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(54) **PROCESS FOR PRODUCING LIQUID CARBON DIOXIDE FROM COMBUSTION GAS AT NORMAL PRESSURE**

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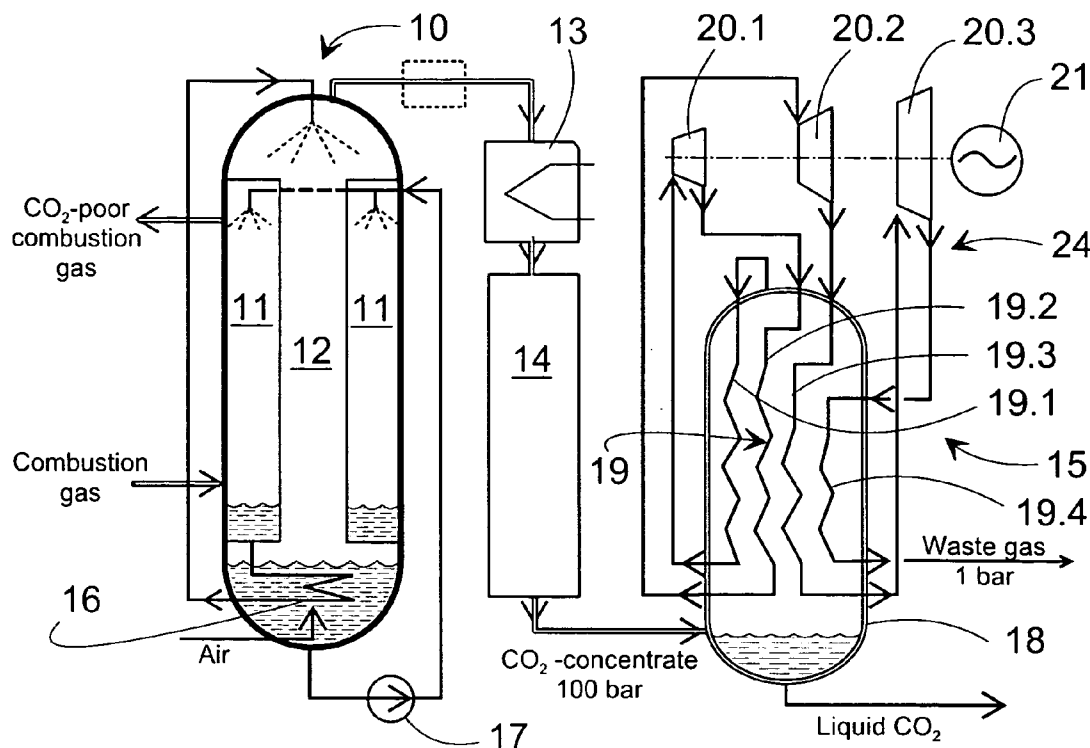
