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(54) Title: EXTRACTING AN AMINOALKANE FROM AN AQUEOUS FEED

(57) Abstract: The present invention provides a process of extracting an aminoalkane from an aminoalkane-containing aqueous feed (5) comprising (a) contacting (7) the aqueous feed (5) with a depleted solvent comprising an alkylated phenol to obtain a first extract (12) enriched in aminoalkane and a first raffinate (2) having a reduced aminoalkane-content; (b) contacting (15) the first extract (12) with an expelling agent (17) and sufficient water (20) to dissolve the expelled aminoalkane to obtain a second extract enriched in aminoalkane (22) and a second raffinate including the expelling agent (25); (c) withdrawing the second extract (22) from the second raffinate (25) to obtain an aqueous product stream enriched in aminoalkane (22); and (d) removing expelling agent (39) from the second raffinate (25) to obtain the depleted solvent (9) for use in step (a).



EXTRACTING AN AMINOALKANE FROM AN AQUEOUS FEED

Field of the invention

The present invention relates to a process of extracting an aminoalkane, such as a monoaminoalkane or a diaminoalkane, such as diaminobutane or diaminopentane, or diaminohexane from an aminoalkane-containing aqueous feed.

5 Aminoalkanes are important industrial chemicals as monomers for polyamide production, for example 1,4-diaminobutane is used in the production of polyamide 4,6 or polyamide 4,10 formed by reacting 1,4-diaminobutane with adipic acid or sebacic acid respectively, or as raw material for several fine chemicals purposes.

10 A process of producing industrial chemicals such as aminoalkanes that is currently drawing interest is fermentation, in which suitable microorganisms produce the aminoalkane in an aqueous environment in a vessel that is called a fermentor.

15 Although in fermentation the aminoalkanes are produced in small quantities, the presence of aminoalkanes will lead to unfavourable pH conditions in the fermentor in which the fermentation is carried out so that the microorganisms are adversely affected during the process.

For this reason the present invention is related to removing aminoalkanes from the fermentor content. More in particular, the present invention is related to removing the aminoalkane from the aqueous stream that is withdrawn from the fermentor to obtain a purified aqueous stream that can be returned to the fermentor.

20 Background of the invention

25 USA patent specification No. 3 433 788 discloses a process of extracting an aminoalkane, such as a monoaminoalkane or a diaminoalkane, such as diaminobutane, from an aqueous aminoalkane-containing feed comprising the steps of (a) contacting the aqueous feed with a depleted solvent including an alkylated phenol to obtain a first extract enriched in aminoalkane and a first raffinate having a reduced aminoalkane-content; (b) separating the first raffinate from the first extract; and (c) recovering the aminoalkane from the first extract to obtain an aminoalkane-containing stream and a depleted solvent for use in step (a), wherein recovering the aminoalkane involves a distillation step.

30 USA patent specification No. 4 582 937 discloses a process of extracting an ethyleneamine from an aqueous ethyleneamine-containing feed comprising the steps

of (a) contacting the aqueous feed with a depleted solvent including an alkylated phenol to obtain a first extract enriched in ethyleneamine and a first raffinate having a reduced ethyleneamine-content; (b) separating the first raffinate from the first extract; and (c) recovering the ethyleneamine from the first extract to obtain a depleted solvent for use in step (a), wherein recovering the ethyleneamine comprises contacting the first extract with an aqueous carbonic acid solution or carbon dioxide gas to back-extract the ethyleneamine in the form of a salt.

In case the aqueous aminoalkane-containing stream is produced by means of fermentation in a fermentor, the first raffinate having a reduced aminoalkane-content can be used as the purified aqueous stream that can be returned to the fermentor.

A disadvantage of the first of the two known processes is that recovering the aminoalkane involves an energy consuming distillation step at elevated temperatures to achieve separation. A disadvantage of the second process is that recovering the aminoalkane involves using an acid to obtain a salt of the aminoalkane, which salt subsequently has to be converted to obtain the aminoalkane.

Summary of the invention

There is a need in the art for a process that overcomes the disadvantages.

