CLAIMS

1. Process for preparing a diamine/dicarboxylic acid salt comprising steps comprising contacting a diamine with a dicarboxylic acid to provide a reaction mixture in which said diamine and said dicarboxylic acid react to form a diamine/dicarboxylic acid salt, wherein
   (a) the dicarboxylic acid comprises an aromatic dicarboxylic acid;
   (b) the dicarboxylic acid is provided in a powder form;
   (c) the diamine is provided in a liquid form;
   (d) the contacting is performed by gradually dosing diamine liquid to dicarboxylic acid powder, while keeping the dicarboxylic acid powder in constant movement;
   (e) the reaction mixture is kept in constant movement for a time period directly following completion of the dosing;
   (f) (d) and (e) are carried out at a temperature above 0°C and below all of the following: the boiling temperature of the diamine and the melting temperatures of the dicarboxylic acid, the diamine/dicarboxylic acid salt and any intermediate reaction product, and
   (g) in (d) and (e) the reaction mixture comprises at most 5 wt.% of water, relative to the total weight of the diamine and dicarboxylic acid.
2. Process according to claim 1, wherein the diamine and the dicarboxylic acid in the reaction mixture are present in a molar ratio of diamine over dicarboxylic acid in the range of 0.9 – 1.1.
3. Process according to claim 1 or 2, wherein the dicarboxylic acid is a mixture of an aliphatic dicarboxylic acid and an aromatic dicarboxylic acid.
4. Process according to claim 3, wherein the mixture of aliphatic dicarboxylic acid and aromatic dicarboxylic acid is a dry blend of solid particles of the aliphatic dicarboxylic acid and solid particles of the aromatic dicarboxylic acid.
5. Process according to claim 3 or 4, wherein the aliphatic dicarboxylic acid and the aromatic dicarboxylic acid are present in a molar ratio between 90:10 and 10:90.
6. Process according to any of claims 1 - 3, wherein the dicarboxylic acid consists of 90 – 100 mole % of aromatic dicarboxylic acid and 10 – 0 mole % of aliphatic dicarboxylic acid.

7. Process according to any one of claims 1 - 6, wherein the aromatic dicarboxylic acid comprises either isophthalic acid, terephthalic acid or naphthalene dicarboxylic acid, or any combination thereof.

8. Process according to any one of claims 1 - 7, wherein the aliphatic dicarboxylic acid comprises adipic acid and/or sebacic acid.

9. Process according to any one of claims 1 - 8, wherein the diamine comprises an aliphatic diamine having 4 - 12 carbon atoms.

10. Process according to claim 9, wherein the diamine comprises 1,4-butane diamine and/or 1,6-hexane diamine.

11. Process according to any one of claims 1 - 10, wherein the diamine and the dicarboxylic acid are contacted by spraying or dripping the diamine onto the moving dicarboxylic acid powder.

12. Process according to any one of claims 1 - 11, wherein the diamine and the dicarboxylic acid contacted and mixed in a tumble mixer, a ploughshare mixer, a conical mixer, a planetary screw mixer or a fluidized bed reactor.

13. Process according to any one of claims 1 - 12, wherein the diamine and the dicarboxylic acid are contacted at a temperature between 0°C and the boiling temperature of water.

14. Process according to any one of claims 1 - 13, wherein neutralization heat produced upon reaction of the diamine and the dicarboxylic acid to form the diamine/dicarboxylic acid salt is removed via a heat exchanger.

15. Process according to any one of claims 1 – 13, wherein the dicarboxylic acid powder has a particle size distribution, measured by the method according to ISO 13320, with a d10 of at least 15 µm and a d90 of at most 1000 µm and with a median particle size (d50) in the range of 40 – 500 µm.

16. Process according to claim 15, wherein the dicarboxylic acid powder has a particle size distribution with a Span, defined by the ratio of (d84-d16)/d50, of at most 5.

