The use of waste incineration residues for the upgrading of biogas

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Abstract

This paper reports the results of the tests carried out on a pilot plant built around the concept of an innovative biogas upgrading method, to produce biomethane, based on the use of solid residues from waste incineration for CO_2 capture. The proposed upgrading method is based on the idea of putting in series two processes, studied in a previous project. In particular the two steps in series process is based first on the direct carbon dioxide adsorption from biogas using bottom ash, followed by absorption with regenerated alkali solutions process, where air pollution control residues are used for regeneration. Results showed that the process is able to effectively produce biomethane. The residues requirement for industrial operation was estimated for different biogas upgrading plant sizes, highlighting a quite high request of bottom ash and low request for air pollution control residues.

Key words: incineration residues, biogas, upgrading, accelerated carbonation, industrial symbiosis.

1. Introduction

Biogas can be generated from organic waste anaerobic digestion or as landfill gas. The heating value of biogas, ranging between 15 and 30 MJ/Nm^3, can be raised when methane content of more than 96-97 % is achieved [1]. This can be realized by an upgrading process, consisting of improving its quality by removing carbon dioxide (CO_2) in order to increase the methane content in the gas. Of course also the trace contaminants (i.e. dust, hydrogen sulphide) and water vapor need to be removed in order to prevent corrosion and mechanical wear of the equipment and machinery. Biogas upgrading allows producing a fuel called *biomethane* which could replace natural gas. Therefore, after proper upgrading, biomethane can be fed into the natural gas distribution grid or used as vehicle fuel. International standards for biomethane quality are not yet available, even if the European Commission asked to the European Committee for Standardization (CEN) a standard definition. Several countries in North Europe adopted their own standards [1]. In this work it is assumed that an upgraded biogas should contain about 96% in vol. of methane (CH_4) for complying with biomethane definitions.

Presently a wide range of different methods and technologies are commercially available for cleaning and upgrading biogas. These include absorption by chemical solvents, physical absorption, cryogenic separation, membrane separation and CO_2 fixation by biological or chemical methods [2]. The most common are High Pressure Water Scrubbing (HPWS), amine scrubbing, and Pressure Swing Adsorption (PSA).

With respect to these commercial processes, two innovative carbon dioxide removal methods were proposed and investigated, characterized by the capture of the removed carbon dioxide, contributing to the overall greenhouse gases balance with a negative emission (sink). Further, the proposed methods use industrial residues for carbon dioxide capture and storage, through an accelerated carbonation process and were developed during the last years in the frame of a

European cofounded project (LIFE08 ENV/IT/000429). Within this frame the two methods were studied by experimental approach in a separate way, as alternative possibilities to upgrade biogas [3-9]. The two methods were based on the following concepts: i) in the first one, CO_2 is removed from the biogas by alkali solution absorption; the load solution, containing carbonate and bicarbonate ions is regenerated through reaction with calcium contained in the Air Pollution Control residues (APC), forming calcium carbonate (APC accelerated carbonation) and reproducing – at least in part – the original alkali compound; ii) in the other method, direct CO_2 adsorption on bottom ash (BA) takes place, by means of gas/solid direct contact process, forming calcium carbonate in the bottom ash (accelerated carbonation). This second process was originally proposed and proved at laboratory scale [10-11].

With respect to the above methods, a further development was carried out, consisting of putting in series the two processes, in order to try to obtain the benefits associated with each of them and reducing the drawbacks which are inevitably related with each of them. Such development activities were carried out within the frame of a project co-financed by the Italian Minister of Environment.

In particular the two steps in series process is based on the preliminary application of the direct carbon dioxide adsorption from biogas using BA, followed by the alkali solution absorption with regeneration by APC. Carbon dioxide adsorption using BA, being quite simple and low cost process [8], is used to remove large part of the carbon dioxide contained in the entering biogas, while the alkali solution absorption, which is more expensive [7] than the other one, is applied as a refining process, able to provide the desired quality of the exiting biomethane. The simplified layout of the two stages process is shown in Figure 1.

The input materials to the process are both solid residues produced as waste streams from waste incineration. In particular BA are the solid residues remaining in the furnace after combustion, mainly consisting of unburnable materials, i.e. inert. APC residues are generated from the flue gas treatment in waste incineration plants, in particular, the type of APC residues useful for the proposed process are obtained as scraps from acid gas removal step, in the case of dry process using calcium based adsorbents. The process products and the reactant excess – which is the useful part containing calcium hydroxide – are collected as a powder by the downstream fabric filter.