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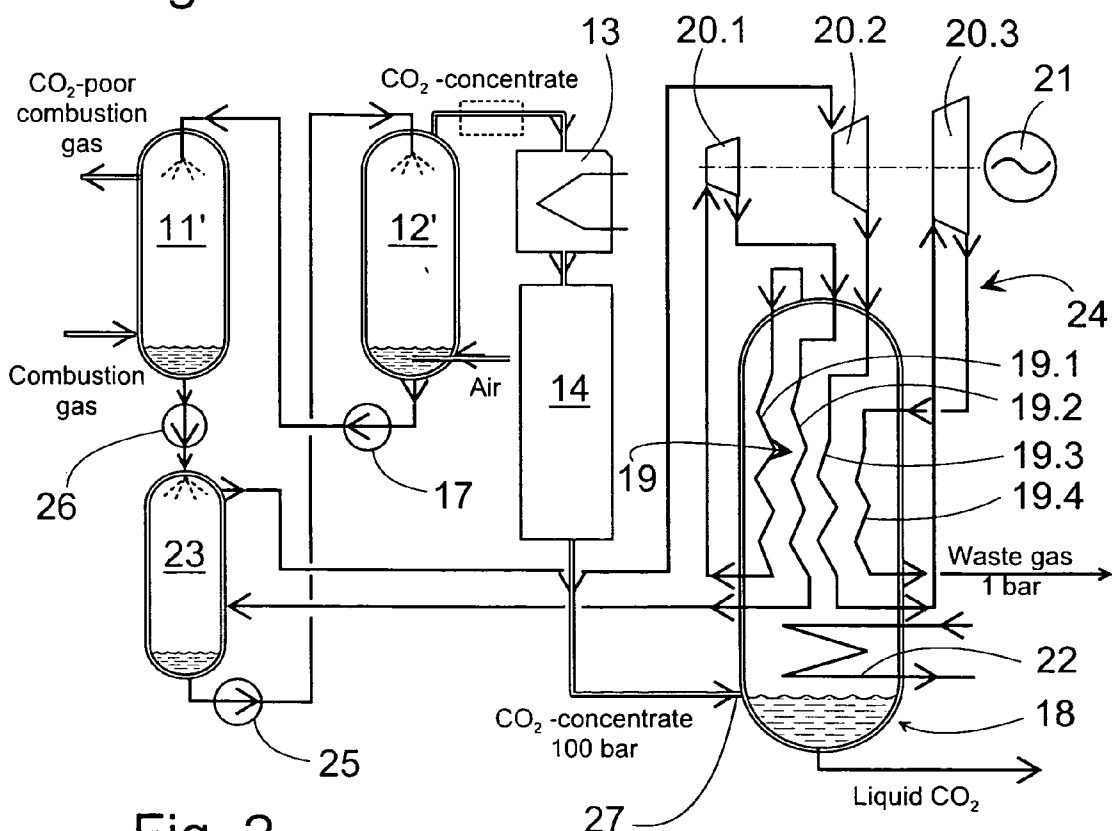
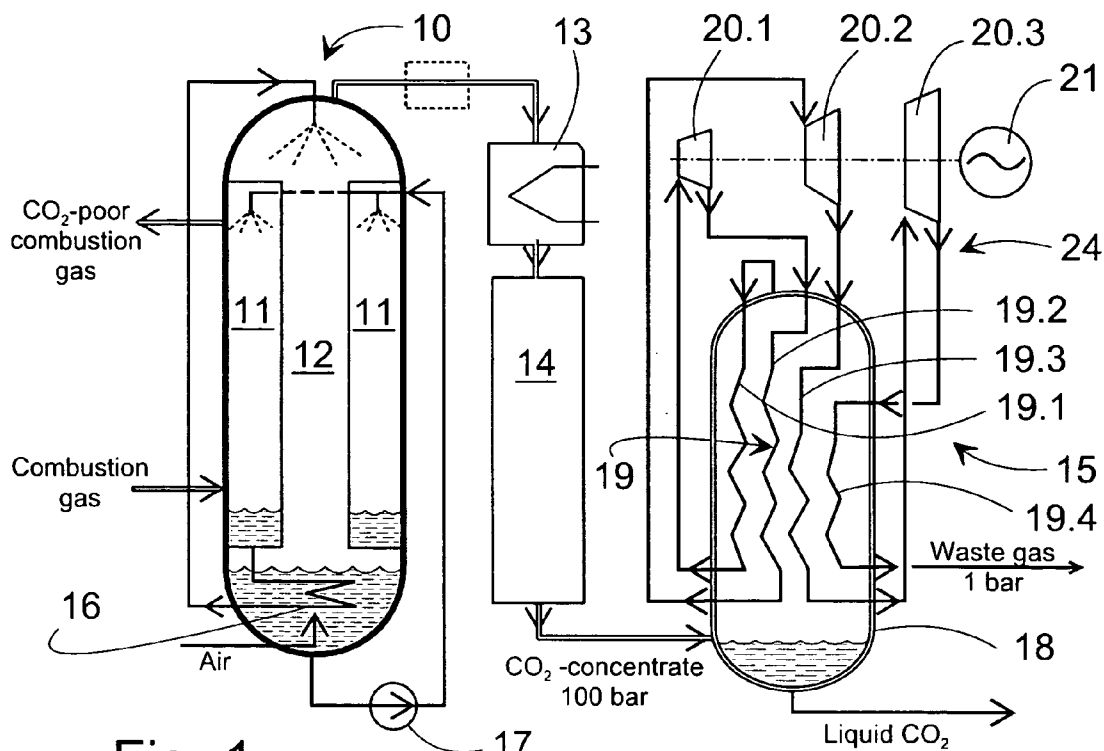
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(57) **ABSTRACT**