17. Diamine/dicarboxylic acid salt wherein the salt is a granulate material obtainable by the process according to any one of claims 1 – 17.

18. Salt according to claim 18, wherein the salt is an anhydrous salt, comprising less than 0.5 wt.% water, relative to the total weight of the salt.

19. Salt according to claim 17 or 18, wherein the salt flowability defined by the ratio (ffc) of consolidation stress, $\sigma 1$, to unconfined yield strength, σc, measured by the shear test method according to ASTM D6773 of at least 10.

20. Salt according to any of claims 17-19, wherein the salt is a granulate material consisting of polycrystalline granules comprising micro-crystallites, wherein the micro-crystallites have a particle size distribution, measured by software supported analysis of SEM images taken from surface areas of granules, with a volume based d90 of at most 5µm.

21. Salt according to any of claims 17-20, wherein the salt is a granulate material consisting of polycrystalline granules, wherein the polycrystalline granules have a particle size distribution, measured by the method according to ISO 13320, with a d10 of at least 20 µm, a d90 of at most 1000 µm, and a median particle size (d50) in the range of 50 – 600 µm.

22. Salt according to any of claims 17-21, wherein the d10 is in the range of 20 - 200 µm, the d50 is in the range of 50 – 500 µm, and the d90 is in the range of 200 – 1000 µm.

23. Salt according to any of claims 17-22, wherein the polycrystalline granules have a particle size distribution with a Span, defined by the ratio of (d84-d16)/d50, of at most 5, preferably at most 2.5.

24. Salt according to any of claims 17-23, wherein the granulate material has a compressibility, expressed by the ratio of (TBD-ABD)/TBD*100%, of at most 35 %, wherein ABD is the aerated bulk density and TBD is the tapped bulk density both measured by the method according to ASTM D6393.

25. Salt according to any of claims 17-24, wherein the salt comprises a salt based on 1,4-butane diamine and terephthalic acid and/or a salt based on 1,6-hexane diamine and terephthalic acid.

26. Process for the preparation of a polyamide, wherein a salt according to any of claims 17-25 is used.

CLAIMS

1. A process for preparing a diamine/dicarboxylic acid salt comprising the steps of:
   (i) forming a reaction mixture by contacting a diamine liquid with a dicarboxylic acid powder comprising an aromatic dicarboxylic acid having a particle size distribution, measured by the method according to ISO 13320, with a d10 of at least 15 μm nd a d90 of at most 1000 μm and with a median particle size (d50) in a range of 40 – 500 μm, and
   (ii) allowing the diamine and the aromatic dicarboxylic acid in the reaction mixture to react to form a diamine/dicarboxylic acid acid salt,
   wherein

   step (i) comprises the steps of:

   (a) gradually dosing the diamine liquid to the aromatic dicarboxylic acid powder, while keeping the aromatic dicarboxylic acid powder in constant movement, and thereafter
   (b) keeping the reaction mixture in constant movement for a time period directly following completion of the dosing; wherein steps of (a) and (b) are carried out at a temperature above 0°C and below all of the following: the boiling temperature of the diamine and the melting temperatures of the dicarboxylic acid, the diamine/dicarboxylic acid salt and any intermediate reaction product, and wherein the reaction mixture in steps (a) and (b) comprises at most 5 wt.% of water, relative to the total weight of the diamine and dicarboxylic acid.

2. The process according to claim 1, wherein the diamine and the dicarboxylic acid in the reaction mixture are present in a molar ratio of diamine over dicarboxylic acid in the range of 0.9 – 1.1.

3. The process according to claim 1, wherein the dicarboxylic acid powder is a mixture of an aliphatic dicarboxylic acid and an aromatic dicarboxylic acid.

4. The process according to claim 3, wherein the mixture of aliphatic dicarboxylic acid and aromatic dicarboxylic acid is a dry blend of solid particles of the aliphatic dicarboxylic acid and solid particles of the aromatic dicarboxylic acid.

