

LACTAM DEHYDRATION PROCESS

The present invention relates to a process for separating the water from an aqueous lactam
5 solution.

It applies more particularly to a mixture resulting from the reaction between an aminonitrile and water (this reaction also being called cyclizing hydrolysis).

10 During the cyclizing hydrolysis of an aminonitrile in order to form a lactam, one molecule of ammonia per molecule of lactam is also formed. At the end of the reaction, the reaction mixture therefore contains at least the lactam produced, the excess water
15 and ammonia.

It may contain lesser amounts of aminonitrile that has not reacted, or possible by-products of the reaction.

It may also comprise a solvent possibly used
20 in the cyclizing hydrolysis reaction.

The cyclizing hydrolysis may be carried out in vapour phase or in liquid phase. The process of the invention may apply to mixtures resulting from one or other of these methods of preparation.

25 For the description of the various processes for preparing a lactam by cyclizing hydrolysis of an aminonitrile, reference may be made, for example, to Patent EP-A-0,659,741 or to International Patent

Application WO-A-96/22974 or to International Patent Applications WO-A-95/14664 and 95/14665.

The aminonitriles used for preparing lactams are more particularly aminonitriles having from 4 to 12 carbon atoms, preferably linear or branched aliphatic aminonitriles.

As examples of such aminonitriles, mention may be made of those which derive from the hydrogenation of one of the two nitrile functional groups of dinitriles, such as adiponitrile, methylglutaronitrile, ethylsuccinonitrile, dimethylsuccinonitrile, succinonitrile, glutaronitrile and dodecanedinitrile, into a primary amine functional group.

Among the solutions resulting from the cyclizing hydrolysis of an aminonitrile into a lactam, those which correspond to the preparation of caprolactam from 6-aminocapronitrile and water are the most important from an industrial standpoint, since the said caprolactam, upon polymerization, leads to nylon-6.

The process of the invention will therefore relate more particularly to the distillation of the water from aqueous solutions of caprolactam, but this distillation may also be transposed to aqueous solutions of other lactams.

It is necessary beforehand to have separated, generally by distillation, the ammonia formed in the

reaction.

One method of separating the water that may be envisaged consists in distilling the water at reduced pressure (less than or equal to 20 mbar absolute, for example) so as not to exceed 145°C at the bottom of the column.

Such a process involves condensing the water thus distilled, by circulating a fluid (such as water) at a temperature of less than or equal to 15°C. By way of example, this requires the presence of a refrigeration unit having a power of approximately 1.2 megawatts in order to treat the order of 8 tonnes/hour of an aqueous lactam solution having a solutes concentration of approximately 60% by weight.

The subject of the present invention is a process for separating the water (which process may also be called dehydration) from an aqueous lactam solution, which process is more economical both from the standpoint of the investment necessary for its implementation and from the standpoint of the energy consumed.

More specifically, it consists of a process for separating the water from an aqueous lactam solution, characterized in that:

- the water is distilled in a first distillation column by maintaining a temperature at the bottom of the column less than or equal to 160°C and

preferably less than or equal to 145°C, and at an absolute pressure of 50 millibars to 200 millibars, the distillate being condensed at a temperature of 30°C to 60°C using a coolant system:

- 5 - the lactam remaining at the bottom of the column is then subjected to a distillation in a second distillation column by maintaining a temperature at the bottom of the column less than or equal to 160°C and preferably less than or equal to 145°C, and at a
10 pressure of 10 to 45 millibars, so as to separate the water that it still contains;

- the water, containing traces of lactam, distilled in the second column, is recovered in the form of vapour at a temperature greater than or equal
15 to 70°C.

By operating the process according to the invention, it is not necessary to have a refrigeration unit for condensing the water at the top of the first column.

- 20 The water, containing traces of lactam, distilled in the second column is preferably recycled into the first distillation column. This recycling may be carried out after condensing the water at the pressure of the second column, either between 10 and
25 45 millibars absolute, corresponding to a temperature of 5°C to 30°C. However, such a variant only requires a refrigeration unit having a power of approximately 100

to 200 times less than that of the refrigeration unit which would have been necessary in the context of a distillation using a single column.

According to another embodiment of the invention, the water containing traces of lactam, distilled in the second column may be compressed by means of a steam ejector to a pressure allowing it to condense at a temperature greater than 15°C, using a standard coolant such as the atmospheric air or water at room temperature. In this case, the delivery pressure of the steam ejectors must be at least equal to the pressure of the first column, advantageously between 25 millibars absolute and 50 to 200 millibars absolute, the pressure of the first column. In this embodiment, the refrigeration units are omitted.

Overall, the present process is characterized, for the same capacity of distillation of an aqueous lactam solution, by a much lower total energy consumption which can be broken down into an electricity consumption which is 100 to 200 times less, as indicated previously, and an additional energy consumption in the form of steam, in order to heat the second column, which represents only less than approximately 15% of the electrical energy saving made.

The water recovered from the top of the first column is condensed at the pressure of this column, i.e. between 50 millibars and 200 millibars corresponding to a temperature of between 35 and 60°C,

compatible with most coolants generally used, such as atmospheric air or river water or water from cooling towers.

The water recovered from the top of the
5 second column may be partially condensed at approximately 70°C before being completely condensed and then returned to the first column. This arrangement makes it possible to limit the amount of water to be condensed under particular conditions and therefore to
10 reduce the amount of energy necessary for carrying out this operation.

Before this condensed water is recycled into the first column, it may be advantageous to add a certain amount of water in order to reduce the
15 caprolactam concentration and to prevent it from precipitating.