The invention relates to a process for producing liquid carbon dioxide (CO₂) from combustion gas at normal pressure, in which process the CO₂ of combustion gas led to the lower part of an absorption column is absorbed in an absorption solution that flows downwards from its upper part, the CO₂-poor combustion gas is removed from the upper part of the absorption column, the said absorption solution is led to the upper part of a desorption column, the CO₂ is desorbed from the absorption solution led to the upper part of the desorption column to form a gaseous CO₂ concentrate, which is removed from the upper part as the absorption solution flows to the lower part of the column, from which it is re-circulated to the upper part of the absorption column, the CO₂ concentrate obtained is brought to a high pressure, and most of the concentrate's CO₂ is liquidized. In the process, the CO₂ is transferred from the normal-pressure combustion gas using a nearly reversible absorption and desorption process, to the CO₂ concentrate at a lower pressure at a temperature near to that of the combustion gas being processed, preferably in the temperature range 35-75° C., in such a way that the absorption and desorption processes take place in columns at more or less the same temperature, preferably without importing external heat to the said processes.





PROCESS FOR PRODUCING LIQUID CARBON DIOXIDE FROM COMBUSTION GAS AT NORMAL PRESSURE

[0001] The present invention relates to a process for producing liquid carbon dioxide (CO₂) from combustion gas at normal pressure, in which process:

[0002] the CO₂ of combustion gas led to the lower part of an absorption column is absorbed in an absorption solution that flows downwards from its upper part,

[0003] the CO₂-poor combustion gas is removed from the upper part of the absorption column,

[0004] the said absorption solution is led to the upper part of a desorption column,

[0005] the CO₂ is desorbed from the absorption solution led to the upper part of the desorption column to form a gaseous CO₂ concentrate, which is removed from the upper part as the said absorption solution flows to the lower part of the column, from which it is re-circulated to the upper part of the said absorption column,

[0006] the CO₂ concentrate obtained is brought to a high pressure, and

[0007] most of the concentrate's CO₂ is liquidized.

[0008] It is wished to limit carbon dioxide emissions globally, due to their effect on the climate. Large amounts of carbon dioxide are released, for example, when fossil fuels are burned. Carbon dioxide recovery from combustion gases has been technically difficult and economically unprofitable, due to the large amount of energy that it consumes.

[0009] One known technique of separating CO₂ from combustion gases is represented by the MEA method, which is disclosed in the publications DE-549556 and DE-606132, among others. In these techniques, the CO₂ is absorbed from the combustion gas into a water solution of monoethylamine (MEA) or other amines. After absorption, the solution is heated to, for example, 120° C. and the CO₂ dissolved in it is desorbed at a pressure of about 2 bar.

[0010] In this process, the desorption heat of the CO₂, which, when using MEA, is 1,6 MJ/kg CO₂, must be brought to the process at 120° C. A significant drawback of this method is the large amount of energy used in principle to transfer the CO₂ from its original partial pressure of about 0,15 bar in the combustion gas to its desorption pressure of about 2 bar.

[0011] Various separation methods are further disclosed, in, for example, publications U.S. Pat. No. 4,797,141, WO-0048709, and JP-59073415. However, these are all strongly characterized by desorption from a solution taking place using external energy, making the methods energetically extremely uneconomical. Publication U.S. Pat. No. 6,228,145 discloses a separation method based on membranes, while publication JP-1230416 discloses a method, in which, as a batch process, CO₂ is first of all bound by adsorption to a solid and then released in a vacuum.

[0012] Patent application PCT/FI01/00629 discloses a thermodynamically advantageous method for separating CO₂ from combustion gas. This method exploits the fact that

the solubility of CO₂ in, for example, methanol close to its condensation point deviates strongly from Henry's Law. The method works best when the partial pressure of the CO₂ is several bars and, for precisely this reason, is particularly suitable for gas-turbine cycles and other processes in which the pressurization of the combustion gas can be used to produce energy.

[0013] Publication DE-843 545 discloses a process where blast-furnace gas with a CO₂ content of 24% boosted to a pressure of 2,5 bar and scrubbed with ethanol at a temperature of -74--67° C. Next, the CO₂ solution obtained is evaporated in two stages at pressures of 0,2 and 0,04 bar, in the same temperature range.

