

DescriptionProcess and apparatus for cryogenic air separation

The invention relates to a process for cryogenic air separation according to the precharacterizing clause of Patent Claim 1.

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Similar processes with residual gas recycling are known from DE 2261234, US 4966002, US 5363657, US 5528906, US 5934106, US 5611218, US 5582034, US 2004244417, DE 19909744 A1, DE 19919933 A1, DE 19954593 A1, US 2007204652 A1, DE 102006027650 A1 and EP 1995537 A2. In this case, and also
10 in US 4966002 and US 5582034, a counter-current subcooler is used, in which the liquid oxygen-containing recycle fraction is "subcooled", i.e. cooled below its boiling point.

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Here, "single column" is understood to mean a distillation column which is operated in a uniform pressure range - which means here that the pressure difference between top and bottom of the column is based exclusively on the pressure loss of the vapour rising in the column - and in which both the feed air is fed in as main feed fraction and also the nitrogen product is produced in the form of part of the nitrogen-rich fraction accumulating in the upper region of the column. Double-column or triple-column
20 processes for nitrogen/oxygen separation are therefore not covered. However, a pure oxygen column, which is connected to the single column and is operated as a pure stripping column, is not ruled out.

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Processes and apparatuses for the cryogenic separation of air are described in general terms in Hausen/Linde, Tieftemperaturtechnik, 2nd Edition 1985, Chapter 4 (pages 281 to 337).

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The invention is based on the object of specifying a process of the type mentioned at the beginning and a corresponding apparatus which are economically particularly beneficial.

This object is achieved by the characterizing features of Patent Claim 1.

Surprisingly, the use of an integrated heat exchanger, which combines the functions of a main heat exchanger and a counter-current subcooler, permits any pre-liquefaction of the air to be avoided. As a result, all of the air in the column is able to rise and participate in the rectification, the separation effect becomes higher and, overall, the process according to the invention is therefore particularly beneficial. The precise layout of the integrated heat exchanger depends on the boundary conditions of the individual case and must be defined for each plant by using the usual calculation tools of the process engineer.

Besides this, the integration according to the invention simplifies the design considerably with respect to the pipework. Since the counter-current subcooler is given a substantially larger cross section as a result of the integration in the main heat exchanger, the liquid streams carried in counter-current in relation to the gas streams are offered an optimum heating surface area. It is merely necessary for a heat exchanger to be supported and piped in the coldbox. The absolute number of headers of the two heat exchangers decreases. The gas streams (residual gas to the turbine, product nitrogen, residual gas from the turbine) from the top of the coldbox do not have to be led via two fixed points (counter-current subcooler and main heat exchanger). Expansion loops can be dispensed with; the integrated solution permits a pipe run with minimized pipe stresses.

The integration of main heat exchanger and counter-current subcooler is certainly known from air separation processes having two or more columns for nitrogen/oxygen separation. However, this measure has not previously been applied to processes of the type mentioned at the beginning, since the manufacturing outlay for a particularly long integrated heat exchanger did not appear to be justified in single column processes. The surprising effect of the avoidance of pre-liquefaction of the air was previously unknown.

In principle, any heat exchanger type can be used as an integrated heat exchanger in the process according to the invention, for example a helically coiled heat exchanger or else a straight pipe exchanger. However, the use of a plate-type heat exchanger, in particular a brazed aluminium plate heat exchanger, is particularly beneficial. In this case, the integrated heat exchanger is formed by a single plate-type heat exchanger block.

It is particularly cost-effective if the single column constitutes the only distillation column of the distillation column system.

- 5 In order to generate refrigerating capacity, a further oxygen-containing fraction can be expanded, producing work, as explained in detail in Patent Claim 4. The integrated heat exchanger is also used for the subcooling of the further oxygen-containing fraction, in that the liquid further oxygen-containing fraction is cooled down in the counter-current subcooler before its evaporation. The integration according to the
10 invention makes it possible to introduce the liquid further oxygen-containing fraction into the heat exchanger above the temperature of the air removal. The temperature difference is, for example, 0.2 to 5 K. This contributes to the avoidance of the pre-liquefaction.
- 15 In addition, before its work-producing expansion, the evaporated further oxygen-containing fraction is warmed up in counter-current to air in the integrated heat exchanger.

