wherein process water streams generated in or in connection with the process contain sufficiently low levels of acetic acid that additional processing treatment of the produced water is eliminated or mitigated. Furthermore, there is a need to treat process water streams generated by the dehydration of methanol feed stock, which methanol feed stock is derived from gaseous mixtures comprising carbon oxide(s) and hydrogen and which methanol feed stock also comprises methyl acetate as a contaminant and, in particular methanol feed stocks derived from gaseous mixtures comprising carbon oxide(s) and hydrogen and methyl acetate contaminant recovered from crude carbonylation product streams produced by carbonylating dimethyl ether with a carbon monoxide-containing gas in the presence of a carbonylation catalyst.

Summary

In an aspect, there is provided a process for the production of dimethyl ether from gaseous mixtures of carbon monoxide, hydrogen and methyl acetate contaminant which process comprises : contacting a gaseous mixture of carbon monoxide, hydrogen and methyl acetate contaminant in a first scrubbing zone with a first portion of methanol to recover a scrubbed gaseous mixture depleted in methyl acetate and a first used methanol stream containing methyl acetate; contacting the scrubbed gaseous mixture in a second scrubbing zone with a second portion of methanol to recover a scrubbed gaseous mixture further depleted in methyl acetate and a second used methanol stream containing no methyl acetate or a reduced amount of methyl acetate compared to the first used methanol stream; dehydrating at least a portion of the second used methanol stream in the presence of at least one catalyst to produce a crude dehydration reaction product comprising dimethyl ether, unconverted methanol and water; recovering from the crude dehydration product a water stream comprising mainly water and 3 mol% or less acetic acid and a dimethyl ether stream.

In another aspect, there is provided a process for the co-production of acetic acid and dimethyl ether by dehydration-hydrolysis of methanol and methyl acetate which process comprises : contacting a gaseous mixture of carbon monoxide, hydrogen and methyl acetate contaminant in a first scrubbing zone with a first portion of methanol to recover a scrubbed gaseous mixture depleted in methyl acetate and a first used methanol stream containing methyl acetate; contacting the scrubbed gaseous mixture in a second scrubbing zone with a second portion of methanol to recover a scrubbed gaseous mixture

further depleted in methyl acetate and a second used methanol stream containing a reduced amount of methyl acetate compared to the first used methanol stream; dehydrating at least a portion of the second used methanol stream in the presence of at least one catalyst to produce a crude dehydration reaction product comprising dimethyl ether, unconverted methanol, water and acetic acid; recovering from the crude dehydration product a water stream comprising mainly water and 3 mol% or less acetic acid and a dimethyl ether stream comprising dimethyl ether and methanol; separating dimethyl ether from the dimethyl ether stream to produce a methanol stream comprising methanol and water; and contacting 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 in the presence of at least one catalyst to generate a dehydration-hydrolysis reaction product comprising dimethyl ether and acetic acid.

Detailed Description

Accordingly, the present invention provides a process for the production of dimethyl ether from gaseous mixtures of carbon monoxide, hydrogen and methyl acetate contaminant which process comprises :

contacting a gaseous mixture of carbon monoxide, hydrogen and methyl acetate contaminant in a first scrubbing zone with a first portion of methanol to recover a scrubbed gaseous mixture depleted in methyl acetate and a first used methanol stream containing methyl acetate;

contacting the scrubbed gaseous mixture in a second scrubbing zone with a second portion of methanol to recover a scrubbed gaseous mixture further depleted in methyl acetate and a second used methanol stream containing no methyl acetate or a reduced amount of methyl acetate compared to the first used methanol stream;

dehydrating at least a portion of the second used methanol stream in the presence of at least one catalyst to produce a crude dehydration reaction product comprising dimethyl ether, unconverted methanol and water;

recovering from the crude dehydration product a water stream comprising mainly water and 3 mol% or less acetic acid and a dimethyl ether stream.

The present invention further provides a process for the co-production of acetic acid and dimethyl ether by dehydration-hydrolysis of methanol and methyl acetate which process comprises :

contacting a gaseous mixture of carbon monoxide, hydrogen and methyl acetate contaminant in a first scrubbing zone with a first portion of methanol to recover a scrubbed gaseous mixture depleted in methyl acetate and a first used methanol stream containing

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

contacting 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 in the presence of at least one catalyst to generate a dehydration-hydrolysis reaction product comprising dimethyl ether and acetic acid.

3. A process according to claim 1 or claim 2 wherein the gaseous mixture provided to the first scrubbing zone comprises methyl acetate in an amount of >0 to 5 mol%.

4. A process according to any one of claims 1 to 3 wherein the water stream recovered from the crude dehydration product comprises water in an amount of at least 95 mol% and 0 to 1 mol% acetic acid.

~~5. A process according to any one of claims 1 to 4 wherein the water stream is recovered from the crude dehydration product by fractional distillation in a distillation column equipped with a reboiler.~~

~~6. A process according to claim 5 wherein distillation is carried out at a reflux ratio of from 0.05 to 1.~~

~~7~~5. A process according to any one of claims 1 to ~~6~~4 wherein the first and second scrubbing zones are integrated within a single scrubbing column.