[0014] The CO₂ obtained at the pressure of 0,2 bar is led to the booster through a regenerator, but the fraction obtained at 0,04 bar is boosted as such, its pressure being too low for a normal regenerator process. The deficit in cooling effect that arises and the rest of the cooling power requirement is covered by expanding the gas fraction, exhausted from the scrubbing process, in a cooling turbine.

[0015] The separation process in the process disclosed in this publication is carried out practically reversibly, without the heating of the CO₂ solution and the subsequent loss of energy. However, the very low process temperature, the low pressure of the CO₂ fraction obtained at 0,04 bar, the evaporation of the ethanol used as a solvent, and its high viscosity under the process conditions, make the entire process uneconomical. As it also demands a very CO₂-rich process gas, the process described has not become generally used in combustion gas cleaning.

[0016] The present invention is intended to create a process, in which CO₂ can be liquified from normal-pressure combustion gas and by means of which the aforementioned defects can be eliminated. The characteristic features of the process according to the invention are stated in claim 1.

[0017] The deviation of the absorption/desorption process from reversibility can be reduced advantageously by using air scavenging in the desorption stage, allowing the air accumulated in the CO₂ concentrate to be used in the liquidization of the CO₂ by exploiting the pressure energy of the remaining waste gas in the liquidization.

[0018] Cooling is not required in the absorption/desorption process according to the invention, because the processes are carried out chemically in a temperature range advantageous to the process, instead of solution and evaporation taking place in cryogenic temperature conditions. Consequently, the viscosity of the absorption solution is less than one tenth of the viscosity of the solution used in the process of the aforementioned DE publication. The low viscosity of the absorption solution makes thermal and mass transfer more efficient and the circulation of the solution requires less energy.

[0019] By means of the process according to the invention, it is possible to avoid the boosting of the CO₂ fractions at a very low pressure, by using air scavenging or other methods described later. Unlike in the state of the art, these methods permit the economical separation and liquidization of CO₂, for example, from natural gas combustion gas, which has a low CO₂ content.

[0020] The process according to the invention does not require chemicals that are expensive, volatile, or liable to

oxidation. Other advantages achieved through the process according to the invention are itemized in the description section.

[0021] In the following, the invention is examined with reference to the accompanying drawings, which show some embodiments of the process according to the invention.

[0022] FIG. 1 shows a schematic diagram of one process according to the invention and

[0023] FIG. 2 shows a schematic diagram of another embodiment of the process.

[0024] FIG. 1 shows one basic solution of the process according to the invention, in which air scavenging is used advantageously in the desorption stage. The separation unit 10 shown in the left-hand half of this figure comprises an absorption column 11 and a desorption column 12, which are thermally connected to each other to arrange thermal transfer, in the same structure.

[0025] The absorption and desorption processes are carried out chemically in an advantageous temperature range of 35-75° C., which corresponds to the temperature of the combustion gas being brought to the process. The temperature of the desorption column 12 is 2-5° C. lower than that of the absorption column 11, in order to transfer the heat released in absorption to the desorption, with both columns 11, 12 operating in nearly isothermic conditions, preferably without importing external heat. The thermal energy required in desorption, for example, when using potassium carbonate is about 790 kJ/kg CO₂.

[0026] Combustion gas at normal pressure is brought to the lower part of the absorption column 11, from where it flows upwards against an absorption solution, which absorbs the CO₂ in the combustion gas, and which is brought to the upper part of the column 11 and flows along the column structures (not shown). A suitable absorption solution is a 2-N water solution of, for example, potassium or sodium carbonate. The relevant compounds are non-volatile and non-oxidizing. In the temperature range referred to, the said solutions have a low viscosity (1,0-0,55 cP), so that thermal and mass transfer take place efficiently and the circulation of the solution requires only a little energy.

[0027] The CO₂-rich absorption solution is led from the lower part of the absorption column 11 to the lower part of the desorption column 12, in which, beneath the surface of the absorption solution, a heat exchanger 16 is arranged, in which the absorption solution that has been brought from the lower part of the column 11 transfers heat to the solution in the bottom of the column 12 and desorbs CO₂ from it.

