Claims

1. A process for preparing a poly(ethylene-2,5-furandicarboxylate) polymer having a number average molecular weight of at least 25,000 (as determined by GPC based on polystyrene standards), wherein the process comprises
(a) a first step which is
(i) a transesterification step wherein dimethyl-2,5-furandicarboxylate ester (diester) is transesterified with ethylene glycol (diol) in the presence of a transesterification catalyst;
or
(ii) providing a molten reaction mixture comprising bis(2-hydroxyethyl)-2,5-furandicarboxylate;
(b) a polycondensation step at reduced pressure, preferably below 1 mbar and at elevated temperature, preferably below 240 °C, wherein under melt conditions the product prepared in step (a) is reacted in the presence of a polycondensation catalyst and removing the condensate from the reactor;
(c) drying and/or crystallizing the obtained condensate at a temperature from about 90 to about 160 °C ; and
(d) subjecting the polymer of step (c) to post condensation conditions comprising a elevated temperature treatment ending at a temperature of at least 190 °C to thereby obtain a poly(ethylene-2,5-furandicarboxylate) polymer having a number average molecular weight of at least 25,000.

2. The process according to claim 1, wherein the diol and diester are used in a diol to diester molar ratio of 1.5 – 3.0, more preferably of 2.0 – 2.5.

3. The process according to any one of claims 1-2, wherein the transesterification is performed at a temperature from about 150 to about 230 °C.

4. The process according to any one of claims 1-3, wherein methanol is removed from the step (a)(i) up to a temperature of 210-230 °C.

5. The process according to any one of claims 1-4, wherein the transesterification catalyst is a calcium or zinc catalyst and the polycondensation catalyst is an antimony catalyst.

6. The process according to any one of claims 1-5, wherein the number average molecular weight of the condensate of step (b) is between 13,000 and 20,000.

7. The process according to any one of claims 1-6, wherein after removal from the reactor the condensate is cooled and shaped into pellets for subsequent solid stating step.

8. The process according to any one of claims 1-7, wherein the conditions in step (d) further comprise providing an inert gas or vacuum.

9. The process according to any one of claims 1-8, wherein the drying and/or crystallisation is performed at a temperature of from about 90 °C to about 145 °C , wherein preferably the temperature is increased by 5 to 25 °C per hour.

10. The process according to any one of claims 1-9, wherein the elevated temperature treatment of step (d) comprises a temperature treatment starting at about 180 °C until a temperature of at least 205 °C.

11. The process according to any one of claims 1-10, wherein the number average molecular weight is determined by GPC based on polystyrene standards.

12. The process according to any one of claims 1-11, wherein the polymer obtained after step (d) has an absorbance as a 5 mg/mL solution in a dichloromethane:hexafluoroisopropanol 8:2 at 400 nm of below 0.05, without intermediate or subsequent purification and/or washing step.

13. The process according to any one of claims 1-12, wherein the polymer is subjected to strain hardening to improve mechanical properties.

14. A polyester-containing bottle or film or fibre-containing (woven or non-woven) object made from melt-processing poly(ethylene-2,5-furandicarboxylate) wherein the poly(ethylene-2,5-furandicarboxylate) is obtainable by the process as defined in any one of claims 1-12.

15. An object according to claim 14 that has been subjected to a step comprising strain hardening improve mechanical properties.

16. The object according to claim 14 or 15, which is an injection stretch blow moulded bottle.

17. The object according to claim 14 or 15, which is biaxially oriented film.

18. The object according to claim 14 or 15, which is a drawn fibre.

19. Process according to any one of claims 1-12, wherein up to 10 mol. % of a diol not being ethylene glycol and/or diacid not being 2,5-furandicarboxylate is present in step (a(i)) and/or step (a(ii)).

AMENDED CLAIMS
received by the International Bureau on 15 March 2013 (15.03.2013)

1. A process for preparing a poly(ethylene-2,5-furandicarboxylate) polymer having a number average molecular weight of at least 25,000, as determined by GPC based on polystyrene standards, wherein the process comprises
(a) a first step which is
(i) a transesterification step wherein dimethyl-2,5-furandicarboxylate ester (diester) is transesterified with ethylene glycol (diol) in the presence of a transesterification catalyst;
or
(ii) providing a molten reaction mixture comprising bis(2-hydroxyethyl)-2,5-furandicarboxylate;
(b) a polycondensation step at reduced pressure, preferably below 1 mbar and at elevated temperature, preferably below 240 °C, wherein under melt conditions the product prepared in step (a) is reacted in the presence of a polycondensation catalyst and removing the condensate from the reactor;
(c) drying and/or crystallizing the obtained condensate at a temperature from 90 to 160 °C ; and
(d) subjecting the polymer of step (c) to post condensation conditions comprising a elevated temperature treatment ending at a temperature of at least 190 °C to thereby obtain a poly(ethylene-2,5-furandicarboxylate) polymer having a number average molecular weight of at least 25,000.

2. The process according to claim 1, wherein the diol and diester are used in a diol to diester molar ratio of 1.5 – 3.0, more preferably of 2.0 – 2.5.

3. The process according to any one of claims 1-2, wherein the transesterification is performed at a temperature from 150 to 230 °C.

4. The process according to any one of claims 1-3, wherein methanol is removed from the step (a)(i) up to a temperature of 210-230 °C.

5. The process according to any one of claims 1-4, wherein the transesterification catalyst is a calcium or zinc catalyst and the polycondensation catalyst is an antimony catalyst.

6. The process according to any one of claims 1-5, wherein the number average molecular weight of the condensate of step (b) is between 13,000 and 20,000.

