

We claim:

1. A process for separating acetaldehyde from a first mixture comprising acetaldehyde, one or more C₁-C₁₂ alkyl iodides, water, and methanol, the process comprising the steps of:
 - 5 distilling the first mixture in a distillation column to form at least two streams selected from the group consisting of an overhead stream, sidecut stream, and lower stream, wherein either the overhead stream or sidecut stream are withdrawn as a second mixture;
separating acetaldehyde from the second mixture; and
controlling the 1,1-dimethoxyethane mass composition in the lower stream to less than or
10 equal to 0.03 wt.% by operating the distillation column under at least one of the following conditions (i) to (iii):
 - (i) temperature in a lower portion of the distillation column is not less than 40 °C;
 - (ii) water mass composition in the lower stream is not less than 0.3 wt.%; or
 - (iii) acetic acid mass composition in the lower stream on weight percentage basis
15 is greater than the acetic acid mass composition in the first mixture;and wherein the methanol mass composition in the first mixture is less than or equal to 2 wt.%.
2. The process of claim 1, wherein the methanol mass composition in the first mixture is less
20 than or equal to 1 wt.%.
3. The process of claim 1, wherein the methanol mass composition in the first mixture is less than or equal to 0.5 wt.%.
- 25 4. The process of claim 1, wherein the pressure in the distillation column is from 0.1 to 0.7 MPa.
5. The process of claim 1, wherein the weight ratio of 1,1-dimethoxyethane mass composition in the lower stream to overhead stream is 100:1 to 10:1.

6. The process of claim 1, wherein the weight ratio of 1,1-dimethoxyethane mass composition in the lower stream to sidecut stream is 100:1 to 10:1.
7. The process of claim 1, wherein the second mixture comprises a portion of the overhead stream and a portion of the sidecut.
8. The process of claim 1, wherein the step of separating acetaldehyde from the second mixture further comprises feeding at least a portion of second mixture to a vessel under conditions sufficient to phase separate the second mixture into an aqueous phase and an organic phase.
9. The process of claim 8, wherein the organic phase is fed to the lower portion of distillation column under the at least one of the conditions (i) to (iii).
10. The process of claim 8, wherein the organic phase comprises methanol, and the total methanol mass composition in the organic phase and first mixture is less than or equal to 2 wt.%.
11. The process of claim 1, wherein the distillation column for distilling the first mixture is an extractive distillation step and further comprising adding an extractant to an upper portion of the distillation column.
12. The process of claim 1, wherein for condition (ii) the water mass composition in the lower stream is not less than 0.6 wt.%.
13. The process of claim 1, wherein for condition (iii) the acetic acid mass composition in the lower stream is not more than 3 wt.%.
14. The process of claim 1, wherein the distillation column has more than 10 stages.
15. The process of claim 1, wherein the reflux ratio of the distillation column is from 1:20 to 20:1.

16. The process of claim 1, wherein the first mixture comprises acetaldehyde in a mass composition from 0.01 to 30 wt.%, one or more C₁-C₁₂ alkyl iodides in a mass composition from 0.1 to 90 wt.%, water in a mass composition from 0.1 to 90 wt.%, and methanol in a mass composition from 0.001 to 2 wt.%, based on the total weight of the first mixture.

17. The process of claim 1, wherein the methanol mass composition in the first mixture is more than the dimethyl ether mass composition.

18. The process of claim 1, wherein the distillation column is operated under conditions to prevent formation of methanol.

19. A process for separating acetaldehyde from a first mixture comprising acetaldehyde, one or more C₁-C₁₂ alkyl iodides, water, and methanol, the process comprising the steps of:

distilling the first mixture in a distillation column to form at least two streams selected from the group consisting of an overhead stream, sidecut stream, and lower stream, wherein the sidecut stream is withdrawn as a second mixture;

separating the second mixture into an aqueous stream comprising acetaldehyde or an organic stream comprising the one or more C₁-C₁₂ alkyl iodides; and

controlling the 1,1-dimethoxyethane mass composition in the lower stream to less than or equal to 0.03 wt.% by operating the distillation column under at least one of the following conditions (i) to (iii):

(i) temperature in a lower portion of the distillation column is not less than 40 °C;

(ii) water mass composition in the lower stream is not less than 0.3 wt.%; or

(iii) acetic acid mass composition in the lower stream on weight percentage basis is greater than the acetic acid mass composition in the first mixture;

and wherein the methanol mass composition in the first mixture is less than or equal to 2 wt.%.

20. The process of claim 19, wherein the distillation column for distilling the first mixture is an extractive distillation step and further comprising adding an extractant to an upper portion of the distillation column.