5. The process according to claim 3, wherein the aliphatic dicarboxylic acid and the aromatic dicarboxylic acid are present in a molar ratio between 90:10 and 10:90.

6. The process according to claim 1, wherein the dicarboxylic acid consists of 90 – 100 mole % of aromatic dicarboxylic acid and 10 – 0 mole % of aliphatic dicarboxylic acid.

7. The process according to claim 1, wherein the aromatic dicarboxylic acid comprises either isophthalic acid, terephthalic acid or naphthalene dicarboxylic acid, or any combination thereof.

8. The process according to claim 1, wherein the aliphatic dicarboxylic acid comprises adipic acid and/or sebacic acid.

9. The process according to claim 1, wherein the diamine comprises an aliphatic diamine having 4 - 12 carbon atoms.

10. The process according to claim 9, wherein the diamine comprises 1,4-butane diamine and/or 1,6-hexane diamine.

11. The process according to claim 1, wherein step (i) comprises contacting the diamine liquid and the dicarboxylic acid powder by spraying or dripping the diamine liquid onto dicarboxylic acid powder while moving the dicarboxylic acid powder.

12. The process according to claim 1, wherein step (i) comprises contacting and mixing the diamine liquid and the dicarboxylic acid powder in a tumble mixer, a ploughshare mixer, a conical mixer, a planetary screw mixer or a fluidized bed reactor.

13. The process according to claim 1, wherein step (i) comprises contacting the diamine liquid and the dicarboxylic acid powder at a temperature between 0°C and the boiling temperature of water.

14. The process according to claim 1, which further comprises the step (iii) removing neutralization heat produced upon reaction of the diamine and the dicarboxylic acid to form the diamine/dicarboxylic acid salt via a heat exchanger.

15. The process according to claim 1, wherein the dicarboxylic acid powder has a particle size distribution with a Span, defined by the ratio of (d84-d16)/d50, of at most 5.

16. A diamine/dicarboxylic acid salt, wherein the salt is a granulate material obtained by the process according to claim 1.

17. The salt according to claim 16, wherein the salt is an anhydrous salt, comprising less than 0.5 wt.% water, relative to the total weight of the salt.

18. The salt according to claim 16, wherein the salt has a flowability defined by the ratio (ffc) of consolidation stress, σ1, to unconfined yield strength, σc, measured by the shear test method according to ASTM D6773 of at least 10.

19. The salt according to claim 16, wherein the salt is a granulate material consisting of polycrystalline granules comprising micro-crystallites, wherein the micro-crystallites have a particle size distribution, measured by software supported analysis of SEM

images taken from surface areas of granules, with a volume based d90 of at most 5µm.

20. The salt according to claim 16, wherein the salt is a granulate material consisting of polycrystalline granules, wherein the polycrystalline granules have a particle size distribution, measured by the method according to ISO 13320, with a d10 of at least 20 µm, a d90 of at most 1000 µm, and a median particle size (d50) in the range of 50 – 600 µm.

21. The salt according to claim 20, wherein the d10 is in the range of 20 – 200 µm, the d50 is in the range of 50 – 500 µm, and the d90 is in the range of 200 – 1000 µm.

22. The salt according to claim 16, wherein the polycrystalline granules have a particle size distribution with a Span, defined by the ratio of (d84-d16)/d50, of at most 5.

23. The salt according to claim 16, wherein the granulate material has a compressibility, expressed by the ratio of (TBD-ABD)/TBD*100%, of at most 35 %, wherein ABD is an aerated bulk density and TBD is a tapped bulk density both measured by the method according to ASTM D6393.

24. The salt according to claim 16, which comprises a salt based on 1,4-butane diamine and terephthalic acid and/or a salt based on 1,6-hexane diamine and terephthalic acid.

25. The salt according to claim 22, wherein the Span of the polycrystalline granules is at most 2.5.

26. A process for the preparation of a polyamide which comprises forming a polyamide using the salt according to claim 16.