To this end the present invention relates to a process of extracting an aminoalkane from an aminoalkane-containing aqueous feed comprising the steps of

(a) contacting the aqueous feed with a depleted solvent comprising an alkylated phenol to obtain a first extract enriched in aminoalkane and a first raffinate having a reduced aminoalkane-content;

(b) separating the first raffinate from the first extract; and

(c) recovering the aminoalkane from the first extract to obtain an aminoalkane-containing stream and a depleted solvent for use in step (a),

wherein recovering the aminoalkane comprises contacting the first extract with an expelling agent which is immiscible with water, selected from the group including C₅-C₁₅ alkanes and C₅-C₂₀ fatty alcohols, and sufficient water to dissolve the expelled

aminoalkane to obtain a second extract enriched in aminoalkane and a second raffinate including the expelling agent, separating the second extract from the second raffinate to obtain an aqueous product stream enriched in aminoalkane, and removing

expelling agent from the second raffinate to obtain the depleted solvent for use in step (a).

In the recovery stage of the process according to the present invention, the aminoalkane is present in the mixture of first extract and expelling agent and in the water, an aqueous phase. Surprisingly it was found that the concentration of aminoalkane in the aqueous phase steeply increases with increasing concentration of expelling agent.

The expelling agent, selected from the group including C₅-C₁₅ alkanes and C₅-C₂₀ fatty alcohols, is immiscible with water and miscible with the solvent.

Detailed description of the invention

The invention will now be described by way of example in more detail with reference to the accompanying Figure 1 showing schematically a plant for producing aminoalkanes and the process according to the present invention for extracting the aminoalkanes.

In a fermentor 1, a process of producing an aminoalkane is carried out wherein suitable microorganisms produce the aminoalkane in an aqueous environment in the presence of a nutrient. A purified aqueous stream is supplied to the fermentor 1 through conduit 2, and an aqueous stream containing aminoalkane is withdrawn from the fermentor through conduit 5. The aqueous stream in conduit 5 is the aminoalkane-containing aqueous feed for the extraction process of the present invention. For the sake of clarity, we do not show auxiliary equipment, such as filters and pumps.

The aqueous feed 5 is contacted in a first contactor 7 with a depleted solvent comprising an alkylated phenol which is supplied to the first contactor 7 through conduit 9, to obtain a first extract enriched in aminoalkane and a first raffinate having a reduced aminoalkane-content. Using a solvent with a low mutual solubility towards water in this contacting step will be beneficial to the overall process economy. After separation, the first extract enriched in aminoalkane is withdrawn from the first contactor 7 through conduit 12, and the first raffinate having a reduced aminoalkane-content is withdrawn from the first contactor 7 through conduit 2. Via

conduit 2 the first raffinate is supplied as the purified aqueous stream to the fermentor 1.

Next, the aminoalkane is recovered from the first extract 12 to obtain an aminoalkane-containing stream and a depleted solvent.

5 In order to recover the aminoalkane from the first extract 12, the first extract is supplied to a second contactor 15 in which the first raffinate is contacted with an expelling agent supplied to the second contactor 15 through conduit 17. Moreover, the first extract is also contacted with just sufficient water, supplied through conduit 20 to dissolve the expelled aminoalkane. In the second contactor 15 are obtained a second
10 extract enriched in aminoalkane and a second raffinate including the expelling agent. After separation, the second extract enriched in aminoalkane is withdrawn from the second contactor 15 through conduit 22. This second extract is the aqueous product stream enriched in aminoalkane. The aminoalkane may subsequently be recovered by any suitable means (not shown).

15 The second raffinate including the expelling agent is withdrawn from the second contactor 15 through conduit 25.

In order to remove the expelling agent from the second raffinate, the second raffinate is heated in heat exchanger 30 so as to evaporate the expelling agent, and supplied to separation vessel 35 to allow separation of the vaporized expelling agent
20 from the second raffinate. The expelling agent is withdrawn from the separation vessel 35 through conduit 39, condensed in heat exchanger 40 and returned through conduit 17 to the second contactor 15. Depleted solvent is passed from the separation vessel 35 through conduit 9 to the first contactor 7.

The contactors 7 and 15 can be any suitable contactors known in the art, such as
25 counter-current contactors.

The invention will now be described by way of example in more detail with reference to the below examples.

In the examples the content of 1,4-diaminobutane and 1,5-diaminopentane in water has been determined with gas chromatography. A Varian WCOT Fused Silica
30 CP Volamine capillary column with dimensions 15x0.32mm inner diameter had been used in a Carlo Erba 8180 gas chromatograph, equipped with an electrometer control module EL 980. A split/splitless injector and FID detector were used.

The amines used were 1,4-diaminobutane (99%) and 1,5-diaminopentane (97%). The solvent used was 4-nonylphenol (99%, mixture of isomers). The expelling agents used were octanol (99% Reagent grade), 2-octyl-1-dodecanol (97%) and heptanes (99% Chromasolv).