20 The further oxygen-containing fraction can, for example, have the same composition as the recycle fraction. In this case, the two fractions can be led in common lines and passages until after the top condenser.

Alternatively, the oxygen-containing recycle fraction is removed from the single column at an intermediate point which is located at least one theoretical or practical plate
25 above the point at which the further oxygen-containing fraction is removed. In this case, separate lines and separate passages must be provided for the two fractions in the top condenser and possibly in the counter-current subcooler.

Advantageously, the expansion machine (21) is coupled mechanically to the re-
30 compressor (31). As a result, the mechanical energy obtained during the work-producing expansion is used for re-compression. This is preferably the only energy source for the drive of the re-compressor.

It is beneficial if the re-compressor (30) is constructed as a cold compressor. Here, a
35 "cold compressor" is understood to mean an apparatus in which the gas to be

compressed is fed in at a temperature which lies considerably below the ambient temperature, in general below 250 K, preferably below 200 K.

It is also beneficial if, in the process according to the invention, the re-compressed
 5 recycle fraction is cooled in the integrated heat exchanger before being introduced into the lower region of the single column, the re-compressed recycle fraction being drawn off from the integrated heat exchanger in completely gaseous form and led into the single column in completely gaseous form. The recycle fraction is therefore also free of pre-liquefaction and participates in the rectification in the single column completely as
 10 rising vapour. Therefore, the pre-liquefaction is avoided completely in both the feed streams for the single column, namely in the feed air and in the recycle fraction.

The invention additionally relates to an apparatus according to Patent Claim 10.

15 The invention and further details of the invention will be explained in more detail below by using an exemplary embodiment illustrated schematically in the drawing.

Atmospheric air 1 is taken in by an air compressor 3 via a filter 2 and there is compressed to an absolute pressure of 6 to 20 bar, preferably about 9 bar. After
 20 flowing through a re-cooler 4 and a water separator 5, the compressed air 6 is cleaned in a cleaning device 7. The cleaning device 7 has a pair of containers which are filled with adsorption material, preferably a molecular sieve. The cleaned air 8 is cooled down to somewhat above the dew point in a main heat exchanger 9 and finally led into a single column 12 as a completely gaseous feed air stream 11.

25 The operating pressure of the single column 12 (at the top) is 6 to 20 bar, preferably about 9 bar. Its top condenser is cooled with an oxygen-containing recycle fraction 18a, 18b and a further oxygen-containing fraction 14a, 14b. The further oxygen-containing fraction 14a is drawn off from the bottom of the single column 12, the recycle fraction
 30 18a from an intermediate point some practical or theoretical plates further above. Before they are fed 14b, 18b into the top condenser 13, both fractions 14a, 18a are cooled down in a counter-current subcooler 100, main heat exchanger 9 and counter-current subcooler 100 according to the invention being formed by an integrated heat exchanger 101, which is implemented here as a single plate-type heat exchanger
 35 block. The height difference between the exit of the stream 14a from the single column

12 (more precisely the liquid level at the bottom of the column) and the entry into the integrated heat exchanger 101 should in principle be chosen such that the proportion of gas as a result of the expansion lies below 5% by volume. If, in a departure from this, the proportion of gas is higher than 5% by volume, a perforated plate must be fitted in the header over the entire region above the entry into the passages, its pressure loss being chosen such that the gas bubbles are distributed over all the passages. The two-phase mixture is then led into the integrated heat exchanger, firstly transversely with respect to the other streams (possibly with one or more deflections), in which the gas proportion is condensed completely, that is to say the adjacent passages are correspondingly colder in every operating case.

As the main product from the single column 12, gaseous nitrogen 15, 16a is drawn off at the top and, as first recirculation stream, is led through a group of passages 102 which goes through from the cold to the warm end of the integrated heat exchanger. In the process, the return stream in the region of the counter-current subcooler 100 comes into indirect heat exchange with the two oxygen-containing fractions 14a, 18a and then, in the region of the main heat exchanger 9, into indirect heat exchange with the feed air stream 8. Via a line 17, it is finally drawn off at approximately ambient temperature as a gaseous pressurized product (PGAN).

