

CLAIMS

1. A process for the production of substituted acroleins which comprises aldolisation of an aldehyde or a mixture of aldehydes in the presence of an effective amount of an alkali aldolisation catalyst followed by dehydration to form at least one substituted acrolein and water, recovering from the aldolisation/dehydration step a reaction mixture comprising unreacted aldehyde or aldehydes, water, aldolisation catalyst, and said at least one substituted acrolein, and distilling the reaction mixture to separate (i) an overhead fraction comprising unreacted aldehyde or aldehydes and water from (ii) a bottoms fraction containing said aldolisation catalyst and said at least one substituted acrolein, recycling unreacted aldehyde or aldehydes of said overhead fraction to the aldolisation/dehydration step, and purging from the system a stream substantially free from said aldolisation catalyst and comprising water from said overhead fraction.

2. A process according to claim 1, which comprises:

(a) providing an aldolisation zone and a distillation zone;

(b) continuously supplying to the aldolisation zone an organic stream containing said aldehyde and an aqueous stream containing an alkali catalyst;

(c) maintaining said aldolisation zone under aldolisation and dehydration conditions effective for conversion of said aldehyde in the presence of said alkali catalyst to an aldolisation product with subsequent dehydration of said aldolisation product to form said substituted acrolein;

(d) recovering from said aldolisation zone a reaction mixture in the form of a reaction product stream comprising mixed organic and aqueous phases;

(e) distilling said reaction product stream in said distillation zone;

(f) recovering overhead from said distillation zone a

vaporous stream comprising unreacted aldehyde and water;

- (g) condensing said vaporous stream;
- (h) allowing the resulting condensate to separate into an upper organic phase and a lower water phase;
- (i) purging at least a part of said lower water phase;
- (j) recycling material of said upper organic phase to said aldolisation zone;
- (k) recovering from a bottom part of said distillation zone a bottoms product comprising an organic phase rich in substituted acrolein and an aqueous phase containing said alkali catalyst;
- (l) cooling said bottoms product;
- (m) allowing said cooled bottoms product to separate into an upper organic phase rich in substituted acrolein and a lower catalyst containing phase;
- (n) recycling material of said lower catalyst containing phase to said aldolisation zone; and
- (o) recovering said upper organic phase of step (m).

3. A process according to claim 2, in which the aqueous stream supplied to the aldolisation zone in step (b) comprises from about 0.1% w/v to about 5% w/v of sodium hydroxide and up to about 35% w/v of sodium salts of organic acids produced by Cannizzaro reactions.

4. A process according to claim 2 or claim 3, in which the aldolisation zone comprises a stirred tank reactor operated under aqueous continuous phase conditions.

5. A process according to any one of claims 2 to 4, in which the aldolisation zone comprises a stirred tank reactor and in which the power supplied to the stirrer ranges from about 1.0 to about 3.0 kW/m³ of liquor.

6. A process according to any one of claims 2 to 5, in which a part of the upper organic phase of step (h) is purged

from the plant in order to control the build up of volatile non aldehyde materials in the plant.

7. A process according to any one of claims 2 to 6, in which the aldehyde is supplied to the aldolisation step in the form of a mixture of isomeric aldehydes.

8. A process according to any one of claims 1 to 7, in which the reaction mixture comprises unreacted aldehyde or aldehydes, aldols, water, aldolisation catalyst, at least one substituted acrolein, and "heavies", in which the reaction mixture is supplied to an intermediate section of the distillation zone which further includes a higher temperature section below said intermediate section and also a lower temperature distillation section, in which distillation conditions are maintained in the higher temperature section effective for decomposition of aldols and "heavies" in the presence of the aldolisation catalyst, in which an aldehyde or aldehydes is or are recovered from the lower temperature section for recycle to the aldolisation/dehydration step, and in which a bottoms fraction comprising substituted acrolein and aldolisation catalyst is recovered from the higher temperature section.

9. A process according to any one of claims 1 to 8, in which the aldehyde or each of the aldehydes contains from 3 to 10 carbon atoms.

10. A process according to any one of claims 1 to 9, in which the aldolisation catalyst is an alkali metal hydroxide or carbonate or an alkaline earth metal hydroxide.