5 Example 1 relates to extraction of 1,4-diaminobutane from an aqueous feed; examples 2 and 2A relate to recovering 1,4 diaminobutane from a first extract, wherein example 2A is a comparative example not according to the invention; examples 3 and 4 relate to recovering 1,4-diaminobutane from a first extract; example 5 relates to extraction of 1,5-diaminopentane from an aqueous feed, and examples 6
10 and 6A relate to recovering 1,5-diaminopentane from a first extract, wherein example 6A is a comparative example not according to the invention.

Example 1: Extraction of 1,4-diaminobutane from an aqueous feed.

15 In Example, 1 an aminoalkane in the form of 1,4-diaminobutane was extracted from an aqueous feed of 1,4-diaminobutane (1 wt% in water, pH = 12.09). The extraction process comprised contacting for 17 hours in an incubator 0.010 kg of the aqueous feed with 0.010 kg depleted solvent consisting of 100 wt% 4-nonylphenol, to obtain a first extract enriched in aminoalkane and a first raffinate having a reduced
20 aminoalkane-content.

 The formation of the complex between aminoalkane and 4-nonylphenol could be observed by the change of the color of organic phase from transparent to dark yellow. Afterwards the phases were allowed to settle for 2 hours and a sample of first raffinate was taken. The pH of the first raffinate was measured and a decrease to
25 pH = 10.177 was observed. Analysis showed that the concentration of 1,4-diaminobutane in the first raffinate was less than 0.006 wt%. Hence, 99.4% of the 1,4-diaminobutane was extracted into the first extract.

Example 2: Recovering 1,4-diaminobutane from a first extract.

In Example 2, an aminoalkane in the form of 1,4-diaminobutane was recovered from 0.002 kg of a first extract consisting of 0.994 wt% 1,4-diaminobutane in 4-nonylphenol to obtain an aminoalkane-containing stream and a depleted solvent for use in the extraction step. Recovering the aminoalkane comprises mixing the first extract with an expelling agent in the form of 0.008 kg octanol and 0.010 kg water to dissolve the expelled aminoalkane for 17 hours in a temperature controlled incubator to obtain a second extract enriched in aminoalkane and a second raffinate including the expelling agent. The second extract was allowed to separate from the second raffinate to obtain an aqueous product stream enriched in aminoalkane. Analysis showed that the concentration of 1,4-diaminobutane in the aqueous product stream was 0.128 wt% and pH increased to 11.29. Hence, 65% of 1,4-diaminobutane had been recovered.

Example 2A: Recovering 1,4-diaminobutane from a first extract (comparative example not according to the invention without expelling agent).

In Example 2A, an aminoalkane in the form of 1,4-diaminobutane was recovered from 0.002 kg of a first extract consisting of 0.994 wt% 1,4-diaminobutane in 4-nonylphenol to obtain an aminoalkane-containing stream and a depleted solvent for use in the extraction step. Recovering the aminoalkane comprises mixing 0.002 kg of the first extract and 0.010 kg water to dissolve aminoalkane for 17 hours in a temperature controlled incubator to obtain a second extract enriched in aminoalkane and a second raffinate including 4-nonylphenol. The second extract was allowed to separate from the second raffinate to obtain an aqueous product stream enriched in aminoalkane. Analysis showed that the concentration of 1,4-diaminobutane in the aqueous product stream was below the detection limit for 1,4-diaminobutane, i.e. 0.0088wt% and pH increased to 10.21. Hence, less than 4.4% of 1,4-diaminobutane was recovered.

Example 3: Recovering 1,4-diaminobutane from a first extract.

In Example 3, an aminoalkane in the form of 1,4-diaminobutane was recovered from 0.002 kg of a first extract consisting of 0.994 wt% 1,4-diaminobutane in 4-nonylphenol to obtain an aminoalkane-containing stream and a depleted solvent for use in the extraction step. Recovering the aminoalkane comprises mixing the first
5 extract with an expelling agent in the form of 0.008 kg 2-octyl-1-dodecanol and 0.010 kg water to dissolve the expelled aminoalkane for 17 hours in a temperature controlled incubator to obtain a second extract enriched in aminoalkane and a second raffinate including the expelling agent. The second extract was allowed to separate from the second raffinate to obtain an aqueous product stream enriched in
10 aminoalkane. Analysis showed that the concentration of 1,4-diaminobutane in the aqueous product stream was 0.129 wt% and pH increased to 11.212. Hence, 65% of 1,4-diaminobutane had been recovered.