[0028] From the heat exchanger 16, the absorption solution is led to the upper part of the desorption column 12, from which it flows downwards along structures (not shown) arranged in the column 12. In the desorption column 12, a pressure of about 0,2 bar prevails, and, according to one preferred embodiment of the invention, scavenging air is led to the lower part of it and flows through the absorption solution in the bottom of the column 12 and rises against the downwards flowing absorption solution in the column. The air flow is used to maintain a constant pressure in the column 12, the partial pressure of the CO₂ being, at each height in the column 12, more or less in equilibrium with the composition of the absorption solution at the same height. The

CO₂ released from the solution evaporates into the scavenging air. From the upper part of the column 12, CO₂ concentrate exhausts from the upper part of the column 12 at a lower pressure than normal, and has a CO₂ concentration of 50-55%, and is led through a steam condenser 13 to be pressurized in a booster-intercooler 14 and from there on to the liquidization process 15.

[0029] The CO₂-poor absorption solution that accumulates at the bottom of the column 12 is transferred by a pump 17 back to the absorption column 11, to bind CO₂.

[0030] In the liquidization process, one preferred embodiment of which is shown in the right-hand half of FIG. 1, the CO₂ concentrate, which has been pressurized, for example, to 100 bar, in the booster-intercooler 14, is taken to the lower part of a liquidization column 18. In the liquidization column 18, the CO₂ concentrate flows upwards while the CO₂ liquidizes on the surface of heat exchangers 19.1, 19.2, 19.3, 19.4 arranged in series in the column 18. The liquidized CO₂ is led out of the bottom of the column 18, to be transported farther away, or for other use.

[0031] The unliquidized waste gas is led from the top of the column 18, through a cooling pipe or group of pipes 19.1 acting as a heat exchanger, to a first cooling turbine 20.1, in which it loses part of its pressure energy. From the cooling turbine 20.1, the cycle continues in a similar manner to a cooling pipe 19.2. The cooling-expansion cycle is repeated in the turbines 20.2, 20.3 and in the heat exchangers 19.3, 19.4, until the pressure of the waste gas has dropped to 1 bar, when it is removed from the cooling-expansion cycle 24 from the lower part of the last heat exchanger 19.4. The heat exchangers 19.1, 19.2, 19.3, 19.4 are arranged in the liquidization column 18 in such a way that their temperature levels are essentially set to equal each other and their cooling effect is concentrated in the lower part of the column 18. The cooling turbines 20.1, 20.2, 20.3 are connected to a generator 21, or to some other device, in order to exploit the mechanical energy they produce.

[0032] If the CO₂ is liquidized at, for example, a pressure of 100 bar, in a temperature range of +15--40° C., the unliquidized waste gas will then contain a residual CO₂ pressure of about 11 bar, taking the so-called 'Press-effect' (Z. phys. Chemie 110, 768-(1924)) into account, while about 259 kJ of cooling effect per kg of CO₂ is bound during the liquidization. As the heat flux released during liquidization is concentrated at the lower end of the column 18, it is preferable to use a lower pressure ratio in some of the cooling turbines and concentrate the cooling effect produced by them at the lower end of the column 18.

[0033] In this basic solution according to the invention, about 88% of the CO₂ in the concentrate can be liquidized. Under these conditions, the expansion and cooling process 24 described above produces about 360 kJ of cooling effect for each Nm³ of expanded gas, i.e. 230-260 kJ per kilogram of liquidized CO₂. By expanding the enrichment-stage scavenging air in a separate turbine (not shown) before feeding it to the desorption column 12, an additional cooling effect of about 45 kJ/kg CO₂, i.e. a total of 280-310 kJ/kg CO₂, can be produced.

[0034] In both the absorption-desorption cycle 10 and in the liquidization 15, it is possible to use a double column, the construction of which is disclosed in the patent application

FI-20011969, 'Process operating at normal pressure for producing oxygen of oxygen-enriched air', in Claim 11 and in the FIGS. 4a and 4b.

