improvement is described in WO 2013/062408, wherein the dimethyl ester of FDCA is transesterified with ethylene glycol, or bis(2-hydroxyethyl)-2,5-furandicarboxylate is used as starting material. The transesterification product or this starting material is then subjected to polycondensation and after a drying/crystallization step the polycondensate is subjected to solid state polymerization to enhance the molecular weight of the polycondensate. In an example the polycondensate was ground and sieved and the fraction with particle size 0,6 - 1.4 mm was crystallized overnight at 110°C overnight. This fraction was subjected to solid state polymerization which took two days.

In WO 2013/120989 a continuous process for the preparation of poly(ethylene furandicarboxylate) is described wherein FDCA or a diester thereof is mixed with ethylene glycol at elevated temperature to give a paste or a homogeneous solution, the paste or solution is converted to an esterification product of FDCA and ethylene glycol, the esterification product is polycondensed under reduced pressure, wherein the polycondensation is performed in two stages. According to an example the dimethyl ester of FDCA was reacted with ethylene glycol in a molar ratio of 1:1.7. In this example the stages following the production of the esterified product took 5 hours. The polycondensation product can be subjected, if desired, to a solid stating polymerization. However, no details are provided. In an example the polycondensation product was reported to have an intrinsic viscosity of 1.05 dL/g.

KR 20140003167 describes a polyester polymer with excellent transparency which is manufactured by using a biomass originated furandicarboxylate ester compound with ethylene glycol. In comparative examples also furandicarboxylic acid has been used. The molar ratio of furandicarboxylate ester to ethylene glycol may be from 1:1.1 to 1:4. The ratio of furandicarboxylic acid to ethylene glycol varies between1:1.2 to 1:2. The document does not disclose a solid state polymerization step.

Summary

In one aspect the present invention provides a process for enhancing the molecular weight of a semi-crystalline starting polyester comprising ethylene 2,5-furandicarboxylate units, by heating the semi-crystalline starting polyester , having a melting point Tm, at a temperature in the range of (Tm-40°C) to Tm to obtain a solid stated polyester, wherein the semi-crystalline starting polyester has an intrinsic viscosity of at least 0.45 dL/g, and an amount of carboxylic acid end groups in the range of 15 to 122 meq/kg.

Description

It is apparent that it would be beneficial if the process could be altered such that a faster enhancement of the molecular weight of such polyesters could be obtained. It has now surprisingly been found that when a semi-crystalline polyester having a particular structure is subjected to solid-state polymerization, the solid state polymerization proceeds faster than as reported in the above-mentioned patent specifications.

Accordingly, the present invention provides a process for enhancing the molecular weight of a semi-crystalline starting polyester comprising ethylene 2,5-furandicarboxylateunits by heating the semi-crystalline starting polyester, having a melting point Tm, at a temperature in the range of (Tm-40°C) to Tm to obtain a solid stated polyester, wherein the semi-crystalline starting polyester has an intrinsic viscosity of at least 0.45 dl/g, and an amount of carboxylic acid end groups in the range of 15 to 122 meq/kg. Preferably, the semi-crystalline starting polyester has an intrinsic viscosity of at most 1.0 dL/g.

sum of the molar amounts of hydroxyl end groups and carboxylic acid end groups is in the range of 0.14 to 0.65.

10. Process according to any one of claims 1 to 9, wherein the semi-crystalline starting polyester is contained in a composition that further comprises one or more esterification and/or polycondensation catalysts.

11. Process according to claim 10, wherein the esterification and/or polycondensation catalysts have been selected from the esterification and polycondensation catalysts comprising a metal selected form the group consisting of titanium, tin, zinc, calcium, antimony, germanium, manganese, cobalt, lead, magnesium, aluminium, cerium, zirconium and hafnium.

12. Process according to any one of claims 1 to 11, wherein the semi-crystalline starting polyester is in the form of pellets having a mass of 2.8 to 28 mg.

13. Process according to any one of claims 1 to 12, wherein the semi-crystalline starting polyester is in the form of pellets having a particle size selected such that there are 40 to 350 particles per gram.

14. Process according to any one of claims 1 to 13, wherein the semi-crystalline starting polyester is heated at a temperature in the range of (Tm-40°C) to Tm for a period of at most 120 hr, preferably from 2 to 60 hr.

15. Process according to any one of claims 1 to 14, wherein the semi-crystalline starting polyester is heated to a temperature in the range of 180 to 220°C to obtain the solid stated polyester.