Claims

1. Process for producing adipic acid crystals from a solution of adipic acid, comprising at least one step of purification by crystallization and one step of separation and recovery of the adipic acid crystals, characterized in that the adipic acid crystals are subjected, before the separation and recovery step, to a grinding phase and in that the step of purification by crystallization and of separation and recovery of the adipic acid crystals comprises the following steps:
   - a crystallization phase in a crystallizer optionally comprising an external circulation loop of the crystallization medium, by cooling and/or by concentrating a solution of adipic acid;
   - optionally a phase of separation of the crystals formed;
   - optionally a phase of repulping the crystals in a liquid medium saturated with adipic acid;
   - a phase of separation and recovery of the crystals, optionally with washing of the crystals;

   and in that
   - when a single step of purification by crystallization and of separation and recovery of the adipic acid crystals is used, the grinding phase, before the phase of separation and recovery of the crystals, is carried out on at least one of the following media:
     - on the suspension of crystals contained in the crystallizer and circulating in the optional loop external to the crystallizer with recycling of this suspension containing the ground crystals into the crystallizer;
     - on the crystals contained in the medium of the optional repulping phase;
     - on the wet crystals after the optional repulping phase;
   - when at least two successive steps of purification by crystallization and of separation and recovery of the adipic acid crystals are carried out, the grinding phase, before the phase of separation and recovery of the crystals, is carried out, at at least any one of the steps preceding the last step of purification by crystallization and of separation and recovery of the adipic acid crystals and/or during the last step of purification by crystallization and of separation and recovery of the adipic acid crystals, on at least one of the following media:

- on the suspension of crystals contained in the crystallizer and circulating in the optional loop external to the crystallizer with recycling of this suspension containing the ground crystals into the crystallizer;
- on the crystals contained in the medium of the optional repulping phase;
- on the wet crystals after the optional repulping phase.

2. Process according to Claim 1, characterized in that the solution of adipic acid is an aqueous solution of adipic acid.

3. Process according to Claim 2, characterized in that the adipic acid is synthesized by nitric acid oxidation of a cyclohexanol/cyclohexanone mixture or of cyclohexanol.

4. Process according to Claim 2, characterized in that the adipic acid is synthesized by oxidation of cyclohexane with molecular oxygen.

5. Process according to one of the preceding claims, characterized in that it comprises at least two successive steps of purification by crystallization of the adipic acid.

6. Process according to Claim 5, characterized in that the first step of purification by crystallization comprises:
   - a phase of crystallization in a crystallizer by cooling and/or concentrating a solution of adipic acid;
   - a phase of separation of the crystals formed;
   - a phase of repulping of the crystals in a liquid medium saturated with adipic acid;
   - a phase of separation and recovery of the crystals, with optional washing of the crystals; and
   - the grinding phase being carried out on the crystals before the phase of separation and recovery of the crystals.

7. Process according to Claim 6, characterized in that the grinding phase of the crystals is carried out on the crystals contained in the repulping medium.

8. Process according to Claim 6, characterized in that the grinding phase of the crystals is carried out on the wet crystals before or after the repulping phase.

9. Process according to Claim 6, characterized in that the grinding of the adipic acid crystals is carried out on the suspension of crystals contained in the crystallizer and circulating in a loop external to the crystallizer with recycling of this suspension containing the ground crystals into the crystallizer.

10. Process according to Claim 6, characterized in that the grinding phase of the crystals is carried out on the crystals recovered after a first repulping phase then a first phase of separation by filtration, said ground crystals then being subjected to a new repulping phase before being recovered in the separation and recovery phase.

11. Process according to one of the preceding claims, characterized in that the separation and recovery of the crystals is carried out by filtration or centrifugal drying.

12. Process according to one of the preceding claims, characterized in that the separated crystals are subjected to one or more washing operations.

13. Process according to one of the preceding claims, characterized in that the grinding of the crystals is carried out by mechanical agitation, pounding and crushing.