The remainder 16b of the gaseous nitrogen 15 is condensed completely or substantially completely in the top condenser 13. Part 53 of the condensate 52 from the top condenser 13 can be obtained as liquid nitrogen product (PLIN); the remainder 54 is put into the top of the single column as reflux. Non-condensed constituents can be drawn off via a purge line 90.

The recycle fraction 18b is evaporated in the top condenser 13 under a pressure of 2 to 9 bar, preferably about 4 bar, and flows in gaseous form via line 29 to a cold compressor 30, in which it is re-compressed approximately to a pressure which is sufficient to feed it back into the single column. The re-compressed recycle fraction 31 is cooled down to column temperature again in the counter-current subcooler 100 and fed to the single column 12 again at the bottom in completely gaseous form via line 32.

The further oxygen-containing fraction 14b is evaporated in the top condenser 13 under a pressure of 2 to 9 bar, preferably about 4 bar, and flows in gaseous form via line 19

to the cold end of the integrated heat exchanger 101. There, in the region of the counter-current subcooler 100, it comes into indirect heat exchange with the two liquid oxygen-containing fractions 14a, 18a and then, in the region of the main heat exchanger 9, into indirect heat exchange with the feed air stream 8. It is removed from
5 the main heat exchanger 9 again (line 20) at an intermediate temperature and is expanded to about 300 mbar above atmospheric pressure, producing work, in an expansion machine 21 which, in the example, is constructed as a turbo-expander . The expansion machine is coupled mechanically to the cold compressor 30 and a braking device 22 which, in the exemplary embodiment, is formed by an oil-filled brake. The
10 expanded further fraction 23 is warmed up to about ambient temperature in the integrated heat exchanger 101. The warm further fraction 24 is blown off into the atmosphere (line 25) and/or used in the cleaning device 7 as regeneration gas 26, 27, possibly following heating in the heating device 28.

15 In the exemplary embodiment, the top condenser 13 is constructed as a forced-flow evaporator. Alternatively, a bath evaporator or falling film evaporator can be used.

Patent claims

1. Process for cryogenic air separation in a distillation column system which has at least one single column (12), wherein

- 5 – a compressed feed air stream (6, 8) is cooled down in a main heat exchanger (9) in counter-current to a first return stream (16, 23) from the distillation column system,
- the cooled feed air stream (11) is led into the single column (12),
- a nitrogen-rich fraction (15) is produced in the upper region of the single column
- 10 (12),
- at least part (16b) of the nitrogen-rich fraction (15) is condensed in a top condenser (13), which is constructed as a condenser-evaporator,
- at least part (54) of the liquid nitrogen-rich fraction (52) produced in the top condenser (13) is led into the single column (12) as reflux,
- 15 – an oxygen-containing recycle fraction (18a) is drawn off from the single column (12) in liquid form,
- the liquid recycle fraction (18a) is cooled down in a counter-current subcooler (100),
- the cooled recycle fraction (18b) is evaporated in the top condenser (13),
- 20 – the evaporated recycle fraction (29) is re-compressed in a re-compressor (30),
- the re-compressed recycle fraction (31, 32) is fed to the lower region of the single column (12),

characterized in that

- 25 – the main heat exchanger (9) and the counter-current subcooler (100) are formed by an integrated heat exchanger (101),
- the integrated heat exchanger (101) having a first group of passages for the first return stream (16, 23), which goes through from the cold to the warm end of the integrated heat exchanger,
- the first return stream (16, 23) being led into this group of passages (102) at the
- 30 cold end and flowing through the integrated heat exchanger (101) as far as its warm end and, in the process,
- being brought into indirect heat exchange both with the liquid recycle fraction (18a) and with the feed air stream (8),
- the cooled feed air stream (11) being drawn off from the integrated heat
- 35 exchanger (101) in completely gaseous form and being led into the single

column (12) in completely gaseous form.

