Claims

1. A method for separating acetonitrile from water, comprising

   (i) providing a stream S1 containing at least 95 wt.-%, based on the total weight of S1, acetonitrile and water, wherein the weight ratio of acetonitrile : water is greater than 1;

   (ii) adding a stream P, comprising at least 95 wt.-% C3, based on the total weight of stream P, to S1 to obtain a mixed stream S2, C3 being propene optionally admixed with propane with a minimum weight ratio of propene : propane of 7:3;

   (iii) subjecting S2 to a temperature in the range of from 5 to 90 °C and a pressure of at least $10x10^5$ Pa (10 bar), obtaining a first liquid phase L1, wherein at least 90 wt.-% of L1 consist of C3, acetonitrile and water, and a second liquid phase L2, wherein at least 90 wt.-% of L2 consist of acetonitrile and water, and wherein the weight ratio of acetonitrile : water in L2 is less than 1;

   (iv) separating L1 from L2.

2. The method of claim 1, wherein in (ii), a liquid stream P is added to a liquid stream S1.

3. The method of claim 1 or 2, wherein S1 contains from 60 to 85 wt.-%, preferably from 65 to 80 wt.-% acetonitrile, and from 10 to 35 wt.-%, preferably from 15 to 30 wt.-% water, in each case based on the total weight of S1.

4. The method of any of claims 1 to 3, wherein in (ii), P is added to S1 in an amount so that the weight ratio of C3 : acetonitrile in S2 is in the range of from 0.2:1 to 5:1, preferably from 0.5:1 to 2:1.

5. The method of any of claims 1 to 4, wherein in (iii), S2 is subjected to a temperature of from 25 to 45 °C, and to a pressure in the range of from $15x10^5$ to $25x10^5$ Pa (15 to 50 bar), in particular from $15x10^5$ to $25x10^5$ Pa (15 to 25 bar).

6. The method of any of claims 1 to 5, wherein at least one liquid phase separation improving agent is added to S1 in (i) and/or to S2 in (ii), preferably in an amount of 1 wt.-% at most, based on the total weight of S1 and/or S2.

7. The method of any of claims 1 to 6, wherein in (iv), L1 is separated from L2 in a gravity settler, preferably in a horizontal gravity settler.

8. The method of any of claims 1 to 7, wherein at least 95 wt.-%, preferably at least 98 wt.-% of L1 consist of C3, acetonitrile and water, the water content of L1 being less than 10 wt.-%, preferably in the range of from 1 to 5 wt.-%, based on the total weight of L1.

9. The method of any of claims 1 to 8, wherein at least 95 wt.-%, preferably at least 98 wt.-% of L2 consist of C3, acetonitrile and water, the C3 content of L2 being 5 wt.-% at most, based on the total weight of L2, and the acetonitrile content of L2 being less than 45 wt.-%, preferably in the range of from 10 to 35 wt.-%, based on the total weight of L2.

10. The method of any of claims 1 to 9, further comprising subjecting L1 to a distillation stage wherefrom a stream TL1 is obtained which contains at least 90 wt.-%, preferably at least 95 wt.-% C3, based on the total weight of TL1.

11. The method of claim 10, wherein, from said distillation stage, a further stream BL1 is obtained, at least 95 wt.-%, preferably at least 98 wt.-% of BL1 consisting of C3, acetonitrile and water, wherein the C3 content of BL1 is in the range of from 7 to 18 wt.-%, preferably from 10 to 15 wt.-%, in each case based on the total weight of BL1.

12. The method of claim 10 or 11, wherein in said distillation stage, one distillation tower is employed and wherein said distillation is carried out at a dew-point at the top of said distillation tower of at least 40 °C, preferably in the range of from 40 to 80 °C.

13. The method of any of claims 10 to 12, wherein at least a portion of TL1 is recycled into (ii).

14. The method of any of claims 1 to 13, further comprising subjecting L2 to a distillation stage wherefrom a stream TL2 is obtained which contains from 75 to 95 wt.-%, preferably from 80 to 85 wt.-% acetonitrile, based on the total weight of TL2.

15. The method of claim 14, wherein at least a portion of TL2 is recycled into (ii).

16. The method of claim 14 or 15, wherein said distillation stage is a two pressure distillation process, wherein in a first distillation tower, distillation is carried out at a top pressure which is higher than the top pressure of a second distillation tower and wherein the condenser used to condense the top stream of the first distillation tower is used simultaneously as the vaporizer of the second distillation tower.