[0035] Because the combustion gas being processed is often warm (60-90° C.), its heat can be exploited by carrying out the absorption and desorption at such a high temperature that the pressure of the water vapour of the combustion gas will have a substantial effect on the process. If, in this second preferred embodiment of the process according to the invention, the temperature of the desorption column 12 is, for example 62,5° C., then the water evaporating from the absorption solution will then maintain a partial pressure of 0,22 bar of the water vapour in it.

[0036] If there is 0,1 bar CO₂ and the same amount of scavenging air in the CO₂ concentrate exhausting from the desorption column 12, then the total pressure of the CO₂ concentrate at 62,5° C. will be 0,42 bar, taking into account the partial pressure of the water vapour. If there is 0,02 bar of water vapour in the CO₂ concentrate after the condenser 13, then its total pressure will still be 0,42 bar, of which 0,2 bar is CO₂ and another 0,2 bar is air. In other words, the partial pressures of the CO₂ and the air double, thus reducing the size and energy requirements of the boosters needed to pressurize the CO₂. Energy for this is obtained from 'sliding' condensation, in the condenser 13, of the water vapour evaporating from the absorption solution in the desorption column, or that produced at a lower than normal pressure. The condensation water is removed from the condenser 13 (not shown) before the CO₂ is pressurized.

[0037] In many cases, especially when the condensation temperature of the combustion gas being processed is higher than the absorption temperature, the thermal energy required to evaporate the water in the desorption column 12 can be extracted from the combustion gas in the absorption column 11. Bled steam (not shown) at 0,5 bar, for example, can also be introduced to the desorption column 12.

[0038] In a third embodiment according to the invention, shown in FIG. 2, instead of the double column shown in FIG. 1 there are two separate columns 11' and 12'. In this case, the absorption heat is stored in the absorption solution in the column 11' and exits from it in the desorption that takes place in the column 12'. If one kilomole (=44 kg) of CO₂ per tonne is absorbed in the solution, its temperature will rise about 9° C. in absorption while, at a specific CO₂ partial pressure, the CO₂ content will diminish by about 21% when using potassium carbonate. In order to reduce this effect, it is possible to use a weak absorption solution and a high solution flow.

[0039] In this embodiment, a from the cooling cycle 24 separate water-cooled pre-condenser 22, which is close to the inlet connection 27 of the CO₂ concentrate, is located in the lower part of the liquidization column 18. In countries with a cool climate, cold ($\leq 6^{\circ}$ C.) cooling water is available from the sea or deep lakes, and in winter from other waterways too. If a temperature of 10° C. is maintained in the pre-condenser 22, and the partial pressure in the CO₂ concentrate is 60 bar (the concentrate having, for example, a pressure of 110 bar and a 55% CO₂ content), then about 25% of the CO₂ will liquidize in the pre-condenser 22, the cooling effect required being correspondingly reduced.

[0040] In the embodiment shown in FIG. 2, an additional absorption column 23, in which the CO₂-rich solution com-

ing from the lower part of the first absorption column 11' absorbs additional CO₂ from the waste gas coming from the cooling cycle 24 of the liquidization, is located at a suitable pressure in the cooling cycle 24 of the liquidization. This increases the CO₂ partial pressure of the concentrate obtained from desorption, while simultaneously returning a considerable part of the waste-gas CO₂ to the liquidization process 15. The impoverished waste gas is returned to the cooling cycle 24 of the liquidization process 15 from the upper part of the column 23.

[0041] Part of the waste gas of the liquidization can also be led at a suitable pressure to an ejector in connection with the evaporation column 12', in order to raise the pressure of the CO₂ concentrate and to reduce the size of the boosters 14 required.

[0042] In a fourth embodiment of the invention, very little or no scavenging air is used, so that the concentrate created in the desorption column 12 is nearly pure CO₂ and most of it can be liquidized at a pressure of 100 bar by water cooling.

[0043] If the CO₂ content of the absorption solution corresponds to a partial pressure of 0,15 bar, and if the CO₂ partial pressure in the evaporation column is 0,05 bar, then the separation efficiency without air scavenging would be 67%. At such a low pressure, it is advantageous to exploit the pressure of the water vapour as described in the second embodiment. Even at an evaporation temperature of +47° C. the vapour pressure of the water is 0,11 bar, so that the pressure of the CO₂ concentrate coming to the booster 14 is 0,16 bar, instead of 0,05 bar.