Using these residues for upgrading the biogas, produced by landfills or biowaste anaerobic digestion, is an interesting example of industrial symbiosis, within the field of waste management industry.

The paper reports the results of some experimental tests focused on the coupling of the adsorption and absorption sections, aimed at defining how much CO_2 shall be removed by the adsorption step and how much by the absorption one, in order to estimate the overall amount of the two types of residues, on annual basis, for industrial scale application.
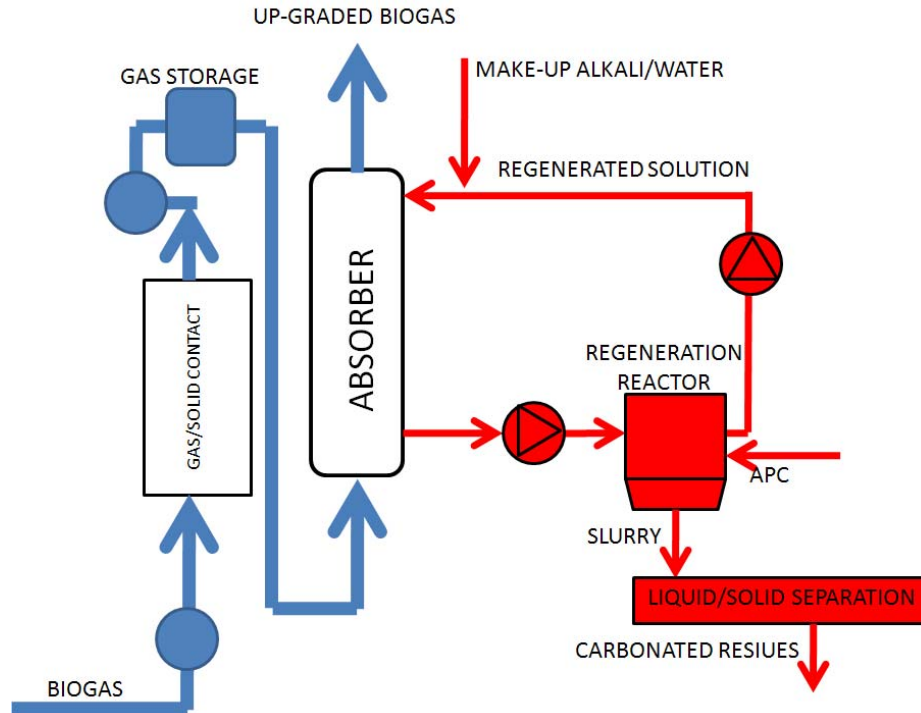
Figure 1. Simplified layout of the innovative biogas upgrading process.

2. Materials and methods

2.1 Pilot plant

The two existing pilot plants – previously used to test the operation of the two processes separately – were modified and integrated in order to allow the operation in series. The pilot plants are hosted at a landfill site, located in central Italy.

Gas extracted from a landfill gas station, is first fed to adsorption bed filled with BA, by a side channel blower. The gas flow rate entering the adsorption section is adjusted by flow regulators and may range from about 4 to 10 Nm^3/h.

The adsorption section is made by two reactors in 316L stainless steel designed to hold about 500 kg of ash with density of about 1,66 t/m^3. The reactors can be opened from the top and frontally by means of covers respectively for loading the ash and unloading them once the test is finished. Landfill gas flows into each filter bed from the bottom to the top. The ashes rest on a perforated plate - at a certain height with respect to the bottom of the reactor - covered by a geotextile fabric, which retains the small particles and allows the passage of the gas. The free space left between the reactor bottom and the perforated plate provides a chamber which allows the distribution of gas before passing through the ash layer. For external thermal insulation, an insulating adhesive material, provided in rolls, was used.

