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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



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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
5 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
10 groups of dinitriles, such as adiponitrile, methylglutaronitrile, ethylsuccinonitrile, dimethylsuccinonitrile, succinonitrile, glutaronitrile and dodecanedinitrile, into a primary amine functional group.

15 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
20 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
25 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, which process comprises:

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

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

- subjecting the lactam remaining at the bottom of the first column to 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 a pressure of 10 to 45 millibars, and

- recovering the water containing traces of lactam, distilled in the second column, in the form of vapour at a temperature greater than or equal 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.

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 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 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 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 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.

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 process described above would be fewer if the number of distillation columns were to be increased significantly.

The present invention also provides a lactam solution which is substantially free of water, whenever obtained by the process described above.

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:

- number of distillation stages (theoretical
- 10 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
20 distillation column are as follows:

- 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

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- 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. A process for separating the water from an aqueous lactam solution, which process comprises:

(a) distilling the water in a first
5 distillation column by maintaining a temperature at the bottom of the column of less than or equal to 160°C and an absolute pressure of 50 millibars to 200 millibars, and condensing the distillate at a temperature of 30°C to 60°C using a coolant;

10 (b) subjecting the lactam remaining at the bottom of the said first column to distillation in a second distillation column by maintaining a temperature at the bottom of the column less than or equal to 160°C and a pressure of 10 millibars to 45 millibars; and

15 (c) recovering the water containing traces of lactam, distilled in the said second column, in the form of vapour at a temperature greater than or equal to 70°C.

2. A process according to claim 1, wherein
20 the temperature maintained at the bottom of the first column is less than or equal to 145°C.

3. A process according to claim 1 or claim

2, wherein the temperature maintained at the bottom of the second column is less than or equal to 145°C.

4. A process according to any one of claims 1 to 3 wherein the lactam is caprolactam.

5 5. A process according to any one of claims 1 to 4 which further comprises recycling, into the first column, the water containing traces of lactam distilled in the second column.

6. A process according to Claim 5 wherein
10 the water is recycled after being condensed at a temperature of 5°C to 30°C and a pressure of 10 to 45 millibars.

7. A process according to claim 5, which
comprises recycling the water containing traces of
15 lactams after compressing the distilled water at a pressure at most equal to the pressure in the first column and condensing at a temperature greater than 15°C.

8. A process according to any one of claims
20 5 to 7 which further comprises adding water to the water containing traces of lactam, which is distilled in the second column, before recycling it into the first column.

9. A process according to any one of claims
1 to 8 wherein the first distillation column and the
second distillation column each has at least two
theoretical trays, and preferably from 2 to 10
5 theoretical trays.

10. A process according to any one of claims
1 to 9 wherein the distillation columns are packed
columns.

11. A lactam solution which is substantially
10 free of water, whenever obtained by a process as
defined in any one of claims 1 to 10.