[0044] By means of the booster 14, CO₂ pressurized to 100 bar is led to the liquidization column 18, in which it is liquidized in a water-cooled liquidizer (not shown) filling the entire column 18. If there are other gases as impurities in the concentrate, then, when the temperature of the liquidizer (not shown) is 15° C., there will be 50% CO₂ in the gas exiting from the top of the column 18. If this CO₂ loss is detrimentally large, then the CO₂ is recovered by means of the liquidizing column described in the embodiment shown in FIG. 1, the cooling power required by which is produced by expanding the waste gas exhausting from the column 18 in the cooling turbines 20.1, 20.2, 20.3, 20.4.

[0045] The conditions of the process, such as the pressurizing of the CO₂ and the content of the CO₂ concentrate, can be preferably selected so that the cooling power requirement of the liquidization process can be precisely covered using the pressure energy of the unliquidized waste gas of the process and other process-related sources of cooling power. The energy consumption of the process per kilogram of liquidized CO₂ is then brought close to its minimum value.

[0046] One advantageous application of the liquidization process according to the invention is its use in connection with oxygen combustion. This embodiment lacks the absorption and desorption cycles described, because in oxygen or oxygen concentrate combustion the CO₂ can be liquidized from the combustion gas without enrichment. One example of this is the liquidization of CO₂ from the pressurized combustion gas of a Värtan-type PFBC power plant.

[0047] In that case, most of the combustion gas flows through a gas turbine, waste-heat boiler, and water vapour condenser back to a booster, in which oxygen concentrate is

mixed with it and from which the gas mixture returns to the boiler. The side flow at a lower temperature is cooled and boosted to liquidization pressure, after which the CO₂ is liquidized as described in the previous examples.

[0048] If the CO₂ content of the combustion gas is <50%, it is preferable to extend the liquidization to close to the triple point of CO₂ (−56,5° C., 5,1 bar), so that its loss to the waste gas is made small. If the CO₂ content is 30% and its liquidization pressure is 100 bar, then in the temperature range −5–−52,5° C. about 81% of the CO₂ will liquidize. In this case, only part of the waste gas pressure energy remaining from the liquidization will be needed to produce the cooling power and there will be only 1-2 cooling turbines in the cooling cycle. Unless excess cooling power is required, for example, in an oxygen enrichment unit in connection with the power plant, the waste gas flow exhausting at excess pressure from the cooling cycle is heated, for example, in the process's heat exchanger, before it is led to a separate turbine to produce energy.

[0049] The nitrogen oxides (NO_xs) in the combustion gas in the process according to the present invention are absorbed in the absorption solution and are converted there into nitrate ions. The nitrate ions can be easily separated from the absorption solution as nitrates at the same time as the cations leaving it are replaced with corresponding hydroxides. This gives a degree of freedom in the power plant's combustion, as there is no need to attempt to minimize the NO_x content of the combustion gas. Because nitrates are considerably more valuable than hydroxides, it can even be economically advantageous to attempt to achieve large NO_x contents in the combustion gas.

[0050] In the process according to the invention, large thermal fluxes are either transferred in the embodiment based on FIG. 1 from the one liquid flow to another without large, inefficient 'dry' heat exchangers, or the absorption heat is stored in the absorption solution, which releases it in desorption, as happens in the embodiment based on FIG. 2.

[0051] The use of the process and apparatus according to the invention creates a considerably more energy-economical method of separating carbon dioxide, compared to the state of the art. On the basis of the above examples of embodiments, it is clear that there are numerous other apparatus concepts implementing the process according to the invention besides those described above. Thus, the different implementation variations of the process are extremely diverse, and are thus not restricted to only the examples of embodiments described above.

1. A process for producing liquid carbon dioxide (CO₂) from combustion gas at normal pressure, in which process:

the CO₂ of combustion gas led to the lower part of an absorption column is absorbed in an absorption solution that flows downwards from its upper part,

the CO₂-poor combustion gas is removed from the upper part of the absorption column,

the said absorption solution is led to the upper part of a desorption column,

the CO₂ is desorbed from the absorption solution led to the upper part of the desorption column to form a gaseous CO₂ concentrate, which is removed from the upper part as the said absorption solution flows to the

lower part of the column, from which it is re-circulated to the upper part of the said absorption column,

the CO₂ concentrate obtained is brought to a high pressure, and

most of the concentrate's CO₂ is liquidized,

characterized in that the CO₂ is transferred from the normal-pressure combustion gas using a nearly reversible absorption and desorption process, to the CO₂ concentrate at a lower pressure at a temperature near to that of the combustion gas being processed, preferably in the temperature range 35-75° C., in such a way that the absorption and desorption processes take place in columns at more or less the same temperature, preferably without importing external heat to the said processes.