2. Process according to Claim 1, characterized in that the integrated heat exchanger is formed by a single plate-type heat exchanger block.

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3. Process according to Claim 1 or 2, characterized in that the single column constitutes the only distillation column of the distillation column system.

4. Process according to one of Claims 1 to 3, characterized in that

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- a further oxygen-containing fraction (14a) is drawn off from the single column (12) in liquid form,
- the liquid further oxygen-containing fraction (14a) is cooled down in the integrated heat exchanger (101),
- the cooled further oxygen-containing fraction is evaporated in the top condenser (13),
- the evaporated further oxygen-containing fraction (19) is warmed in the integrated heat exchanger in counter-current to air, and
- the warmed further oxygen-containing fraction is expanded in an expansion machine (21), producing work,
- the temperature of the liquid further oxygen-containing fraction (14a) as it is introduced into the integrated heat exchanger (101) being higher than the temperature of the cooled feed air stream (11) as it is drawn off from the integrated heat exchanger (101).

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5. Process according to Claim 4, characterized in that, before its work-producing expansion, the evaporated further oxygen-containing fraction is warmed up in counter-current to air in the integrated heat exchanger.

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6. Process according to either of Claims 4 and 5, characterized in that the oxygen-containing recycle fraction is removed from the single column at an intermediate point which is located at least one theoretical or practical plate above the point at which the further oxygen-containing fraction is removed.

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7. Process according to one of Claims 4 to 6, characterized in that the expansion machine (21) is coupled mechanically to the re-compressor (30).

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8. Process according to one of Claims 1 to 7, characterized in that the re-compressor (30) is constructed as a cold compressor.

9. Process according to one of Claims 1 to 8, characterized in that the re-compressed recycle fraction (31) is cooled in the integrated heat exchanger (101) before being introduced into the lower region of the single column (12), the re-compressed recycle fraction (32) being drawn off from the integrated heat exchanger (101) in completely gaseous form and led into the single column (12) in completely gaseous form.

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10. Apparatus for cryogenic air separation in a distillation column system which has at least one single column (12),

- having a main heat exchanger (9) for cooling a compressed feed air stream (6, 8) in counter-current to a first return stream (16, 23) from the distillation column system,
- having means for leading the cooled feed air stream (11) into the single column (12),
- having means for producing a nitrogen-rich fraction (15) in the upper region of the single column (12),
- having a top condenser (13) for condensing at least part of the nitrogen-rich fraction (15, 16b), the top condenser being constructed as a condenser-evaporator,
- having means for leading the liquid nitrogen-rich fraction (52) produced in the top condenser (13) into the single column (12) as reflux,
- having means for drawing off an oxygen-containing recycle fraction (18a) from the single column (12) in the liquid state,
- having a counter-current subcooler (100) for cooling down the liquid recycle fraction (18a),
- having means for introducing the cooled recycle fraction (18b) into the top condenser (13),
- having a re-compressor (30) for compressing the evaporated recycle fraction (29) from the top condenser (13), the re-compressor (30) being constructed in particular as a cold compressor, and
- having means for leading the re-compressed recycle fraction (31, 32) into the

lower region of the single column (12),

characterized in that

- the main heat exchanger (9) and the counter-current subcooler (100) are formed by an integrated heat exchanger (101),
- 5 – the integrated heat exchanger (101) having a first group of passages (102) for the first return stream (16, 23), which goes through from the cold to the warm end of the integrated heat exchanger,
- the cold end of the integrated heat exchanger (101) being connected to means for leading the first return stream (16, 23) into this group of passages
- 10 – the warm end of the integrated heat exchanger (101) being connected to means for drawing the first return stream (16, 23) off from this group of passages,
- the integrated heat exchanger (101) being constructed in such a way that, during the operation of the apparatus, the first return stream (16, 23) is
- 15 brought into indirect heat exchange both with the liquid recycle fraction (18a) and with the feed air stream (8), and
- the passages in the integrated heat exchanger (101) being laid out in such a way that, during the operation of the plant, the cooled feed air stream (11) is drawn off from the integrated heat exchanger (101) in completely gaseous
- 20 form and is led into the single column (12) in completely gaseous form.