The gas exiting from the adsorption section is compressed up to 8 bar, by a two stages piston compressor, and stored in a gas tank with a volume of 0,5 m^3. From the storage tank, gas is fed to the absorption column with a constant flow rate ranging from 16 to 20 Nm^3/h. The column is built in stainless steel and packed with Sulzer laboratory DX packing, with an overall height of 990 cm and a diameter of 80 mm. Here the gas is scrubbed with an aqueous solution of sodium hydroxide (NaOH) with a flow rate ranging from 30 to 90 l/h. At the exit of the absorption column, the gas is cooled down of about 10 °C in a condenser and then heated up again of a few degrees by a heating cable.

Input and output gas flow rates to/from the adsorption reactors and to/from the absorption

column are measured by means of rotameters (Yokogawa - RAMC01).

On the input and output gas streams to/from the adsorption reactors and to/from the absorption column, differential pressure is measured by a diaphragm pressure transducer (Delta Ohm-HD 408T 100MBG) able to work in the range from -100 to +100 mbar relative to the atmospheric pressure. Atmospheric pressure is measured by means of a barometric pressure transducer (Delta Ohm HD 9908 BARO) able to work in the range from 700 to 1100 mbar. Gas temperature and the temperature inside the two adsorption beds are measured by means of K-type thermocouples.

Gas flow rate, pressure and temperature are measured and registered in a discontinuous manner (every 1-5 minutes). The measurement instruments are controlled by a programmable automation controller (Compact Field Point – National Instrument) composed by rugged I/O modules and intelligent communication interfaces.

Volumetric gas composition is measured by means of a portable gas analyzer (GA2000) which measures CH_4 and CO_2 by infra-red absorption ($\pm$ 3% absolute accuracy for >15% vol.), O_2 by internal electro-chemical cells. The composition is measured every 1-5 minutes, manually.

Spent solution collected at the exit of the absorption column is fed to a dedicated batch reactor where the liquid and the APC residues are put in contact and stirred for the time necessary for the reaction to take place. Detailed description of the regeneration reactor is reported in [5].

Before admitting the gas, nitrogen was flushed (4,5 m^3/h) through the adsorption reactor, in order to clear the BA voids from air/O_2. The nitrogen supply was stopped when the oxygen content in the output gas was less than 0,2% by volume, then the gas started to be flushed into the two reactors. The storage tank started to be loaded after that nitrogen content in the output gas approached the input gas ones, in order to avoid the feeding into the tank of the nitrogen trapped inside the reactors.

2.2 Materials

Used BA originated from a Municipal Solid Waste (MSW) incinerator, located in central Italy, based on a grid furnace and equipped with waste pre-treatment. The pre-treatment of MSW consists of bag opening and shredding of waste; size separation by rotating drum; magnetic metals removal; secondary shredding. BA are discharged at the bottom of the furnace in a water channel. After receiving the BA, they were stored on pallets of wood covered with geotextile fabric to allow the discharge of a fraction of the contained water. They were also covered by a plastic liner in order to protect them from weather conditions.

The APC residues were sampled from the fabric filter section of a hospital waste incineration plant located in central Italy and they are the same employed in previous tests [3, 5-6]. APC residues require to be pre-washed before being used for regeneration: a mass loss of about 45% takes place [3].

The total mass fraction of $Ca(OH)_2$ of the residues after the washing pre-treatment was 0.555. The amount of APC residues to be used for regeneration is calculated according to the specific ratio of reactive calcium in the APC to carbonate ions in the spent solution (eq_{CaR}/eq_{CO3^-}) equal to 1,55 [3].

An absorption solution prepared with NaOH characterized by 99% reagent grade was used for the experimental tests.

2.3 Experimental tests

Several tests were carried out focusing on the coupling of the adsorption and absorption sections.

In the first adsorption step, CO_2 is suddenly removed and its concentration goes to zero at the very beginning of the operation. Then, after a given amount of time, called here *breakthrough*

time (strictly dependent on the specific gas flow/solid ratio expressed in $Nm^3/(t \cdot h)$), CO_2 starts again to appear in the gas stream exiting from the adsorption reactor [8].

