It has now been found that in the presence of solid acid catalysts, such as zeolites, the dehydration reaction is relatively slow and since water is consumed more quickly by the hydrolysis reaction, it is typically necessary to provide water to the system to maintain a steady-state concentration of water in the reaction. Water may be added to the dehydration-hydrolysis process as a component of process streams such as water-containing feed and recycle streams to the process.

In general, methanol obtained by commercial synthesis processes contains water and may also contain some dimethyl ether. The amount of water in the methanol product can vary depending upon such factors as the composition of the feed to the process and the process conditions, and in particular the amount of carbon dioxide employed in the methanol synthesis process.

Methyl acetate may be produced by processes for carbonylating ethers, such as the carbonylation of dimethyl ether with carbon monoxide, as described, for example in US 7,465,822, WO 2008/132438 and WO 2008/132468. Although the principal reaction of dimethyl ether with carbon monoxide does not itself produce water, it has now been found that low levels of water can be produced via side-reactions taking place in the carbonylation process.

Thus the amount of water present in feeds, particularly in methanol feeds, to dehydration-hydrolysis processes may be sub-optimal for maintaining or optimising the operation of such processes. Furthermore, if such processes are operated as continuous processes, recycling of water-containing streams to the process can cause or contribute to fluctuations in the water concentration within the process. Water losses due to, for example leaks in the process can also create fluctuations in water concentration within the system. Such fluctuations need to be managed to maintain effective process operation.

Thus, there remains a need for a process for the co-production of acetic acid and dimethyl ether from methanol and methyl acetate in which the amount of water in the process can be controlled.

Summary

In an aspect, there is provided a process for the co-production of acetic acid and dimethyl ether by the dehydration-hydrolysis of a mixture of methanol and methyl acetate carried out at a temperature of 100 to 350 °C and at atmospheric or greater pressure in the presence of at least one solid acid catalyst and water to generate a reaction product

comprising dimethyl ether and acetic acid in which process the amount of water to the dehydration-hydrolysis is controlled by : dehydrating a methanol feed comprising methanol and water to generate a crude dehydration product comprising dimethyl ether, unconverted methanol and water; recovering from the crude dehydration product i) a dimethyl ether stream comprising dimethyl ether, water and methanol and ii) a water stream; separating dimethyl ether from the dimethyl ether stream to produce a methanol stream comprising methanol and water; and supplying to the dehydration-hydrolysis reaction the methanol stream or a part thereof, methyl acetate and optionally one or more recycle streams comprising one or more of methanol, methyl acetate and water.

Detailed Description

Accordingly, the present invention provides a process for the co-production of acetic acid and dimethyl ether by the dehydration-hydrolysis of a mixture of methanol and methyl acetate carried out at a temperature of 100 to 350 °C at atmospheric or greater pressure in the presence of at least one solid acid catalyst and water to generate a reaction product comprising dimethyl ether and acetic acid in which process the amount of water to

Claims:

1. A process for the co-production of acetic acid and dimethyl ether by the dehydration-hydrolysis of a mixture of methanol and methyl acetate carried out at a temperature of 100 to 350 °C and at atmospheric or greater pressure in the presence of at least one solid acid catalyst and water to generate a reaction product comprising dimethyl ether and acetic acid in which process the amount of water to the dehydration-hydrolysis is controlled by :

dehydrating a methanol feed comprising methanol and water to generate a crude dehydration product comprising dimethyl ether, unconverted methanol and water

recovering from the crude dehydration product i) a dimethyl ether stream comprising dimethyl ether, water and methanol and ii) a water stream;

separating dimethyl ether from the dimethyl ether stream to produce a methanol stream comprising methanol and water; and

supplying to the dehydration-hydrolysis reaction the methanol stream or a part thereof, methyl acetate and optionally one or more recycle streams comprising one or more of methanol, methyl acetate and water.

2. A process according to claim 1 wherein recovery of the water stream from the crude dehydration product is carried out by fractional distillation in a single distillation column equipped with a reboiler to recover the dimethyl ether stream as a heads product from the column and the water stream as a base stream from the column.

3. A process according to claim 2 wherein the amount of water recovered as a base stream from the distillation column is controlled by adjusting one or both of the reflux ratio and reboiler duty (boil-up ratio) to the column.

4. A process according to claim 2 or claim 3 wherein distillation is carried out at a reflux ratio of from 0.05 to 1.

5. A process according to claim 4 wherein the distillation is carried out at a boil-up ratio of 0.01 to 5.

6. A process according to any one of claims 2 to 5 wherein distillation is carried out at a pressure of 5 to 30 barg (500 to 3000kPa) and at a heads temperature of 120 to 180 °C.