~~8~~6. A process according to any one of claims 1 to ~~7~~5 wherein the first portion of methanol and the second portion of methanol are provided from a single methanol feed.

~~9~~7. A process according to claim ~~8~~6 wherein the single methanol feed is split to provide a ratio of the amount of the first portion of methanol to the amount of the second portion of methanol in the range 1 : 5 to 1 : 15.

~~10. A process according to any one of claims 1 to 9 wherein one or both of the first and second methanol portions comprises a mixture of methanol, water and dimethyl ether and wherein water and dimethyl ether are present in the mixture in a total amount of >0 to 35 mol%.~~

~~11~~8. A process according to any one of claims 1 to ~~10~~7 wherein the ratio of the amount of the first portion of methanol to the amount of the second portion of methanol is in the range 1 : 5 to 1 : 15.

~~12~~9. A process according to any one of claims 1 to ~~11~~8 wherein the first portion of methanol removes ~~50~~90 to <100 wt% methyl acetate from the gaseous mixture.

~~13. A process according to claim 12 wherein the first portion of methanol removes 90 to <100 wt% methyl acetate from the gaseous mixture.~~

~~14~~10. A process according to any one of claims 1 to ~~13~~9 wherein the second used methanol stream comprises 0 to 0.5 mol% methyl acetate.

~~15. A process according to any one of claims 1 to 14 wherein the gaseous mixture comprising carbon monoxide, hydrogen and methyl acetate contaminant contacted with the first portion of methanol is a gaseous mixture recovered from a crude carbonylation product comprising methyl acetate, unreacted carbon monoxide and hydrogen, which crude carbonylation product is produced by carbonylating dimethyl ether with a carbon monoxide-containing gas in the presence of a carbonylation catalyst and hydrogen.~~

~~16~~11. A process according to any of claims 1 to ~~15~~10 wherein dehydration of the second used methanol stream is conducted as a heterogeneous process ~~at a temperature of from 100 °C to 450 °C.~~

~~17. A process according to claim 16~~and wherein the heterogeneous process is conducted in the vapour phase at temperatures of from 150 °C to 300 °C.

~~18~~12. A process according to any one of claims 1 to ~~17~~11 wherein the water stream recovered from the crude dehydration product is recovered by fractional distillation in a distillation column and the amount of water present in the water stream withdrawn from the column is controlled by adjusting one or both of the reflux ratio to the column and the reboiler duty.

13. A process according to any one of claims 1 to 12 wherein the gaseous mixture comprising carbon monoxide, hydrogen and methyl acetate contaminant contacted with the first portion of methanol is a gaseous mixture recovered from a crude carbonylation product comprising methyl acetate, unreacted carbon monoxide and hydrogen, which crude carbonylation product is produced by carbonylating dimethyl ether with a carbon monoxide-containing gas in the presence of a carbonylation catalyst and hydrogen.

~~19. A process according to any one of claims 1 to 18 wherein the first used methanol stream or a portion thereof is supplied to a distillation column and distilled therein with the dimethyl ether stream recovered from the crude dehydration product to recover from the distillation column, dimethyl ether and a methanol stream comprising methanol, water and methyl acetate.~~

~~20~~14. A process according to any one of claims 1 to ~~19~~13 wherein the first and second portions of methanol are provided by methanol produced from a methanol synthesis process in which synthesis process a gaseous reactant mixture of carbon monoxide, hydrogen and carbon dioxide is fed to a methanol synthesis reactor and contacted therein in the presence of a methanol synthesis catalyst to produce a methanol product and which methanol synthesis process forms an integrated process with the process of any one of claims 1 to 13~~19~~.

~~21~~15. A process according to claim ~~20~~14 wherein the gaseous mixture or a part thereof recovered from the second scrubbing zone is supplied as a feed to the methanol synthesis process.

~~22~~16. A process according to any one of claims 1 to ~~21~~15 wherein one or more of the dehydration of methanol and the dehydration-hydrolysis of methanol and methyl acetate is carried out in the presence of a solid acid catalyst.

~~23~~17. A process according to claim ~~22~~16 wherein the solid acid catalyst is selected from aluminas, acidic zirconia, aluminium phosphate, silica-alumina supported tungsten oxides, heteropolyacids and salts thereof and aluminosilicate zeolites.

~~24~~18. A process according to any one of claims 2 to ~~23~~17 wherein one or more recycle streams comprising one or more of methanol, methyl acetate and water are returned to dehydration-hydrolysis.

~~25~~19. A process according to any one of claims 2 to ~~24~~18 wherein dehydration-hydrolysis is carried out at temperatures in the range 100 to 350 °C and at atmospheric pressure or pressures greater than atmospheric.

~~26~~20. A process according to any one of claims 2 to ~~25~~19 wherein the co-production process is operated as a continuous process.