We decided to store the gas exiting from the adsorption step in the tank, until the CO_2 concentration in the tank has a value lower than 4% in vol. Actually, the CO_2 volumetric content equal to 4% not always corresponds to a CH_4 volumetric content of 96%. This is due to the possible presence of oxygen and nitrogen in the entering gas and/or to the possible hydrogen formation by reacting with aluminum [12-14]. Discrepancies in the measured concentrations may be attributed also to the low accuracy of the used gas analyzer. However, the gas stored in the tank has, at the beginning, zero or very low CO_2 concentration, while it increases as long as the process proceeds. Stopping the gas storage according to the assumed criteria assures to obtain, in the mixture accumulated in the tank, a content of CH_4 higher than 96% in vol. in most of the carried out tests, as shown later on in the results. This gas does not require a further treatment, since it already complies with biomethane quality. In the pilot plant, the gas accumulation tank is simply emptied, while, in a real plant this gas would be ready for use as biomethane.

After empting the gas accumulation tank, the gas exiting from the adsorption section is compressed and stored again in the accumulation tank. At the beginning of the gas storing, low CO_2 concentration gas accumulates into the tank, while as long as the operation goes on, gas with higher CO_2 content is stored. The result is that an intermediate CO_2 concentration value is found in the storage tank, after the tank maximum capacity is reached. The CO_2 and CH_4 concentrations are measured and, obviously, were found not suitable for biomethane, so, when maximum load of the tank is reached, the gas is fed to the absorption column, for further refining, with the aim of obtaining a CH_4 content higher than 96% vol.

Several tests were carried out in order to determine the way of coupling the two removal processes. These tests were carried out considering as reference parameter the specific biogas flow rate through the BA bed, which was varied from 4 to 9,9 $Nm^3/(t \cdot h)$. Table 1 reports the assumed values for the biogas specific flow rate in the experimental tests, along with the amount of BA loaded in the reactors, the actual gas inflow rate and the CO_2 and CH_4 contents in the inlet gas.

The absorption tests were carried out using an aqueous solution of NaOH, with a target concentration of 2,8 eq./l [3] and adjusting the solution flow rate to achieve the desired CO_2 removal, according to the reaction:

$$2\ NaOH_{(aq)} + CO_{2(g)} \rightarrow Na_2CO_{3(aq)} + H_2O \qquad (1)$$
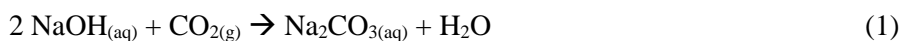
The duration of each absorption test was of around 10 min after which the spent solution was collected, at the exit of the absorption column, and analyzed to determine its main composition in terms of hydroxide and carbonate/bicarbonate content by nitric acid titration following the procedure described in [3].

For some of the performed tests (n. 1, 2, 3, 4, 5 and 9), also the regeneration of the spent solution was carried out, according to the procedure described in [5]. The regeneration process was already proved in previous experimental works [5-6]. The experiments reported here were aimed at checking the yield of regeneration under some different operating conditions, mainly, the lower CO_2 content in the gas entering the column. For lower CO_2 content in the gas stream (i.e. lower partial pressure), the absorption process requires high excess of NaOH (i.e. high solution flow rate) in order to achieve high CO_2 removal. For this reason, an excess of unreacted NaOH is expected in the spent solution.

The global regeneration efficiency (η_R) was calculated according to the equation (2):

$$\eta_R = \frac{[NaOH]_R \cdot V_R}{[NaOH]_{ABS} \cdot V_{ABS}} \qquad (2)$$

Where [NAOH] is the concentration of sodium hydroxide; V is the liquid volume; subscript R refers to regenerated solution; subscript ABS refers to absorption inlet. The measured value of the NaOH concentration in the regenerated solution includes also the NaOH which does not take part in the reaction and, thus, is assumed to be found unaltered after the regeneration, too.

Test number	1	2	3	4	5	6	7	8	9	10
Specific flow rate [Nm³/(t·h)]	4,0	5,0	5,9	6,0	6,9	7,0	7,9	8,1	9	9,9
Amount of BA [t]	930 ± 14,1	940 ± 14	930 ± 14,1	900 ± 14,1	980 ± 14,1	920 ± 14,1	980 ± 14,1	980 ± 14,1	940 ± 14	980 ± 14,1
Biogas flow rate [Nm³/h]	3,7 ± 0,5	4,7 ± 0,2	5,5 ± 0,4	5,4 ± 0,4	6,8 ± 0,4	6,4 ± 0,4	7,7 ± 0,3	7,9 ± 0,5	8,5 ± 0,1	9,7 ± 0,4
CH₄in [% vol,]	59,7 ± 1	61,4 ± 0,9	60,3 ± 1,2	60,2 ± 0,4	61,0 ± 0,7	60,0 ± 0,3	60,8 ± 0,4	60,4 ± 0,5	60,0 ± 0,2	60,6 ± 0,5
CO₂in [% vol,]	39,9 ± 0,7	39,9 ± 0,7	40,4 ± 0,5	40,9 ± 0,5	41,1 ± 0,7	40,8 ± 0,6	41,4 ± 0,6	40,8 ± 0,6	40,7 ± 0,6	42,2 ± 0,4

Table 1. Operating conditions for the performed experimental tests.

