i) polycondensing a dicarboxylic acid component comprising terephthalic acid, and a diamine component having a 1,9-nonanediamine and/or 2-methyl-1,8-octanediamine content of 60 to 100 mol% in the presence of 15 to 35% by weight of water at a reaction temperature of 250 to 280°C and a reaction pressure which satisfies the following equation:

$$P_0 \geq P \geq 0.7\, P_0$$

where P_0 is the saturation vapor pressure of water at the reaction temperature,

to obtain a primary polycondensate,

(ii) discharging the primary polycondensate from step i) in an atmospheric environment with the same temperature range and at the same water content as in step i),

(iii) increasing the molecular weight by subjecting the discharge from step ii) to a solid state polymerization or a melt polymerization.

It is an object of the present invention to provide an improved process for preparing polyamides. More particularly, the transition from the oligomerization at lower temperatures in the presence of a high water content to the polymerization at higher temperatures in the presence of a low water content is to be effected under moderate conditions, especially at minimum pressures. The process is to lead to polyamides having the desired high molecular weight without any requirement for costly and inconvenient postpolymerization steps. The polyamides thus obtained are to feature advantageous product properties, more particularly not too broad a molecular weight distribution and/or a low gel content. Moreover, the typical disadvantages of a batchwise process, such as limitation of the batch size, loss of time resulting from filling, emptying and cleaning of the reaction vessel, etc. are also to be avoided.

It has been found that, surprisingly, this object is achieved by the process according to the invention, in which a starting mixture having a low water content is used for the oligomerization, the reaction mixture is subjected to at least one expansion to reduce the water content in the course of the polycondensation, and the final polymerization is effected at a temperature above the melting temperature of the aliphatic or semiaromatic polyamide.

SUMMARY ~~OF THE INVENTION~~

The disclosure ~~invention firstly~~ provides a process for continuously preparing an aliphatic or semiaromatic polyamide, in which

a) an aqueous composition comprising at least one component which is suitable for polyamide formation and is selected from dicarboxylic acids, diamines, salts of at least one dicarboxylic acid and at least one diamine, lactams, ω-amino acids, aminocarbonitriles and mixtures thereof is provided, and the composition provided is supplied to an oligomerization zone,

b) in the oligomerization zone, the composition is subjected to an oligomerization at a temperature of 170 to 290°C and an absolute pressure of at least 20 bar and a liquid output comprising the polyamide oligomers is withdrawn from the oligomerization zone,

c) the output from the oligomerization zone is optionally fed into a flash zone E1) and subjected to expansion to obtain a water-containing gas phase and a liquid phase comprising the polyamide oligomers, and at least a portion of the water-containing gas phase is removed,

d) the liquid output from the oligomerization zone or the liquid phase from the flash zone E1) is subjected to rapid heating to a temperature above the melting temperature Tm_2 of the aliphatic or semiaromatic polyamide, and

e) the heated composition from step d) is fed into a flash zone E2) and expanded to obtain a water-containing gas phase and a polyamide-containing liquid phase, at least a portion of the water-containing gas phase is removed and the polyamide-containing phase is subjected to a postpolymerization at a temperature above the melting temperature Tm_2 of the aliphatic or semiaromatic polyamide.

In a first aspect, there is provided a process for continuously preparing an aliphatic or semiaromatic polyamide, in which

a) an aqueous composition comprising at least one component which is suitable for polyamide formation and is selected from dicarboxylic acids, diamines, salts of at least one dicarboxylic acid and at least one diamine, lactams, ω-amino acids, aminocarbonitriles and mixtures thereof is provided, and the composition provided is supplied to an oligomerization zone,

b) in the oligomerization zone, the composition is subjected to an oligomerization at a temperature of 170 to 290°C and an absolute pressure of at least 20 bar, the oligomerization in step b) being effected without mass transfer with the environment, and a liquid output comprising the polyamide oligomers is withdrawn from the oligomerization zone,

c) the output from the oligomerization zone is optionally fed into a flash zone E1) and subjected to expansion to obtain a water-containing gas phase and a liquid phase comprising the polyamide oligomers, and at least a portion of the water-containing gas phase is removed,

d) the liquid output from the oligomerization zone or the liquid phase from the flash zone E1) is subjected to rapid heating to a temperature above the melting temperature Tm_2 of the aliphatic or semiaromatic polyamide, and

e) the heated composition from step d) is fed into a flash zone E2) and expanded to obtain a water-containing gas phase and a polyamide-containing liquid phase, at least a portion of the water-containing gas phase is removed and the polyamide-containing phase is subjected to a postpolymerization at a temperature above the melting temperature Tm_2 of the aliphatic or semiaromatic polyamide.

The ~~invention~~ disclosure further provides polyamide oligomers obtainable by a process as defined above and hereinafter.

In another aspect, there is provided a polyamide obtainable by a process as defined above and hereinafter, and a molding composition obtainable therefrom by compounding.

The ~~invention~~ disclosure further provides a process for preparing a polyamide, in which a polyamide oligomer obtainable by a process as defined above and hereinafter is subjected to a further polymerization. The ~~invention~~ disclosure also provides the polyamides thus obtainable.

~~The invention further provides~~In another aspect, there is provided a polyamide molding composition comprising at least one polyamide, obtainable by a process as defined above and hereinafter. The ~~invention~~ disclosure further provides a molding produced from such a polyamide molding composition.

In another aspect, there is provided a molding produced from a polyamide molding composition as defined above and hereinafter.

The ~~invention~~ disclosure further provides for the use of a semiaromatic polyamide obtainable by a process as defined above and hereinafter, preferably for production of electrical and electronic components and for high-temperature automotive applications.

In another aspect, there is provided the use of a semiaromatic polyamide obtainable by a process as defined above and hereinafter for production of electrical and electronic components and for high-temperature automotive applications.

~~The invention further provides for~~In another aspect, there is provided the use of an aliphatic polyamide obtainable by a process as defined above and hereinafter for production of films, monofilaments, fibers, yarns or textile fabrics.

BRIEF DESCRIPTION OF THE FIGURES

Figure 1 shows a simple apparatus for performance of the process according to the invention, comprising

- a mixing vessel for provision of the aqueous composition comprising at least one component suitable for polyamide formation (in the specific implementation, the aqueous composition is preferably provided in a mixing vessel and transferred into a reservoir vessel for continuous feeding),
- a tubular reactor as an oligomerization zone (in a departure from the specific implementation, other reactors can also be used for oligomerization, as described in detail hereinafter),
- a draw point from the oligomerization zone with an apparatus for pressure reduction,
- a heat exchanger for rapid heating of the reaction mixture,

- a flash tank E2) for separation of a water-containing gas phase and for postpolymerization of the reaction mixture, without forming a polyamide-containing solid phase.

Figure 2 shows a preferred embodiment of an apparatus for performance of the process according to the invention, comprising