

Improved process for preparing acetaldehyde from
ethylene and oxygen

The invention relates to a process for oxidizing a
5 reaction gas in the presence of a catalyst solution, in
which process

a) the reaction gas is oxidized in a reactor in
the presence of the catalyst solution, a reaction
10 mixture being formed which comprises droplets of
the catalyst solution, and which

b) is passed into a first separator and in this
droplets of the catalyst solution are removed from
15 the reaction mixture, and in which process

c) the remaining reaction mixture is passed from
the first separator into a scrubbing apparatus and
in this the oxidation product is removed from the
20 reaction mixture.

Said process is known from the prior art (DE 1 190 451)
and is used for the industrial-scale preparation of
aldehydes, ketones or the acids corresponding to the
25 aldehydes by oxidizing olefins. In particular,
acetaldehyde is prepared from ethylene and oxygen by
this process. To catalyze the highly exothermic
oxidation reaction with oxygen as oxidizing agent,
customarily an aqueous solution of copper chloride and
30 palladium chloride is used.

The oxidation takes place in a reactor at the boiling
point of the aqueous catalyst solution. The heat of
reaction is primarily removed by evaporating water and
35 acetaldehyde from the catalyst solution (evaporative
cooling). The resultant reaction mixture essentially
comprising liquid catalyst, steam, gaseous acetaldehyde
and mist of catalyst solution is passed into a first
separator, which is termed a mist eliminator, in which
40 the vapor/gas mist contents are for the most part

removed from the reaction mixture, for example using scrubbing water which is injected into the mist eliminator in countercurrent. The remaining reaction mixture is then cooled in heat exchangers, catalyst-
5 containing condensate streams, some of which already have a significant acetaldehyde content, being produced, and being scrubbed out with water in a scrubbing apparatus, customarily a scrubbing column, the majority of the acetaldehyde still present in the
10 gas phase dissolving in the water. The scrubbing phase, combined with the condensate streams distinguished by an elevated acetaldehyde concentration, is taken off for workup. The reaction mixture then remaining which still comprises small amounts of acetaldehyde is
15 recycled to the reactor as cycle gas. Catalyst-containing condensate having only a low acetaldehyde content is used as scrubbing water for the mist eliminator.

20 It is a disadvantage of the known process that the catalyst separation is not complete. This leads, firstly, to losses of valuable noble metal catalyst and copper, which can also disadvantageously affect the oxidation reaction, and, secondly, to increased
25 disposal costs, since the catalyst and the copper pass into the wastewater of the workup unit and have to be disposed of with this in an environmentally compatible manner.

30 The object underlying the invention therefore was to improve the separation of the catalyst.

This object is achieved according to the invention by passing the reaction mixture from the first separator
35 through a second separator which is downstream of the first separator and upstream of the scrubbing apparatus and, in the second separator, removing residual catalyst solution from the reaction mixture.

The invention thus relates to a process for oxidizing a reaction gas in the presence of a catalyst solution, in which process

5 a) the reaction gas is oxidized in a reactor in the presence of the catalyst solution, a reaction mixture being formed which comprises droplets of the catalyst solution, and which

10 b) is passed into a first separator and in this droplets of the catalyst solution are removed from the reaction mixture, and in which process

15 c) the remaining reaction mixture is passed from the first separator into a scrubbing apparatus and in this the oxidation product is removed from the reaction mixture,

which comprises

20 d) passing the reaction mixture from the first separator through a second separator which is downstream of the first separator and upstream of the scrubbing apparatus and, in the second
25 separator, removing residual catalyst solution from the reaction mixture.

The invention relates in particular to the corresponding process for preparing acetaldehyde from
30 ethylene and oxygen. Preferred developments are given by the subclaims. Individual or a plurality of the developments disclosed in the subclaims can also each individually or in combination represent solutions of the underlying object and the individual features can
35 also be combined as desired within the claim categories.

The second separators used can advantageously be what are termed centrifugal separators or centrifugal mist

collectors. In these, the flow of the reaction mixture is forced into a spiral path by appropriate internals such as deflection blades, so that a rotary flow is formed, as a result of which, owing to the centrifugal forces arising, droplets present in the gas stream are deposited on the outer inner wall of the separator. The deposited droplets can be collected, for example, in a surrounding channel, taken off and recycled to the first scrubbing apparatus as scrubbing water.

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The solution of the object underlying the invention is surprising in as far as those skilled in the art could not reckon on the gas stream from the first scrubbing apparatus arising continuously, since in exothermic reactions which are carried out under boiling conditions (evaporative cooling) greatly fluctuating gas flows are to be expected, which argues against the use of a centrifugal separator, in particular when the actual pressure drop in the separator is not known or cannot be determined as a result of such greatly variable gas flows. Accordingly, no applications are known of such separators for gas flows in the saturated vapor area, i.e. under conditions under which gas and liquid are present in equilibrium. In addition, under these conditions, the droplet size distribution is unknown and also cannot be determined. The droplets could therefore have also been too small for a successful separation.