3. Results and discussion

From Table 1 it is possible to highlights that the different performed tests were, luckily, characterized by quite similar values for the volumetric concentration of CO_2 and CH_4 in the entering real biogas coming from the landfill, which allows comparing the results from different test. CH_4 average concentration for the tests was 60,4% (min: 59,7%; max: 61,4%). CO_2 average concentration for the tests was 40,8% (min: 39,9%; max: 42,2%).

The main results collected during the experimental tests are reported in Table 2.

Table 2 reports the composition of the gas accumulated in the tank before CO_2 concentration in the gas, at the BA bed exit, exceeds the 4% in vol. As it can be noticed, the gas always complies with biomethane quality, with the exception of tests 5 and 10. In these two cases the same gas accumulated into the tank was further processed in the absorption column. In the other cases the tank was simply emptied and gas storage started again.

The gas supplied to the absorption column was characterized by a CH_4 average concentration equal to 90,9% (min: 89,2 %; max: 94,9%) and CO_2 average concentration equal to 8,6% (min: 6,0%; max: 10,8%).

Quite obviously, in order to reach good removal of CO_2 and consequently adequate concentration of CH_4 for biomethane, it is necessary to use higher liquid solution to gas (L/G) ratio in the absorption column, as the CO_2 content in the entering gas increases (i.e. CH_4 content in the entering gas decreases). Figure 1 shows how the experimental tests were able to produce biomethane in all the cases, with the exception of the tests conducted at 4, 6 and 9 Nm³/(t·h), for which CH_4 exiting concentration was about 95,3-95,5%. In these cases, a too low L/G ration was used. For the absorption process of test n. 2, the solution obtained from the regeneration step of test n. 3, was used, after appropriate water and NaHO make-up.

Test number	1	2	3	4	5	6	7	8	9	10
Specific flow rate $Nm^3/(t \cdot h)$	4	5	5,9	6	6,9	7	7,9	8,1	9	9,9
Adsorption step results										
Breakthrough time [min]	196	129	94	117	64	99	60	97	56	37
Total entering volume [Nm^3]	12,87	19,37	22,13	13,75	6,07	19,35	14,72	19,88	21,05	9,10
Produced biomethane volume [Nm^3]	3,31	6,34	10,34	5,12	-	6,84	5,08	7,60	6,15	-
Volume of gas to be refined [Nm^3]	5,20	6,28	4,83	3,58	3,72	5,15	4,48	4,59	7,88	5,81
Composition of the gas stored in the accumulation tank before the stop criteria										
$CH_{4 \, stored}$ [% vol,]	96	98,2	96,9	97,1	92,1*	97,7	97,1	97	96,8	94,9*
$CO_{2 \, stored}$ [% vol,]	3,8	1,7	2,5	2,4	9,7	1,6	2,3	1-2,1	2,1	6
Absorption step conditions and results										
$CO_{2 \, in}$ [% vol,]	8,8 ± 0	6,95 ± 1,5	10,8 ± 0	8	9,7 ± 0	9	9,0 ± 0	8,8 ± 0	9,4 ± 1,9	6
CH_{4in} [% vol,]	90,1 ± 0	89,2 ± 1,7	90,0 ± 0	90,0 ± 0	92,1 ± 0,1	91,0 ± 0	90,2 ± 0	91,8 ± 0,4	89,5 ± 0	94,9 ± 0
Gas flow rate in [Nm^3/h]	19,78 ± 0,5	19,94 ± 0,2	19,8 ± 0,1	19,8 ± 0,5	19,7 ± 0,8	20,0 ± 0,2	19,0 ± 0,5	19,6 ± 1,2	20,5 ± 0,3	20,0 ± 1,1
Solution flow rate [l/h]	40	60	60	30	40	40	40	40	40	20
NaOH [eq,/l]	2,75	2,85	2,75	2,7	2,75	2,75	2,8	2,65	2,7	2,75
$CO_{2 \, out}$ [% vol,]	2,8 ± 0,2	2,26 ± 0,3	2,8 ± 0,9	2,9 ± 0,5	3,7 ± 0,7	3,1 ± 0,5	2,6 ± 0,6	2,3 ± 0,8	2,9 ± 0,2	2,1 ± 0,2
$CH_{4 \, out}$ [% vol,]	95,5 ± 0,2	96,3 ± 0,3	96,8 ± 1	95,5 ± 0,5	96,8 ± 0,5	96,5 ± 0,5	96,1 ± 0,6	96,8 ± 1,1	95,3 ± 0,2	96,9 ± 0
Gas flow rate out [Nm^3/h]	18,55 ± 0,1	18,4 ± 0,2	18 ± 0	18,2 ± 0,2	18,6 ± 0,4	18,6 ± 0,1	17,1 ± 0,3	18,4 ± 0,8	19,1 ± 0,5	19 ± 1,1

