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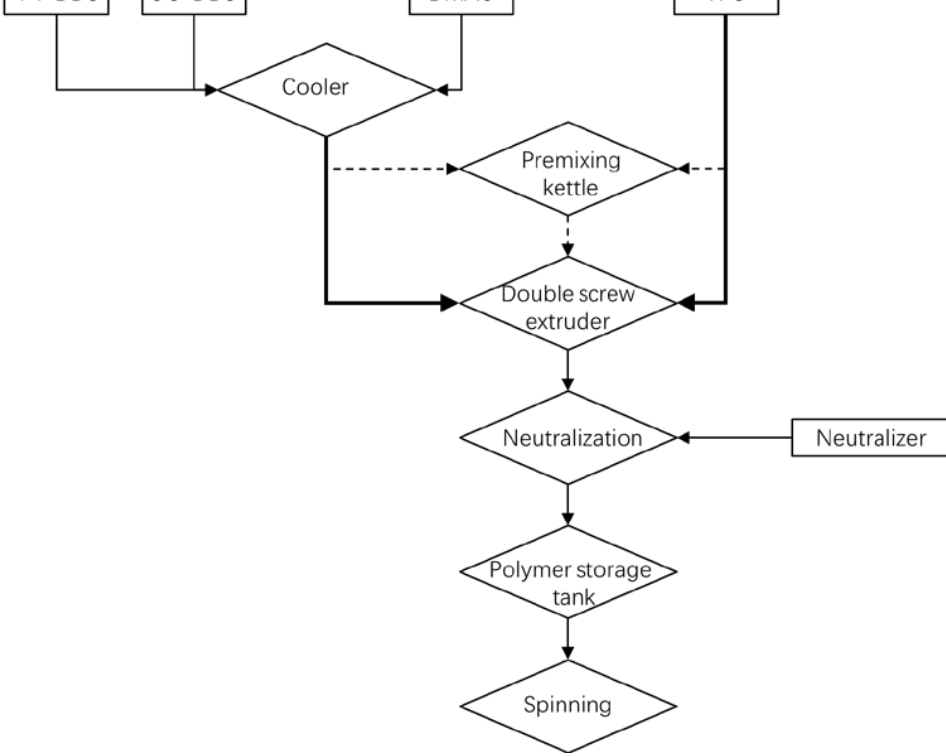
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[54] Invention Title

Method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder

[57] Abstract

The present invention relates to a method for continuous preparation of polysulfone amide spinning solution, wherein 4'4-DDS and 3' 3-DDS by mass ratio 10:90 ~ 90:10 are dissolved in DMAC polar solvent, cooled down in a cooler, then the reactants, pre-polymerized or not, are loaded into a twin-screw extruder which functions as main reactor and remained for 10 ~ 25 minutes. Polysulfone amide containing hydrogen chloride is extruded, and neutralized with calcium hydroxide or calcium oxide. The obtained polymer solution can be directly applied for spinning. The use of the twin-screw extruder in the present invention is beneficial for solving heat dissipation problem of the system, avoiding the inhomogeneity of the polymer molecular weight, as well as helpful to partially remove hydrogen chloride gas, the low molecular weight byproduct from the polymerization system.



1. A method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder, wherein it comprises following steps:

(a) Dissolution: 4' 4-diaminodiphenylsulfone and 3' 3-diaminodiphenylsulfone by mass ratio of 10:90 ~ 90:10 are dissolved in N, N'-dimethylacetamide solvent, and cooled down in a cooler;

(b) Pre-polymerization: the solution in step (a) and paraphthaloyl chloride are pre-polymerized, generating a prepolymer product containing hydrogen chloride byproduct;

(c) Polymerization: the prepolymer is loaded into a twin-screw extruder, which is used as a main reactor, retained for 10 ~ 25 minutes and the temperature is divided into three zones with temperature at -5 to 5 °C, 0 to 20 °C and 10 to 100 °C, respectively. The polysulfone amide containing hydrogen chloride is extruded.

(d) Neutralization: the polysulfone amide containing hydrogen chloride generated in step (c) is neutralized with calcium hydroxide or calcium oxide, and the obtained polymer solution can be directly applied for spinning.

2. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder according to Claim 1, wherein the concentration of 4' 4-diaminodiphenylsulfone and 3' 3-diaminodiphenylsulfone in the polymerization system is 0.3 ~ 0.7 mol/L, and the pre-polymerization temperature is -20 to 20 °C.

3. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder according to Claim 1 or 2, wherein the

concentration of 4' 4-diaminodiphenylsulfone and 3' 3-diaminodiphenylsulfone in the polymerization system is 0.4 ~ 0.6 mol/L, and the pre-polymerization temperature is -20 to 10 °C.

4. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder according to Claim 1, wherein the twin-screw extruder as a main reactor is self-cleaning, comprised of two intermeshed screws, the right-hand thread has a left-hand thread groove, the length-diameter ratio of the screw is higher than 1 : 30 and the diameter is greater than 25 mm.

5. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder according to Claim 1 or 4, wherein the length-diameter ratio of the screw is 1:40 to 1:60; the temperature of three temperature zones is -5 to 0 °C, 0 to 10 °C and 10 to 60 °C, respectively.

6. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder according to Claim 1, wherein the solid content is 12 ~ 20% by weight in the final spinning solution and it contains calcium chloride at 1 ~ 20 % by weight according to polysulfone amide.

7. A method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder as main reactor, wherein it comprises following steps:

(a) Dissolution: 4' 4-diaminodiphenylsulfone and 3' 3-diaminodiphenylsulfone by mass ratio of 10:90 ~ 90:10 are dissolved in N, N'-dimethylacetamide solvent, and cooled down in a cooler;

(b) Polymerization: the solution in step (a) and molten paraphthaloyl chloride of equal mole are loaded into a twin-screw extruder, which is used as main reactor, retained for 10 ~ 25 minutes and the temperature is divided into three zones with temperature at -5 to 5 °C, 0 to 20 °C and 10 to 100 °C,

respectively. The polysulfone amide containing hydrogen chloride was extruded.

(c) Neutralization: the polysulfone amide containing hydrogen chloride generated in step (b) is neutralized with calcium hydroxide or calcium oxide, and the obtained polymer solution can be directly applied for spinning.

8. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder as main reactor according to Claim 7, wherein the twin-screw extruder as main reactor is self-cleaning, comprised of two intermeshed screws, the right-hand thread has a left-hand thread groove, the length-diameter ratio of the screw is higher than 1:30 and the diameter is greater than 25 mm.

9. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder according to Claim 7 or 8, wherein the length-diameter ratio of the screw is 1:40 to 1:60; the temperature of three temperature zones is -5 to 0 °C, 0 to 10 °C and 10 to 60 °C, respectively.

10. Said method for continuous preparation of polysulfone amide spinning solution with a twin-screw extruder according to Claim 7, wherein the solid content is 12 ~ 20% by weight in the final spinning solution, and it contains calcium chloride at 1 ~ 20 % by weight according to polysulfone amide.

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