17. The method of claim 16, wherein

(aa) L2 is introduced into the first distillation tower from which a vapor top stream VTL2 is obtained containing from 50 to 70 wt.-% acetonitrile, based on the total weight of top stream VTL2, the distillation preferably being carried out at a top pressure of from 10×10^5 to 20×10^5 Pa (10 to 20 bar); and

(bb) at least partially condensing VTL2 obtained in (aa) and introducing the condensed stream into the second distillation tower wherefrom TL2 is obtained as top stream, the distillation preferably being carried out at a top pressure of from 1×10^5 to 5×10^5 Pa (1 to 5 bar),

wherein the condenser used to condense VTL2 is simultaneously used as vaporizer of the second distillation tower.

18. The method of any of claims 1 to 17, wherein according to (i), S1 is provided by a process comprising

(a) reacting propene, optionally admixed with propane, with hydrogen peroxide in a reaction apparatus in the presence of acetonitrile as solvent, obtaining a stream S0 leaving the reaction apparatus, S0 containing acetonitrile, water, propylene oxide, and optionally non-reacted propene, oxygen, and further optionally propane;

(b) optionally separating propene, oxygen, and propane from S0 to obtain a stream essentially consisting of acetonitrile, water, and propylene oxide;

(c) separating propylene oxide from S0 or the stream obtained from (b), thus obtaining stream S1.

19. The method of claim 18, wherein in (a), propene is reacted with hydrogen peroxide in the presence of a heterogeneous catalyst, said catalyst preferably comprising a zeolite, preferably a titanium zeolite, more preferably a titanium zeolite of the structure type MWW.

20. The method of claim 18 or 19, wherein in (b), a distillation tower is used and at the top of said distillation tower, acetonitrile, optionally admixed with water, is added as a stripping agent.

21. The method of any of claims 18 to 20, wherein at least a portion of stream BL1 according to claim 11 is recycled into (a).

22. The method of any of claims 18 to 21, wherein prior to (ii), S1 is subjected to a hydrogenation stage, the resulting hydrogenated stream optionally being subjected to a distillation stage.

23. The method of any of claims 1 to 22, wherein S1 additionally contains at least one glycol, preferably in an amount of 1 wt.-% or less, based on the total weight of S1.

24. The method of claim 23, wherein the at least one glycol is selected from the group consisting of propylene glycol, dipropylene glycol, tripropylene glycol, and a mixture of two or three thereof.

25. The method of claim 23 or 24, the bottoms stream obtained from the first distillation tower according to claim 16 or 17 containing at least a portion of the at least one glycol, wherein said bottoms stream, optionally after combining with the bottoms stream obtained from the second distillation tower according to claim 16 or 17, is subjected to a glycol separation stage.

26. A highly integrated process for the preparation of propylene oxide, the process comprising

(a) reacting propene, optionally admixed with propane, with hydrogen peroxide in a reaction apparatus in the presence of acetonitrile as solvent, obtaining a stream S0 leaving said reaction apparatus, S0 containing acetonitrile, water, propylene oxide, and optionally non-reacted propene, oxygen, and optionally propane;

(b) optionally separating propene, optionally together with propane, and oxygen from S0 to obtain a stream, at least 99 wt.-% thereof consisting of acetonitrile, water, and propylene oxide;

(c) separating propylene oxide from S0 or the stream obtained from (b), thus obtaining a stream S1 containing at least 95 wt.-% acetonitrile and water, based on the total weight of S1, wherein the weight ratio of acetonitrile : water is greater than 1;

(d) adding a stream P, comprising at least 95 wt.-% C3, based on the total weight of stream P, to S1 to obtain a mixed stream S2, C3 being propene optionally admixed with propane with a minimum weight ratio of propene : propane of 7:3;

(e) subjecting S2 to a temperature of from 5 to 90 °C and a pressure of at least $10x10^5$ Pa (10 bar), obtaining a first liquid phase L1, wherein at least 90 wt.-% of L1 consist of C3, acetonitrile and water, and a second liquid phase L2, wherein at least 90 wt.-% of L2 consist of acetonitrile and water, and wherein the weight ratio of acetonitrile : water in L2 is less than 1;

(f) separating L1 from L2;

(g) subjecting L1 to a distillation stage wherefrom a stream TL1 is obtained which contains at least 90 wt.-% C3, based on the total weight of TL1; and wherefrom a further stream BL1 is obtained, at least 95 wt.-% of BL1 consisting of C3, acetonitrile and water, wherein the C3 content of BL1 is in the range of from 7 to 18 wt.-% based on the total weight of BL1;

(h) subjecting L2 to a distillation stage wherefrom a stream TL2 is obtained which contains from 75 to 95 wt.-%, preferably from 80 to 85 wt.-% acetonitrile, based on the total weight of TL2;

wherein at least a portion of stream BL1 is recycled into (a); and/or

wherein at least a portion of TL1 is recycled into (d); and/or

wherein at least a portion of TL2 is recycled into (d).