Claims

1. Process for producing adipic acid crystals from a solution of adipic acid, comprising at least one step of purification by crystallization and one step of separation and recovery of the adipic acid crystals, characterized in that the adipic acid crystals are subjected, before the separation and recovery step, to a grinding phase and in that the step of purification by crystallization and of separation and recovery of the adipic acid crystals comprises the following steps:
   - a crystallization phase in a crystallizer optionally comprising an external circulation loop of the crystallization medium, by cooling and/or by concentrating a solution of adipic acid;
   - optionally a phase of separation of the crystals formed;
   - optionally a phase of repulping the crystals in a liquid medium saturated with adipic acid;
   - a phase of separation and recovery of the crystals, optionally with washing of the crystals;

   and in that
   - when a single step of purification by crystallization and of separation and recovery of the adipic acid crystals is used, the grinding phase, before the phase of separation and recovery of the crystals, is carried out on at least one of the following media:
     - on the suspension of crystals contained in the crystallizer and circulating in the optional loop external to the crystallizer with recycling of this suspension containing the ground crystals into the crystallizer;
     - on the crystals contained in the medium of the optional repulping phase;
     - on the wet crystals after the optional repulping phase;
   - when at least two successive steps of purification by crystallization and of separation and recovery of the adipic acid crystals are carried out, the grinding phase, before the phase of separation and recovery of the crystals, is carried out, at at least any one of the steps preceding the last step of purification by crystallization and of separation and recovery of the adipic acid crystals and/or during the last step of purification by crystallization and of separation and recovery of the adipic acid crystals, on at least one of the following media:
     - on the suspension of crystals contained in the crystallizer and circulating in the optional loop external to the crystallizer with recycling of this suspension containing the ground crystals into the crystallizer;
     - on the crystals contained in the medium of the optional repulping phase;
     - on the wet crystals after the optional repulping phase.

2. Process according to Claim 1, characterized in that the grinding of the crystals is obtained by applying a mechanical force to the crystals either directly to the wet crystals or to the crystals suspended in a liquid such as water, advantageously saturated with adipic acid.

3. Process according to any of claims 1 or 2, characterized in that the solution of adipic acid is an aqueous solution of adipic acid.

4. Process according to Claim 3, characterized in that the adipic acid is synthesized by nitric acid oxidation of a cyclohexanol/cyclohexanone mixture or of cyclohexanol.

5. Process according to Claim 3, characterized in that the adipic acid is synthesized by oxidation of cyclohexane with molecular oxygen.

6. Process according to one of the preceding claims, characterized in that it comprises at least two successive steps of purification by crystallization of the adipic acid.

7. Process according to Claim 6, characterized in that the first step of purification by crystallization comprises:
   - a phase of crystallization in a crystallizer by cooling and/or concentrating a solution of adipic acid;
   - a phase of separation of the crystals formed;
   - a phase of repulping of the crystals in a liquid medium saturated with adipic acid;
   - a phase of separation and recovery of the crystals, with optional washing of the crystals; and
   - the grinding phase being carried out on the crystals before the phase of separation and recovery of the crystals.

8. Process according to Claim 7, characterized in that the grinding phase of the crystals is carried out on the crystals contained in the repulping medium.

9. Process according to Claim 7, characterized in that the grinding phase of the crystals is carried out on the wet crystals before or after the repulping phase.

10. Process according to Claim 7, characterized in that the grinding of the adipic acid crystals is carried out on the suspension of crystals contained in the crystallizer and circulating in a loop external to the crystallizer with recycling of this suspension containing the ground crystals into the crystallizer.

11. Process according to Claim 7, characterized in that the grinding phase of the crystals is carried out on the crystals recovered after a first repulping phase then a first phase of separation by filtration, said ground crystals then being subjected to a new repulping phase before being recovered in the separation and recovery phase.

12. Process according to one of the preceding claims, characterized in that the separation and recovery of the crystals is carried out by filtration or centrifugal drying.

13. Process according to one of the preceding claims, characterized in that the separated crystals are subjected to one or more washing operations.

14. Process according to one of the preceding claims, characterized in that the grinding of the crystals is carried out by mechanical agitation, pounding and crushing.