* CH_4 lower than biomethane quality

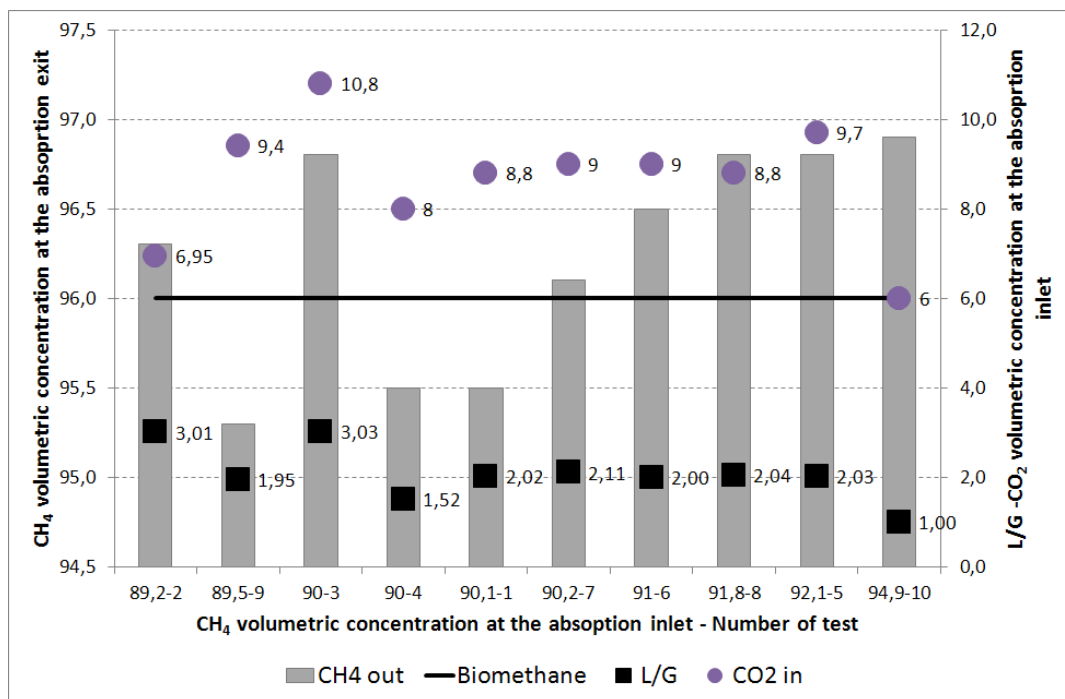Table 2. Main results for the performed experimental tests.

Figure 2. Comparison of entering/exiting CH_4, entering CO_2 and L/G ratio for the absorption column.

3.1 Regeneration

The main results of regeneration tests are reported in Table 3. Regeneration efficiency – as defined before - lays in the range 48,96-65,60%. The lowest value is obtained for the regeneration step of test n. 2, in which the solution fed to the absorber, came from a previous regeneration, in turn. The accumulation of carbonate ions is probably responsible for lowering regeneration performances.