7. The process according to any one of claims 1-6, wherein after removal from the reactor the condensate is cooled and shaped into pellets for subsequent solid stating step.

8. The process according to any one of claims 1-7, wherein the conditions in step (d) further comprise providing an inert gas or vacuum.

9. The process according to any one of claims 1-8, wherein the drying and/or crystallisation is performed at a temperature of from 90 °C to 145 °C , wherein preferably the temperature is increased by 5 to 25 °C per hour.

10. The process according to any one of claims 1-9, wherein the elevated temperature treatment of step (d) comprises a temperature treatment starting at 180 °C until a temperature of at least 205 °C.

11. The process according to any one of claims 1-10, wherein the number average molecular weight is determined by GPC based on polystyrene standards.

12. The process according to any one of claims 1-11, wherein the polymer obtained after step (d) has an absorbance as a 5 mg/mL solution in a dichloromethane:hexafluoroisopropanol 8:2 at 400 nm of below 0.05, without intermediate or subsequent purification and/or washing step.

13. The process according to any one of claims 1-12, wherein the polymer is subjected to strain hardening to improve mechanical properties.

14. A polyester-containing bottle or film or fibre-containing (woven or non-woven) object made from melt-processing poly(ethylene-2,5-furandicarboxylate) wherein the poly(ethylene-2,5-furandicarboxylate) is obtainable by the process as defined in any one of claims 1-12.

15. An object according to claim 14 that has been subjected to a step comprising strain hardening improve mechanical properties.

16. The object according to claim 14 or 15, which is an injection stretch blow moulded bottle.

17. The object according to claim 14 or 15, which is biaxially oriented film.

18. The object according to claim 14 or 15, which is a drawn fibre.

19. Process according to any one of claims 1-12, wherein up to 10 mol. % of a diol not being ethylene glycol and/or diacid not being 2,5-furandicarboxylate is present in step (a(i)) and/or step (a(ii)).

AMENDED CLAIMS
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1. A process for preparing a poly(ethylene-2,5-furandicarboxylate) polymer having a number average molecular weight of at least 25,000, as determined by GPC based on polystyrene standards, wherein the process comprises

(a) a first step which is

(i) a transesterification step wherein dimethyl-2,5-furandicarboxylate ester (diester) is transesterified with ethylene glycol (diol) in the presence of a transesterification catalyst;

or

(ii) providing a molten reaction mixture comprising bis(2-hydroxyethyl)-2,5-furandicarboxylate;

(b) a polycondensation step at reduced pressure, preferably below 1 mbar and at elevated temperature, preferably below 240 °C, wherein under melt conditions the product prepared in step (a) is reacted in the presence of a polycondensation catalyst and removing the condensate from the reactor;

(c) drying and/or crystallizing the obtained condensate at a temperature from 90 to 160 °C ; and

(d) subjecting the polymer of step (c) to post condensation conditions comprising a elevated temperature treatment ending at a temperature of at least 190 °C to thereby obtain a poly(ethylene-2,5-furandicarboxylate) polymer having a number average molecular weight of at least 25,000.

2. The process according to claim 1, wherein the diol and diester are used in a diol to diester molar ratio of 1.5 – 3.0, more preferably of 2.0 – 2.5.

3. The process according to any one of claims 1-2, wherein the transesterification is performed at a temperature from 150 to 230 °C.

4. The process according to any one of claims 1-3, wherein methanol is removed from the step (a)(i) up to a temperature of 210-230 °C.

5. The process according to any one of claims 1-4, wherein the transesterification catalyst is a calcium or zinc catalyst and the polycondensation catalyst is an antimony catalyst.

6. The process according to any one of claims 1-5, wherein the number average molecular weight of the condensate of step (b) is between 13,000 and 20,000.

7. The process according to any one of claims 1-6, wherein after removal from the reactor the condensate is cooled and shaped into pellets for subsequent solid stating step.

8. The process according to any one of claims 1-7, wherein the conditions in step (d) further comprise providing an inert gas or vacuum.

9. The process according to any one of claims 1-8, wherein the drying and/or crystallisation is performed at a temperature of from 90 °C to 145 °C , wherein preferably the temperature is increased by 5 to 25 °C per hour.

10. The process according to any one of claims 1-9, wherein the elevated temperature treatment of step (d) comprises a temperature treatment starting at 180 °C until a temperature of at least 205 °C.

11. The process according to any one of claims 1-10, wherein the number average molecular weight is determined by GPC based on polystyrene standards.

12. The process according to any one of claims 1-11, wherein the polymer obtained after step (d) has an absorbance as a 5 mg/mL solution in a dichloromethane:hexafluoroisopropanol 8:2 at 400 nm of below 0.05, without intermediate or subsequent purification and/or washing step.

13. The process according to any one of claims 1-12, wherein the polymer is subjected to strain hardening to improve mechanical properties.

14. A polyester-containing bottle or film or fibre-containing (woven or non-woven) object made from melt-processing poly(ethylene-2,5-furandicarboxylate) wherein the poly(ethylene-2,5-furandicarboxylate) is obtainable by the process as defined in any one of claims 1-12.

15. An object according to claim 14 that has been subjected to a step comprising strain hardening improve mechanical properties.

16. The object according to claim 14 or 15, which is an injection stretch blow moulded bottle.

17. The object according to claim 14 or 15, which is biaxially oriented film.

18. The object according to claim 14 or 15, which is a drawn fibre.

19. Process according to any one of claims 1-12, wherein up to 10 mol. % of a diol not being ethylene glycol and/or diacid not being 2,5-furandicarboxylate is present in step (a(i)) and/or step (a(ii)).