Furthermore, despite the need for an additional column, the investment necessary, for the same production, is also less in the process according
20 to the invention than in a process carried out using a single column, on account of the considerable refrigerating capacity needed in this latter process.

The aqueous lactam solution, preferably caprolactam, from which the ammonia has been removed,
25 has a lactam concentration which varies widely depending on the conditions under which the said lactam is prepared, especially the initial water/aminonitrile molar ratio and whether or not an organic solvent is

present. The lactam concentration may generally vary from 5% to 80% by weight with respect to the total weight of the solution, and preferably from 20% to 75%.

Since the water to be distilled has physical
5 properties that are very different from the lactam from which it must be separated, it is not necessary to use distillation columns having a very large number of theoretical trays.

Columns, particularly packed columns, with at
10 least two theoretical trays are very suitable for implementing the process of the invention. Columns having a number of theoretical trays ranging from 2 to 10 may, for example, be used. The packing used may be of the loose packing or ordered packing type, as
15 proposed by the various manufacturers and sized according to the rules of the art. It is also possible to use columns with trays of the same efficiency, although this is less advantageous from the standpoint of head losses.

20 The process of the invention does not exclude the possibility of separating the water from the aqueous lactam solution using more than two columns.

However, in general this is unnecessary and, furthermore, the economic advantages provided by the
25 process described above would be fewer if the number of distillation columns were to be increased significantly. The examples which follow illustrate the invention.

EXAMPLE 1: Distillation with two columns in series operating at different pressures.

Distillation of an aqueous caprolactam solution having the following composition:

- 5 - water: 40% by weight
- caprolactam: 60% by weight.

The characteristics of the first distillation column are as follows:

- 10 - number of distillation stages (theoretical trays): 4
- reflux ratio: 0.1
- absolute operating pressure at the top of the column: 0.13 bar
- temperature at the top: 50°C
- 15 - temperature at the bottom: 135°C
- heat load of the boiler: 3.9 Gcal/h
- heat load of the condenser: 3.6 Gcal/h.

The characteristics of the second distillation column are as follows:

- 20 - number of distillation stages (theoretical trays): 3
- absolute operating pressure at the top of the column: 0.025 bar
- 25 - top temperature: 130°C
- bottom temperature: 145°C
- heat load of the boiler: 0.08 Gcal/h

- heat load of the 70°C-moderated partial
condenser: 0.02 Gcal/h

- heat load of the condenser for the water to
be recycled into the first column: 0.02 Gcal/h

- 5 - power consumed by the refrigeration unit in
order to condense the water at the top of the second
column: 10 kW, with condensation at 35°C.

The caprolactam, stripped of its water, which
was obtained in this example has the following

10 composition:

- water: 0.010% by weight
- caprolactam: 99.990% by weight.

COMPARATIVE TEST: Conventional distillation using a
15 refrigeration unit at the top of the column

Distillation of a product having the
following composition:

- water: 40% by weight
- caprolactam: 60% by weight.

20 The characteristics of the distillation
column are as follows:

- number of distillation stages: 4
- reflux ratio: 0.1
- absolute operating pressure at the top of
25 the column: 0.015 bar
- temperature at the top: 13°C
- temperature at the bottom: 140°C

- heat load of the boiler: 3.6 Gcal/h
- heat load of the condenser: 3.4 Gcal/h
- power consumed by the refrigeration unit:

1200 kW, with condensation at 35°C.

5 The caprolactam, stripped of its water, which was obtained in the comparative test has the following composition:

- water: 0.004% by weight
- caprolactam: 99.996% by weight.

CLAIMS

1. Process for separating the water from an aqueous lactam solution, characterized in that:

5 - the water is distilled in a first distillation column by maintaining a temperature at the bottom of the column less than or equal to 160°C and at an absolute pressure of 50 millibars to 200 millibars, the distillate being condensed at a temperature of 30°C
10 to 60°C using a coolant;

 - the lactam remaining at the bottom of the column is then subjected to a distillation in a second distillation column by maintaining a temperature at the bottom of the column less than or equal to 160°C and at
15 a pressure of 10 millibars to 45 millibars;

 - the water, containing traces of lactam, distilled in the second column, is recovered in the form of vapour at a temperature greater than or equal to 70°C.

20 2. Process according to Claim 1, characterized in that a temperature less than or equal to 145°C is maintained at the bottom of the first column.

 3. Process according to either of Claims 1
25 and 2, characterized in that a temperature less than or equal to 145°C is maintained at the bottom of the second column.

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4. Process according to one of Claims 1 to 3, characterized in that the lactam is caprolactam.

5. Process according to one of Claims 1 to 4, characterized in that the water containing traces of lactam, distilled in the second column, is recycled into the first column.

6. Process according to Claim 5, characterized in that the water is recycled after condensing it at a temperature of 5°C to 30°C at a pressure of 10 to 45 millibars.

7. Process according to Claim 5, characterized in that the water containing traces of lactams is recycled after compressing the distilled water at a pressure at most equal to the pressure of the first column and condensing at a temperature greater than 15°C.

8. Process according to one of the preceding claims, characterized in that water is added to the water containing traces of lactam, which is distilled in the second column, before it is recycled into the first column.

9. Process according to one of Claims 1 to 8, characterized in that the first distillation column and the second distillation column each have at least two theoretical trays, and preferably from 2 to 10 theoretical trays.

10. Process according to one of Claims 1 to 9, characterized in that the distillation columns are



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packed columns.

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