27. The process of claim 26, wherein BL1 is recycled into (a) and TL1 is recycled into (d) and TL2 is recycled into (d).

28. The process of claim 26 or 27, further comprising

(y) subjecting the stream obtained from (c) to a hydrogenation stage;

(z) subjecting the stream obtained from (y) to a distillation stage to obtained stream S1 and subjecting S1 to (d).

Claims

1. A method for separating acetonitrile from water, comprising
   (i) providing a stream S1 containing at least 95 wt.-%, based on the total weight of S1, acetonitrile and water, wherein the weight ratio of acetonitrile : water is greater than 1;
   (ii) adding a stream P, comprising at least 95 wt.-% C3, based on the total weight of stream P, to S1 to obtain a mixed stream S2, C3 being propene optionally admixed with propane with a minimum weight ratio of propene : propane of 7:3;
   (iii) subjecting S2 to a temperature of 92 °C at most and a pressure of at least 10 bar, obtaining a first liquid phase L1 essentially consisting of C3, acetonitrile, and water, and a second liquid phase L2 essentially consisting of water and acetonitrile wherein the weight ratio of acetonitrile : water in L2 is less than 1;
   (iv) separating L1 from L2.

2. The method of claim 1, wherein in (ii), a liquid stream P is added to a liquid stream S1.

3. The method of claim 1 or 2, wherein S1 contains from 60 to 85 wt.-%, preferably from 65 to 80 wt.-% acetonitrile, and from 10 to 35 wt.-%, preferably from 15 to 30 wt.-% water, in each case based on the total weight of S1.

4. The method of any of claims 1 to 3, wherein in (ii), P is added to S1 in an amount so that the weight ratio of C3 : acetonitrile in S2 is in the range of from 0.2:1 to 5:1, preferably from 0.5:1 to 2:1.

5. The method of any of claims 1 to 4, wherein in (iii), S2 is subjected to a temperature of from 5 to 90 °C, in particular from 25 to 45 °C, and to a pressure in the range of from 15 to 50 bar, in particular from 15 to 25 bar.

6. The method of any of claims 1 to 5, wherein at least one liquid phase separation improving agent is added to S1 in (i) and/or to S2 in (ii), preferably in an amount of 1 wt.-% at most, based on the total weight of S1 and/or S2.

7. The method of any of claims 1 to 6, wherein in (iv), L1 is separated from L2 in a gravity settler, preferably in a horizontal gravity settler.

8. The method of any of claims 1 to 7, wherein at least 95 wt.-%, preferably at least 98 wt.-% of L1 consist of C3, acetonitrile and water, the water content of L1 being

less than 10 wt.-%, preferably in the range of from 1 to 5 wt.-%, based on the total weight of L1.

9. The method of any of claims 1 to 8, wherein at least 95 wt.-%, preferably at least 98 wt.-% of L2 consist of C3, acetonitrile and water, the C3 content of L2 being 5 wt.-% at most, based on the total weight of L2, and the acetonitrile content of L2 being less than 45 wt.-%, preferably in the range of from 10 to 35 wt.-%, based on the total weight of L2.

10. The method of any of claims 1 to 9, further comprising subjecting L1 to a distillation stage wherefrom a stream TL1 is obtained which contains at least 90 wt.-%, preferably at least 95 wt.-% C3, based on the total weight of TL1.

11. The method of claim 10, wherein, from said distillation stage, a further stream BL1 is obtained, at least 95 wt.-%, preferably at least 98 wt.-% of BL1 consisting of C3, acetonitrile and water, wherein the C3 content of BL1 is in the range of from 7 to 18 wt.-%, preferably from 10 to 15 wt.-%, in each case based on the total weight of BL1.

12. The method of claim 10 or 11, wherein in said distillation stage, one distillation tower is employed and wherein said distillation is carried out at a dew-point at the top of said distillation tower of at least 40 °C, preferably in the range of from 40 to 80 °C.

13. The method of any of claims 10 to 12, wherein at least a portion of TL1 is recycled into (ii).

14. The method of any of claims 1 to 13, further comprising subjecting L2 to a distillation stage wherefrom a stream TL2 is obtained which contains from 75 to 95 wt.-%, preferably from 80 to 85 wt.-% acetonitrile, based on the total weight of TL2.

15. The method of claim 14, wherein at least a portion of TL2 is recycled into (ii).

16. The method of claim 14 or 15, wherein said distillation stage is a two pressure distillation process, wherein in a first distillation tower, distillation is carried out at a top pressure which is higher than the top pressure of a second distillation tower and wherein the condenser used to condense the top stream of the first distillation tower is used simultaneously as the vaporizer of the second distillation tower.

17. The method of claim 16, wherein

(aa) L2 is introduced into the first distillation tower from which a vapor top stream VTL2 is obtained containing from 50 to 70 wt.-% acetonitrile, based on the total weight of top stream VTL2, the distillation preferably being carried out at a top pressure of from 10 to 20 bar; and

(bb) at least partially condensing VTL2 obtained in (aa) and introducing the condensed stream into the second distillation tower wherefrom TL2 is obtained as top stream, the distillation preferably being carried out at a top pressure of from 1 to 5 bar,

wherein the condenser used to condense VTL2 is simultaneously used as vaporizer of the second distillation tower.