Example 4: Recovering 1,4-diaminobutane from a first extract

In Example 4, an aminoalkane in the form of 1,4-diaminobutane was recovered from 0.002 kg of a first extract consisting of 1.12 wt% 1,4-diaminobutane in 4-nonylphenol to obtain an aminoalkane-containing stream and a depleted solvent for use in the extraction step. Recovering the aminoalkane comprises mixing the first
20 extract with an expelling agent in the form of 0.008 kg heptane and 0.010 kg water to dissolve the expelled aminoalkane for 17 hours at 25 °C in a temperature controlled incubator to obtain a second extract enriched in aminoalkane and a second raffinate including the expelling agent. The second extract was allowed to separate from the second raffinate to obtain an aqueous product stream enriched in aminoalkane.
25 Analysis showed that the concentration of 1,4-diaminobutane in the aqueous product stream was 0.026 wt% and pH increased to 10.6. Hence, 11.6% of 1,4-diaminobutane had been recovered.

Example 5: Extraction of 1,5-diaminopentane from an aqueous feed.

In Example 5, an aminoalkane in the form of 1,5-diaminopentane was extracted from an aqueous feed of 1,5-diaminopentane (1 wt% in water, pH = 11.886). The extraction process comprised contacting for 17 hours in an incubator 0.010 kg of the

aqueous feed with 0.010 kg depleted solvent consisting of 100 wt% 4-nonylphenol, to obtain a first extract enriched in aminoalkane and a first raffinate having a reduced aminoalkane-content.

The formation of the complex between aminoalkane and 4-nonylphenol could be observed by the change of the color of organic phase from transparent to dark yellow. Afterwards the phases were allowed to settle for 2 hours and a sample of first raffinate was taken. The pH of the first raffinate was measured and a decrease to pH = 9.734 was observed. Analysis showed that the concentration of 1,5-diaminopentane in the first raffinate was less than 0.005 wt%. Hence, 99.5% of the 1,5-diaminopentane was extracted into the first extract.

Example 6: Recovering 1,5-diaminopentane from a first extract.

In Example 6, an aminoalkane in the form of 1,5-diaminopentane was recovered from 0.002 kg of a first extract consisting of 1.30 wt% 1,5-diaminopentane in 4-nonylphenol to obtain an aminoalkane-containing stream and a depleted solvent for use in the extraction step. Recovering the aminoalkane comprises mixing the first extract with an expelling agent in the form of 0.008 kg 2-octyl-1-dodecanol and 0.010 kg water to dissolve the expelled aminoalkane for 17 hours at 25 °C in a temperature controlled incubator to obtain a second extract enriched in aminoalkane and a second raffinate including the expelling agent. The second extract was allowed to separate from the second raffinate to obtain an aqueous product stream enriched in aminoalkane. Analysis showed that the concentration of 1,5-diaminopentane in the aqueous product stream was 0.093 wt% and pH increased to 11.3. Hence, 36% of 1,5-diaminopentane had been recovered.

Example 6A: Recovering 1,5-diaminopentane from a first extract (comparative example not according to the invention without expelling agent).

In Example 6A, an aminoalkane in the form of 1,5-diaminopentane was recovered from 0.002 kg of a first extract consisting of 1.30 wt% 1,5-diaminopentane in 4-nonylphenol to obtain an aminoalkane-containing stream and a depleted solvent for use in the extraction step. Recovering the aminoalkane comprises mixing 0.002 kg

of the first extract and 0.010 kg water to dissolve aminoalkane for 17 hours in a temperature controlled incubator to obtain a second extract enriched in aminoalkane and a second raffinate including 4-nonylphenol. The second extract was allowed to separate from the second raffinate to obtain an aqueous product stream enriched in aminoalkane. Analysis showed that the concentration of 1,5-diaminopentane in the aqueous product stream was below the detection limit for 1,5-diaminopentane, i.e. 0.0102wt% and pH increased to 10.116. Hence, less than 3.9% of 1,5-diaminopentane was recovered.

Examples 2 and 2A and 6 and 6A show clearly the effect of the expelling agent.

Suitably, the expelling agent is a C₅-C₁₅ alkane, for example heptane.

Alternatively, the expelling agent is a C₅-C₂₀ alcohol, such as octanol or an octyldodecanol.

Suitably the alkylated phenol in the solvent used in step (a) is a nonylphenol or a di-alkylated-phenol. The solvent may contain a suitable diluent.