Values of the regeneration efficiency are generally quite similar to those found in previous works [5-6], even if the regeneration efficiency was defined in a slightly different way. The regeneration efficiency values were also calculated in the same way of previous works (not shown here for conciseness), showing lower values, ascribed to negative effects of unreacted excess of NaOH in the spent solution.

Test number	1	2	3	4	5	9
Spent solution [l]	5,52	7,93	8,36	5,18	6,23	5,16
Total alkalinity [eq,/l]	2,78	2,88	2,75	2,73	2,75	2,75
NaOH [eq,/l]	0,70	1,05	1,05	0,50	0,65	0,50
Na_2CO_3 [eq,/l]	2,08	1,83	1,70	2,23	2,10	2,25
APC residues [kg]	1,66	1,16	2,06	1,67	1,90	1,68
Regenerated solution [l]	4,97	6,51	7,33	4,55	5,38	4,94
Total alkalinity [eq,/l]	2,13	2,00	2,20	2,10	2,10	2,10
NaOH [eq,/l]	1,90	1,70	1,98	1,80	1,85	1,85
Na_2CO_3 [eq,/l]	0,23	0,30	0,23	0,30	0,25	0,25
Regeneration efficiency [%]	62,29	48,96	62,93	58,55	58,09	65,60

Table 3. Main results for the performed regeneration tests.

3.2 Use of residues

From the results reported before it is possible to extrapolate a preliminary relationship between the amount of processed biogas and the amount of used residues for the overall process (BA and APC residues).

In the adsorption process an average of 71 kg of BA per Nm^3 of processed biogas are required (min 42; max 161), with lower values in general for lower specific gas flow rate.

From the reported results it is possible to calculate the amount of APC residues required to process 1 Nm^3 of entering biogas (considering the specific ratio between the volume that needs to be further refined in the absorption step and the entering biogas, the L/G ration and the average APC consumption per each regeneration test; assuming that the volume of absorption entering solution equals the volume of spent solution). An average value of 0,7 liters of absorption solution per Nm^3 of entering biogas are required, while about 0,27 kg of APC residues are requested for regenerating 1 liter of spent solution. This leads to an approximate specific value of APC residues consumption about 0,2 kg/Nm^3 of entering biogas.

Form these values it is possible to preliminary estimate the amount of residues required for the operation on industrial scale of a biogas upgrading plant, in a range of size in term of entering biogas, considering 7920 hours of operation per year. For the BA consumption, consumption values are reported for the calculated minimum, maximum and average specific consumption values.

Biogas plant size [Nm3/h]	100	200	300	400	500	1000
BA min [t/y]	33.283	66.567	99.850	133.133	166.417	332.833
BA max [t/y]	127.868	255.736	383.605	511.473	639.341	1.278.682
BA media [t/y]	56.232	112.464	168.696	224.928	281.160	562.320
APC [t/y]	158	317	475	634	792	1.584

The specific production of BA and APC residues from an incineration plant is, respectively, about 20-30% and 1,5-4,0 % [15] For BA the production from one medium (100.000-250.000 t/y) to large (> 250.000 t/y) size incineration plant (classification according to [16]) would be required to process up to 400 Nm^3/h f biogas (in the case of minimum BA specific consumption). On the contrary the amount of required APC residues is quite small compared to those that may be produced by small size incineration plants.

It was not reported in this work that accelerated carbonation of BA and APC also has some beneficial effects on their environmental behavior, reducing the leaching of several metals [6, 8]. This aspect should be considered and evaluated, together with the overall process, from an environmental and economic perspective.

Conclusions

An innovative process for biogas upgrading was proposed, tested and demonstrated by pilot plant experiments. The process is based on two steps: first an adsorption one based on the use of bottom ash from incineration to capture the CO_2 in the biogas. The second step, used to refine the gas exiting form the first one, is based on absorption of CO_2 by alkali chemical absorption. The Air Pollution Control residues from incineration are used as a source of calcium hydroxide to regenerate the absorption spent solution.

The process is able to produce biomethane, but the bottom ash requirement is quite high, when the annual operation is considered. Nevertheless, the process could be quite interesting to be applied in the case of an industrial site where the different types of waste treatment plants (i.e. incinerator and anaerobic digestion or landfill) are present.

The effective feasibility of the process should be further investigated both from the economic and environmental point of view.

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