18. The method of any of claims 1 to 17, wherein according to (i), S1 is provided by a process comprising

(a) reacting propene, optionally admixed with propane, with hydrogen peroxide in a reaction apparatus in the presence of acetonitrile as solvent, obtaining a stream S0 leaving the reaction apparatus, S0 containing acetonitrile, water, propylene oxide, and optionally non-reacted propene, oxygen, and further optionally propane;

(b) optionally separating propene, oxygen, and propane from S0 to obtain a stream essentially consisting of acetonitrile, water, and propylene oxide;

(c) separating propylene oxide from S0 or the stream obtained from (b), thus obtaining stream S1.

19. The method of claim 18, wherein in (a), propene is reacted with hydrogen peroxide in the presence of a heterogeneous catalyst, said catalyst preferably comprising a zeolite, preferably a titanium zeolite, more preferably a titanium zeolite of the structure type MWW.

20. The method of claim 18 or 19, wherein in (b), a distillation tower is used and at the top of said distillation tower, acetonitrile, optionally admixed with water, is added as a stripping agent.

21. The method of any of claims 18 to 20, wherein at least a portion of stream BL1 according to claim 11 is recycled into (a).

22. The method of any of claims 18 to 21, wherein prior to (ii), S1 is subjected to a hydrogenation stage, the resulting hydrogenated stream optionally being subjected to a distillation stage.

23. The method of any of claims 1 to 22, wherein S1 additionally contains at least one glycol, preferably in an amount of 1 wt.-% or less, based on the total weight of S1.

24. The method of claim 23, wherein the at least one glycol is selected from the group consisting of propylene glycol, dipropylene glycol, tripropylene glycol, and a mixture of two or three thereof.

25. The method of claim 23 or 24, the bottoms stream obtained from the first distillation tower according to claim 16 or 17 containing at least a portion of the at least one glycol, wherein said bottoms stream, optionally after combining with the bottoms stream obtained from the second distillation tower according to claim 16 or 17, is subjected to a glycol separation stage.

26. A highly integrated process for the preparation of propylene oxide, the process comprising

(a) reacting propene, optionally admixed with propane, with hydrogen peroxide in a reaction apparatus in the presence of acetonitrile as solvent, obtaining a stream S0 leaving said reaction apparatus, S0 containing acetonitrile, water, propylene oxide, and optionally non-reacted propene, oxygen, and optionally propane;

(b) optionally separating propene, optionally together with propane, and oxygen from S0 to obtain a stream, at least 99 wt.-% thereof consisting of acetonitrile, water, and propylene oxide;

(c) separating propylene oxide from S0 or the stream obtained from (b), thus obtaining a stream S1 containing at least 95 wt.-% acetonitrile and water, based on the total weight of S1, wherein the weight ratio of acetonitrile : water is greater than 1;

(d) adding a stream P, comprising at least 95 wt.-% C3, based on the total weight of stream P, to S1 to obtain a mixed stream S2, C3 being propene optionally admixed with propane with a minimum weight ratio of propene : propane of 7:3;

(e) subjecting S2 to a temperature of 92 °C at most and a pressure of at least 10 bar, obtaining a first liquid phase L1 essentially consisting of C3, acetonitrile, and water, and a second liquid phase L2 essentially consisting of water and acetonitrile wherein the weight ratio of acetonitrile : water in L2 is less than 1;

(f) separating L1 from L2;

(g) subjecting L1 to a distillation stage wherefrom a stream TL1 is obtained which contains at least 90 wt.-% C3, based on the total weight of TL1; and wherefrom a further stream BL1 is obtained, at least 95 wt.-% of BL1 consisting of C3, acetonitrile and water, wherein the C3 content of BL1 is in the range of from 7 to 18 wt.-% based on the total weight of BL1;

(h) subjecting L2 to a distillation stage wherefrom a stream TL2 is obtained which contains from 75 to 95 wt.-%, preferably from 80 to 85 wt.-% acetonitrile, based on the total weight of TL2;

wherein at least a portion of stream BL1 is recycled into (a); and/or

wherein at least a portion of TL1 is recycled into (d); and/or

wherein at least a portion of TL2 is recycled into (d).

27. The process of claim 26, wherein BL1 is recycled into (a) and TL1 is recycled into (d) and TL2 is recycled into (d).

28. The process of claim 26 or 27, further comprising

(y) subjecting the stream obtained from (c) to a hydrogenation stage;

(z) subjecting the stream obtained from (y) to a distillation stage to obtained stream S1 and subjecting S1 to (d).