increases. In order to arrive at an acceptable rate the particles must be uneconomically small. Therefore, there is a need for a method that ensures that the esterification reaction of 2,5-furandicarboxylic acid with ethylene glycol does not yield "over-esterified" polyester with an inadequate level of carboxylic end groups. It has now been found that the extent of the esterification reaction can be controlled by using a dimensionless parameter.

Summary

In one aspect the present invention provides a method for the preparation of a polyester, wherein a starting mixture comprising 2,5-furandicarboxylic acid and ethylene glycol is subjected to esterification to form an ester composition, and the ester composition thus obtained is subjected to polycondensation at reduced pressure in the presence of a polycondensation catalyst to obtain a polycondensate, wherein 2,5-furandicarboxylic acid and ethylene glycol are reacted under conditions such that the esterification potential as defined by Esterification Potential (EsPo) = $(MR-1)^2 * P_{H2O}(T)$, wherein MR represents the molar ratio of ethylene glycol over 2,5-furandicarboxylic acid, MR being greater than 1; $P_{H2O}(T)$ represents the pure component vapor pressure (in bar) of water at temperature T, which is the final reaction temperature in the esterification mixture before the pressure is reduced to effect the polycondensation, is less than 0.8.

Description

Accordingly, the present invention provides a method for the preparation of a polyester, wherein a starting mixture comprising 2,5-furandicarboxylic acid and ethylene glycol is subjected to esterification to form an ester composition, and the ester composition thus obtained is subjected to polycondensation at reduced pressure in the presence of a polycondensation catalyst to obtain a polycondensate,

wherein 2,5-furandicarboxylic acid and ethylene glycol are reacted under conditions such that the esterification potential as defined by

Esterification Potential (EsPo) = $(MR-1)^2*P_{H2O}(T)$,

wherein MR represents the molar ratio of ethylene glycol over 2,5-furandicarboxylic acid, MR being greater than 1;

$P_{H2O}(T)$ represents the pure component vapor pressure (in bar) of water at temperature T, which is the final reaction temperature in the esterification mixture before the pressure is reduced to effect the polycondensation,

is less than 0.8.

The Antoine equation

$\log_{10} P = A - B/(C + T)$, where T is the temperature at the end of esterification, expressed in °C, A = 5.2594, B = 1810.94, and C = 244.485 gives the required vapor pressure of pure water in bar.

It has been found that the best results as to polycondensate are obtained if the esterification potential is at least 0.03, preferably from 0.05 to 0.5.

It has been found that when the preparation is conducted in accordance with the present invention a level of carboxylic end groups is obtained, typically in the range of 0.10 to 0.70 calculated as the number of carboxylic end groups divided by the sum of carboxylic end groups and hydroxyl end groups. It has further been found that when solid particles of the polyester prepared according to the invention are subjected to solid state polymerization the duration of the solid state polymerization can be shortened considerably.

In general there are a number of methods to determine the end groups in polyesters. Such methods include titration, infrared and nuclear magnetic resonance (NMR) methods. Often the separate methods are used to quantify the four main end groups: carboxylic acid end groups, hydroxyl end groups, alkyl ester groups, such as the methyl ester end groups (for polyesters from the dialkyl ester of a dicarboxylic acid) and the end groups that are obtained after decarboxylation. A.T Jackson and D.F. Robertson have published an ^{1}H-NMR method for end group determination in "Molecular Characterization and Analysis of Polymers" (J.M.