2. A process according to claim 1, characterized in that, in order to improve the reversibility of the desorption process, an air flow is led to the lower part of the desorption column, by means of which a constant pressure is maintained in the column the partial pressure of the CO₂ at each height in the column being more or less in equilibrium with the composition of the absorption solution at the same height.

3. A process according to claim 1, characterized in that, in the liquidization of the CO₂ concentrate desorbed in the desorption column, the CO₂ concentrate is cooled, brought to a high pressure and led for liquidization to the lower part of a liquidization column, from where it flows upwards at the same time as part of the CO₂ of the concentrate liquidizes on heat-exchanging surfaces arranged in the column and the unliquidized waste gas is removed from the top of the column and the liquidized CO₂ is removed from the lower part of the column.

4. A process according to claim 3, characterized in that a series of several heat exchangers is arranged in the liquidization column, between which the expansion of the unliquidized waste gas is arranged to take place using cooling turbines with pressure levels diminishing in stages, in such a way that the unliquidized waste gas removed through the top of the liquidization column is led to a cooling-expansion cycle, in which the pressure energy of the waste gas is used to produce at least part of the cooling power required for the liquidization of the CO₂ and in which the said cycle comprises the following sub-stages

the waste gas is led to the upper part of the heat exchanger,

the waste gas is removed from the lower part of the heat exchanger and led to a cooling turbine to be expanded, and

the expanded waste gas is led from the cooling turbine to a new cycle to the upper part of the next heat exchanger

and the said cooling-expansion cycle is continued until the pressure of the waste gas is essentially 1 bar, when it is removed from the lower part of the last heat exchanger in the cycle.

5. A process according to claim 3, characterized in that the said heat exchangers are arranged in the liquidization column in such a way that their temperature levels essentially equal each other and their cooling effect is concentrated in the lower part of the column.

6. A process according to claim 3, characterized in that, in the lower part of the liquidization column near to the inlet connection for the CO₂ concentrate, a pre-condenser cooled

by cold water is arranged essentially separately from the cooling-expansion cycle, and in which part of the CO₂ of the concentrate coming to the column is liquidized before it flows onto the surface of the heat exchangers.

7. A process according to claim 1, characterized in that beneath the surface of the absorption solution there are heat-exchanging means arranged in the lower part of the desorption column, with the aid of which the absorption solution brought from the lower part of the column evaporates CO₂ from the solution in the bottom of the column, before it is transferred to the absorption column, while the said absorption solution is led from the heat-exchanging means to the upper part of the desorption column.

8. A process according to claim 1, characterized in that the absorption heat is stored in the absorption solution, from which it is released in desorption.

9. A process according to claim 1, characterized in that an additional absorption unit is arranged in the process between the absorption and desorption columns, in order to increase the CO₂ content of the absorption solution brought from the lower part of the absorption column and the CO₂ partial pressure of the concentrate obtained from the desorption, and in which

the unliquidized waste gas of the cooling-expansion cycle of the liquidization column is led in from the lower part of the additional absorption unit and

the said CO₂-impoverished waste gas is led out from the upper part of the column back to the following sub-stage of the cooling-expansion cycle of the liquidization column.

10. A process according to claim 1, characterized in that a water solution potassium and/or sodium carbonates is used as the absorption solution.

11. A process according to claim 1, characterized in that the evaporation/condensation cycle of the water vapour evaporating into the concentrate in the desorption column or brought to the column, and/or an ejector is utilized to increase the pressure level of the CO₂ concentrate.

12. A process according to claim 1, characterized in that the energy consumption of the CO₂ liquidization process is minimized by adjusting the composition and pressure of the concentrate and the other conditions of the liquidization process in such a way that the cooling effect produced by the process covers precisely the cooling power requirement of the liquidization process.

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