpolyamides. With great success they may be used as softeners for films or film-like structures of water sensitive cellulosic materials such as regenerated cellulose. They can also be used for the improvement of textiles, paper, leather and other fibrous materials, especially artificial fibres; for these latter purposes oxalkylation products from polyamides or superpolyamides which are soluble or readily dispersible in water are especially suitable. They are, for instance, useful as finishing, sizing, equalizing, softening, dispersing or impregnating agents. They may be employed by the manner commonly known in using other textile assistants for similar purposes.

The following examples serve to illustrate how the present invention may be carried out in practice, but the invention is not restricted to these examples. The parts are by weight.

Example 1

10 parts of a polyamide obtained by heating an 80 per cent aqueous solution of 1 mol of ϵ-aminocaprolactam for 5 hours in the presence of $\frac{1}{50}$ mol of acetic acid is heated, after removing the water by distillation, with 50 parts of ethylene oxide at from 100 to 120°C for 12 hours. After distilling off the excess ethylene oxide a swollen gelatinous mass is left behind, the chief part of which dissolves in water and which is suitable for use as an adhesive or softener for plastics and as a thickening agent for printing pastes.

Example 2

10 parts of a mixed polyamide obtained by the mixed condensation of 50 parts of hexamethylenediammonium adipinate with 50 parts of ϵ-aminocaprolactam are heated with 100 parts of ethylene oxide at from 120 to 150°C for 15 hours. After removing the excess ethylene oxide a faintly yellowish jelly remains which dissolves in water forming a clear solution. When replacing the ethylene oxide by an equivalent amount of propylene oxide, products showing similar properties are obtained.

Example 3

100 parts of the high-molecular pulverulent product obtainable by heating 1 mol of hexamethylene diisocyanate with 1 mol of 1.4-butyleneglycol in the presence of orthodichlorbenzene are heated together with 500 parts of ethylene oxide to 150° C for 10 hours. A yellow viscous oil is obtained which dissolves in alcohols forming clear solutions.

Example 4

Untreated skeins of viscose artificial silk are treated in a bath (at a ratio of 1:20) containing

1 gram of the product according to Example 2 to each liter, at room temperature, whereupon the material is centrifuged and dried. By this treatment the artificial silk is imparted a full and soft touch, otherwise obtainable only with products prepared on a fatty acid basis.

Example 5

Skeins of viscose artificial silk are treated in a bath containing per liter 75 grams of the oxalkylation product employed in Example 4, at room temperature, centrifuged and dried, whereby an excellent sizing effect is obtained. Each thread of the so treated artificial silk is perfectly closed, elastic and resistant to mechanical treatment so that it is very well adapted for weaving. The size can easily be removed by means of warm water. The sizing effect is equal to one obtained with a size from linseed oil from benzine solution, but the size obtained according to this invention stands storage and does not resinify.

Example 6

Cotton is dyed with 0.5 per cent of Indanthrene Brilliant Green 5G (cf. G. Schulz, Farbstofftabellen 1931, Nr.1269) as usual in a bath which contains 0.25 gram of the oxalkylation product employed in Example 4 per each liter of bath. A very uniform dyeing is thus obtained.

Example 7

Woollen cloth is dyed with 3 per cent of a complex chromium compound of an acid dyestuff and from 5 to 10 per cent of sulphuric acid in a bath (ratio 1:50) which contains per each liter 1 gram of the oxalkylation product according to Example 2, at boiling temperature for 1 hour. A well developed, very uniform dyeing is obtained which is perfectly fast to rubbing.

Example 8

The oxalkylation product according to Example 2 is dissolved in water of 40 degrees hardness (German scale) at the rate of 0.5 gram to each liter, whereupon soap is added to the solution at the rate of 2 grams to each liter. While the solution shows slight turbidity, there is no precipitation of lime soap.

Similarly may soap solutions be rinsed out of textile materials by admixing each liter of the hard water with which rinsing is done with 0.5 gram of the product according to Example 2. Lime soap which has already precipitated can also be redissolved by the addition of the said product.

Similar results are obtained by using another oxalkylation product of a polyamide or superpolyamide, for example an equal amount of the oxethylation product of the polyamide or superpolyamide from ϵ-aminocaprolactam or from adipic acid and $\omega.\omega'$-diaminooctane.

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