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In addition, the disposition of the separator was chosen in opposition to the customary conditions familiar to those skilled in the art, i.e. the flow through the vertically disposed separator takes place from top to bottom, the flow therefore has a principal component in the direction of gravity. Furthermore there was also the risk that the deflection blades would become coated with catalyst deposits, which would have impaired the acceleration of the gas flow in the peripheral direction (spin generation). It is further

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surprising that neither a spiral for collecting the liquid separated off nor a flushing connection are required.

- 5 The invention therefore likewise relates to the use of a centrifugal separator for separating off droplets from gas streams in the saturated vapor region.

10 The advantages of the process according to the invention are that, inter alia, the loss of valuable noble metal catalyst and, accompanying this, the pollution of the plant wastewater with heavy metals, are decreased. Furthermore, it has been found that damage due to pitting and/or stress-cracking corrosion
15 decreases or is avoided on those apparatuses and plant components downstream of the centrifugal separator, since less catalyst solution which strongly promotes pitting corrosion and stress-corrosion cracking passes into these plant components. Therefore, cheaper
20 materials than hitherto can then be used for these apparatuses. In addition, fouling phenomena (deposits) decrease in the scrubbing column. A further beneficial effect is that under otherwise identical conditions the production of acetaldehyde can be increased, for the
25 following particular reasons: If the cycle gas stream is increased, which is beneficial from the aspect of greater production, this leads to increased foaming of the catalyst solution and thus to a markedly increased discharge of catalyst solution from the reactor to the
30 downstream apparatuses, with said disadvantages. Using the separator according to the invention, this behavior is no longer so disadvantageous, since, owing to the excellent separation, the catalyst solution which is discharged more intensively can be recirculated again.

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An exemplary development of the invention is described in more detail below with reference to the process flow chart depicted in the diagram and with reference to

operational experience. A restriction of the invention in any manner not intended thereby.

The known part of the process has already been
5 described above, for which reason only the reference numbers will be mentioned here. These are: 1 reactor, 2 ethylene, 3 oxygen, 4 reaction mixture, 5 first separator (mist eliminator), 6 scrubbing water, 7 a first cooler, 8 a second cooler, 9 catalyst-containing
10 condensate, 10 scrubbing apparatus (scrubbing column), 11 water and 12 acetaldehyde-containing water.

According to the invention, a centrifugal separator 13 is disposed vertically as second separator downstream
15 of the first scrubbing apparatus 5 and upstream of the scrubbing column 10 and, in particular, upstream of the first cooler 7. In this centrifugal separator, the reaction mixture 4 is conducted downwards in a rotary flow (arrow) by means of deflection blades (which are
20 not shown). The centrifugal separator 13 has a peripheral channel 14 on the interior. From this channel 14, the catalyst solution which has been separated off is removed, mixed with the catalyst-containing condensate 9 which was produced from the
25 first cooler 7 and injected as scrubbing water 6 into the first scrubbing apparatus 5.

Operational experience:

30 In a commercial industrial plant for preparing acetaldehyde from ethylene and oxygen, a centrifugal mist collector was installed according to the invention in accordance with the figure. Result: The plant capacity was increased from 120,000 metric t/yr of
35 acetaldehyde to 135,000 metric t/yr, and the pollution of wastewater from the plant by copper and palladium decreased by a factor of 50.

Patent claims

1. A process for oxidizing a reaction gas in the presence of a catalyst solution, in which process
- 5 a) the reaction gas is oxidized in a reactor in the presence of the catalyst solution, a reaction mixture being formed which comprises droplets of the catalyst solution, and which .
- 10 b) is passed into a first separator and in this droplets of the catalyst solution are removed from the reaction mixture, and in which process
- 15 c) the remaining reaction mixture is passed from the first separator into a scrubbing apparatus and in this the oxidation product is removed from the reaction mixture,
- 20 which comprises
- d) passing the reaction mixture from the first separator through a second separator which is downstream of the first separator and upstream of
- 25 the scrubbing apparatus and, in the second separator, removing residual catalyst solution from the reaction mixture.
2. The process as claimed in claim 1, wherein the residual catalyst solution is separated off by conducting the reaction mixture in the second separator in a rotary flow, so that the catalyst solution droplets separate off on the inner wall of the second separator.
- 35 3. The process as claimed in claim 1 or 2, wherein, to generate the rotary flow, deflection blades are used.



4. The process as claimed in one or more of claims 1 to 3, wherein the reaction gas is an olefin, in particular ethylene.
- 5 5. A separator for carrying out the process as claimed in one or more of claims 1 to 4, wherein the separator is a centrifugal separator and has means for generating a rotary flow.
- 10 6. The separator as claimed in claim 5, wherein the means are deflection blades.
7. The separator as claimed in claim 5 or 6, wherein the separator is fabricated from titanium.
- 15 8. The use of a centrifugal separator for separating off droplets from gas streams in the saturated vapor region.