7. A process according to any one of ~~the preceding~~ claims 1 to 6 wherein the water stream recovered from the crude dehydration product comprises ~~essentially water.~~

~~8. A process according to claim 7 wherein the water stream comprises~~ 90 mol% or more water.

98. A process according to any one of claims 2 to 8-7 wherein the dimethyl ether stream recovered as a heads product from the column comprises > 0 to 60 mol% methanol, 5 to 45 mol% water and the balance dimethyl ether.

109. A process according to any one of the preceding claims wherein the methanol feed to dehydration comprises water in an amount of from >0 to 35 mol%.

1110. A process according to any one of the preceding claims wherein the methanol feed to dehydration comprises 50 to 99 mol% methanol, >0 to 35 mol% water and 0 to 10 mol% dimethyl ether.

1211. A process according to any one of the preceding claims wherein the methanol feed to dehydration is derived from the conversion of a gaseous mixture of carbon monoxide, hydrogen and optionally carbon dioxide in the presence of a methanol synthesis catalyst.

~~13. A process according to any one of the preceding claims wherein dehydration of the methanol feed is conducted as a heterogeneous process, either as a liquid phase or vapour phase process.~~

1412. A process according to any one of the preceding claims wherein the dehydration is conducted in the vapour phase at a pressure of atmospheric to 30 barg (atmospheric to 3000kPa) and at a temperature of 100 °C to 450 °C

1513. A process according to any one of claims 1 to 13-11 wherein the dehydration is conducted in the liquid phase at a pressure of 40 to 100 barg (4000 to 10000kPa) and at a temperature of 140 °C to 210 °C.

1614. A process according to any one of the preceding claims wherein the dehydration of the methanol feed is carried out in the presence of a solid acid catalyst, ~~effective to dehydrate methanol to generate dimethyl ether and water.~~

~~17. A process according to claim 16 wherein the solid acid catalyst is selected from the group consisting of aluminas, acidic zirconias, aluminium phosphate, silica-alumina supported tungsten oxides, heteropolyacids and salts thereof and aluminosilicate zeolites.~~

~~18. A process according to claim 17 wherein the~~ which solid acid catalyst is a zeolite selected from zeolites having a 2-dimensional or 3-dimensional channel system and at least one channel of which has a 10-membered ring.

1915. A process according to any one of the preceding claims wherein dimethyl ether is separated from the dimethyl ether stream comprising dimethyl ether, water and methanol to produce a methanol stream comprising methanol and water by distillation in a single distillation

column.

~~20. A process according to claim 19 wherein the distillation column is operated at a pressure of 0.5 to 30 barg (50 to 3000kPa).~~

~~21. A process according to claim 19 or claim 20 wherein the column is operated at a reflux ratio in the range 1 to 10 and a boil-up ratio of 0.01 to 5.~~

~~22. A process according to any one of claims 18 to 21 wherein dimethyl ether is recovered as a heads product from the distillation column as a vapour.~~

~~23. A process according to any one of claims 18 to 22 wherein there is further introduced to the distillation column one or more methyl acetate-rich streams comprising 50 to 95 mol% methyl acetate and 5 to 45 mol% dimethyl ether.~~

~~24. A process according to any one of claims 18 to 23 wherein the methanol stream or a part thereof and comprising methanol, water and methyl acetate is supplied as feed to the dehydration-hydrolysis process.~~

~~25~~16. A process according to any one of the preceding claims wherein water is introduced to the dehydration-hydrolysis reaction in an amount of 0.1 to 50 mol% based on the total feed of methyl acetate, water and methanol.

~~26. A process according to any one of the preceding claims wherein the methanol stream supplied to the dehydration-hydrolysis reaction further comprises methyl acetate.~~

~~27. A process according to any one of the preceding claims wherein at least a portion of methyl acetate for supply to the dehydration-hydrolysis is recovered from a process for the carbonylation of dimethyl ether with carbon monoxide in the presence of a carbonylation catalyst.~~

~~28~~17. A process according to any one of the preceding claims wherein the solid acid catalyst for the dehydration-hydrolysis reaction is selected from one or more heteropolyacids and salts thereof and zeolites.

~~29. A process according to any one of the preceding claims wherein the dehydration-hydrolysis reaction is conducted as a vapour phase process at a temperature of 150 to 350 °C and a pressure of atmospheric to 30 barg (atmospheric to 3000kPa).~~

~~30. A process according to any one of the preceding claims wherein the dehydration-hydrolysis reaction is conducted as a liquid phase process at a temperature of 140 to 210 °C and a pressure of atmospheric to 40 barg (atmospheric to 4000kPa).~~

~~31. A process according to any one of the preceding claims wherein the dehydration-~~

~~hydrolysis is carried out in a fixed bed or slurry bed reactor.~~

~~32~~18. A process according to any one of the preceding claims wherein the process is operated as a continuous process.