11. A process according to any one of claims 1 to 11, in which the aldolisation catalyst is selected from sodium hydroxide, sodium carbonate, potassium hydroxide, and potassium carbonate.

12. A process according to any one of claims 1 to 11, in which the aldolisation step is carried out at a temperature in the range of from about 80°C to about 150°C and at a pressure of from about 0.1 bar to about 10 bar.

13. A process according to any one of claims 1 to 12, in which the distillation step is conducted at a pressure of from about 0.1 bar to about 2 bar.

14. A process according to any one of claims 1 to 13, in which the overhead fraction is condensed and allowed to separate to form an upper organic phase containing unreacted aldehyde or aldehydes and a lower water phase.

15. A process according to claim 14, in which a part of the lower water phase is returned to the distillation step as a reflux stream.

16. A process according to claim 14 or claim 15, in which a part of the upper organic phase is recycled to the distillation step as a reflux stream.

17. A process according to any one of claims 14 to 16, in which a part of the upper organic phase is purged from the plant in order to control the build up of volatile non-aldehyde materials in the process.

18. A process according to any one of claims 1 to 17, in which the bottoms fraction from the distillation step is cooled and allowed to separate into an upper organic phase rich in substituted acrolein and a lower catalyst containing phase.

19. A process according to claim 18, in which lower catalyst containing phase is recycled to the aldolisation/dehydration step.

20. A process according to claim 19, in which a part of the lower catalyst containing phase is purged from the process in order to control the build up of Cannizzaro reaction products and in which an amount of make up aldolisation catalyst corresponding to any metal ions lost in the purge stream is supplied to the aldolisation step.

21. A process according to any one of claims 18 to 20, in which the lower catalyst containing phase contains from about 0.1% w/v to about 5% w/v of aldolisation catalyst and up to about 35%w/v of a salt or salts of an organic acid or acids produced by Cannizzaro reactions.

22. A process according to any one of claims 1 to 21, in which aldolisation is effected in a stirred tank reactor provided with internal baffles.

23. A process according to any one of claims 1 to 22, in which aldolisation is carried out in an aldolisation zone with a residence time of from about 2 minutes to about 75 minutes.

24. A process according to any one of claims 1 to 23, in which aldolisation is carried out with an organic phase:aqueous phase ratio of from about 3:1 to about 1:15 by volume.

25. A process according to any one of claims 1 to 24, in which aldolisation is carried out in a tank reactor provided with a stirrer and in which the power supplied to the stirrer ranges from about 1.0 to about 3.0 kW/m³ of liquor.

26. A process according to any one of claims 1 to 25, in which the aldolisation/dehydration step is effected using a mixture of C₅ aldehydes which comprises at least 88 molar % (preferably at least 94 molar % and even more

preferably at least 98.8 molar %) of n-valeraldehyde, up to 10 molar % (preferably not more than 5 molar % and even more preferably not more than 1.2 molar %) of 2-methylbutyraldehyde, and up to 2 molar % (preferably not more than 1 molar % (preferably not more than 1 molar % and even more preferably not more than 0.01 molar %) of 3-methylbutyraldehyde and in which the bottoms fraction from the distillation step contains less than 0.3% w/v of aldols.

~~27. A C₁₀ substituted acrolein mixture produced by a process according to any one of claims 1 to 26, which comprises (i) at least 76 molar % (preferably at least 88 molar %, more preferably at least 95.8 molar %, and even more preferably at least 97.6 molar %) 2-propylhept-2-enal, (ii) up to 20 molar % (preferably no more than 10 molar %, more preferably no more than 4.0 molar %, and even more preferably no more than 2.4 molar %) 2-propyl-4-methylhex-2-enal, and (iii) up to 4 molar % in total (preferably no more than 2 molar % in total, more preferably no more than 0.2 molar % in total, and even more preferably no more than 0.01 molar % in total) of 2-propyl-5-methylhex-2-enal, 2-iso-propylhept-2-enal, 2-iso-propyl-5-methylhex-2-enal, and 2-iso-propyl-4-methylhex-2-enal.~~