Claims

1. Process of extracting an aminoalkane from an aminoalkane-containing aqueous feed comprising the steps of

(a) contacting the aqueous feed with a depleted solvent comprising an alkylated phenol to obtain a first extract enriched in aminoalkane and a first raffinate having a reduced aminoalkane-content;

(b) separating the first raffinate from the first extract; and

(c) recovering the aminoalkane from the first extract to obtain an aminoalkane-containing stream and a depleted solvent for use in step (a),

wherein recovering the aminoalkane comprises contacting the first extract with an expelling agent which is immiscible with water, selected from the group including C₅-C₁₅ alkanes and C₅-C₂₀ fatty alcohols, and sufficient water to dissolve the expelled aminoalkane to obtain a second extract enriched in aminoalkane and a second raffinate including the expelling agent, separating the second extract from the second raffinate to obtain an aqueous product stream enriched in aminoalkane, and removing expelling agent from the second raffinate to obtain the depleted solvent for use in step (a).

2. Process according to claim 1, wherein the aminoalkane is a monoaminoalkane.

3. Process according to claim 1, wherein the aminoalkane is diaminoalkane

4. Process according to claim 3, wherein the diaminoalkane is a diaminobutane

5. Process according to claim 3, wherein the diaminoalkane is a diaminopentane.

6. Process according to claim 1, wherein the expelling agent is heptane.

7. Process according to claim 1, wherein the expelling agent is octanol.

8. Process according to claim 1, wherein the expelling agent is an octyldodecanol.

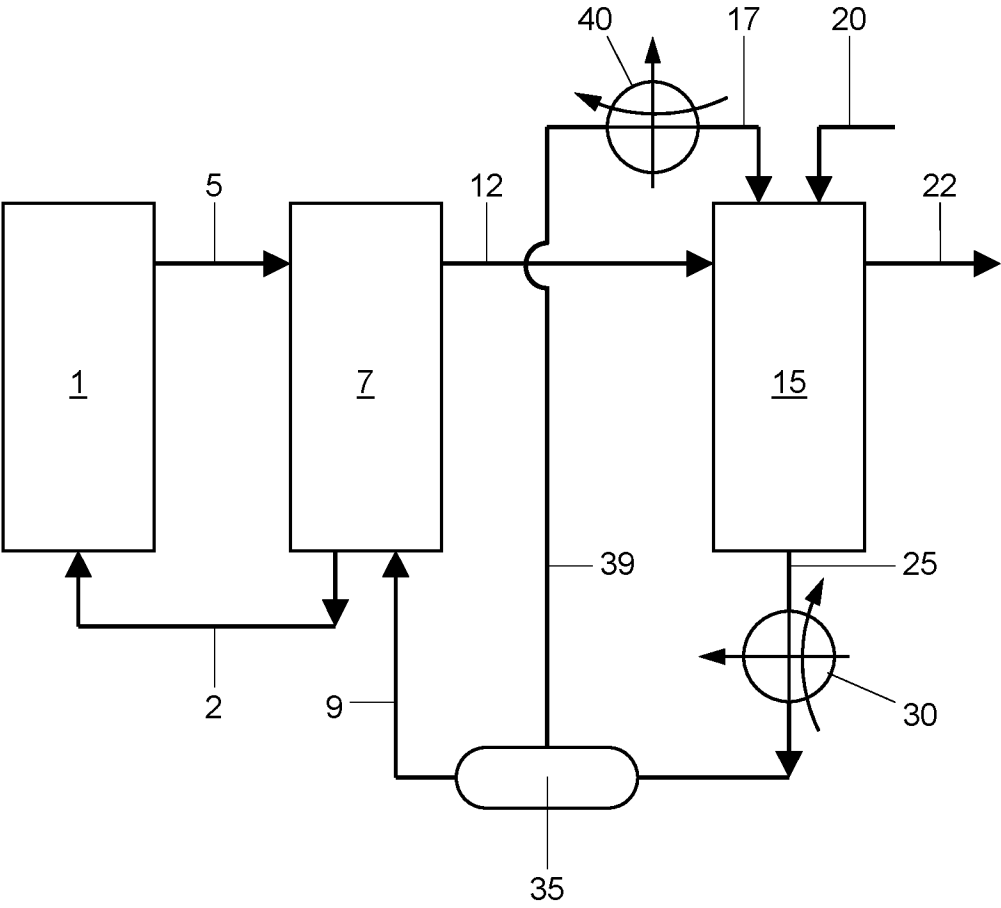


Fig. 1