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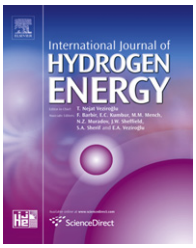

MSW landfill biogas desulfurization

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ABSTRACT

Biogas utilization in MCFC systems requires a high level of gas purification in order to meet the stringent sulfur tolerance limits of both the fuel cells and the reformer catalysts. In this study, two commercial activated carbons (ACs) have been tested for H_2S removal from the biogas produced at the Montescarpino Municipal Solid Waste landfill in Genoa, Italy. The performed analyses show a low selectivity of activated carbon towards the adsorption of only sulfur species. This represents a drawback for the use of this type of system, however, the use of mixed beds of different ACs has demonstrated to be advantageous in improving the removal efficiency of H_2S. Thus, the adsorption treatments with AC can ensure the high level of gas desulfurization required for fuel cell application. Nevertheless, the low adsorption capacity observed using landfill biogas would lead to high operative costs that suggest the application of a preliminary gas-scrubbing stage.

1. Introduction

Biogas is a renewable biofuel that could have a significant impact on future energy scenarios [1–5]. Biogas may be either landfill gas or anaerobic digestion gas from organic fractions of Municipal Solid Wastes (MSW), agricultural waste, industrial wastewater, sewage sludge, etc. This biofuel is produced in large quantities in Italy and most of it comes from MSW landfills. Landfills are the main waste disposal sites in Italy and they can produce enough biogas for at least another 20 years.

The current Solid Waste Management Policy is oriented towards increasing differentiated waste collection, with a subsequent gradual reduction of MSW disposal in landfills. In this way, the separated organic fraction of municipal wastes will be treated in anaerobic digesters to produce biogas, which may be increased through the use of livestock effluents and energy crops [6].

The present work was performed as part of the BioH_2Power project, whose objective was to conduct a feasibility study of a biogas-fuelled processing system for decentralized electricity and hydrogen production. This system is made up of a power unit based on a Molten Carbonate 250 kW Fuel Cell (MCFC) and a hydrogen upgrading section. The latter will be specifically tailored for a fuelling station that is capable of supplying about 20–100 H_2-vehicles per day (according to easy alculations this should correspond to a production of a minimum of 750 m^3_N/d or about 0.4 mol/s of pure hydrogen at 99.99%) [7].

MCFCs offer several advantages [8], but their anodes, as well as the CH_4-reforming catalysts, are very sensitive to the poisoning effects of some molecules that are often found in biogas: sulfur compounds, halogenated hydrocarbons and siloxanes [9]. In fact, depending on its source, biogas can have different compositions, but in general it is mainly constituted by

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CH_4 (45–75%) and CO_2 (25–50%). The main sulfur compounds present in biogas are H_2S (100–1000 ppm), mercaptanes (0–100 ppm) and traces of COS, CS_2 and SO_2, but it also contains other compounds in traces such as chlorinated and fluorinated hydrocarbons, siloxanes, organic and inorganic acids and NH_3.

Currently, adsorption is the most common technology applied to obtain ultra-low sulfur levels for fuel cell applications [10]. At present, many commercial adsorbents are used for natural gas desulfurization at ambient temperature and pressure: activated carbon (AC), silica, alumina, zeolites and some metal oxides [11]. The physical/chemical absorption method is also applied for biogas purification, since several hazardous components can be removed at the same time, together with dust particles. Scrubbing methods, with pressurized water or chemical solvents in aqueous solutions, are generally employed for this purpose. Nevertheless, if this approach is used, it is not possible to reach the low concentration of H_2S (<0.1 ppm) that is necessary to avoid poisonings of the MCFC catalysts, which instead could be possible if adsorption processes were used.

Different strategies for biogas desulfurization have been evaluated in this study. Experimental adsorption tests were conducted on the biogas extracted from the Montescarpino MSW landfill, in Genoa, Italy. On the basis of the experimental results, a cost estimation of a different solution, employing a preliminary scrubbing purification step, has also been made.

2. Experimental

The adsorption of sulfur compounds by different materials was studied in an experiment at the Montescarpino (GE) generation plant, which is managed by Asja Ambiente Italia. Different activated carbons were tested to purify the biogas extracted from the landfill.

2.1. The Montescarpino power plant

Biogas produced through anaerobic fermentation of the organic matter disposed in the landfill was collected and burned in endothermic engines to produce electric energy.

The landfill biogas power plant is composed of several sections:

- biogas collector;
- biogas conveyor;
- biogas suction, treatment, analysis and torch combustion; and
- electric energy generation, transformation and distribution.

2.1.1. Biogas collector section

Landfill biogas is collected through captation wells drilled on the site. One meter diameter wells were drilled using rotation and vertical progress techniques. Draining pipes in 200 mm diameter HDPE for biogas collection were pulled down into the wells and the interstitial space was filled with siliceous gravel. The well heads, made of stainless steel, are flanged at the pipes, and include sleeves with plugs in order to introduce percolate evacuation pumps.

2.1.2. Biogas conveyor section

This section is composed of HDPE pipelines, of different diameters, in function of the class of the combustible gas that is transported. The substations consist of manifolds for line grouping, with the lines consisting of hot galvanised carbon steel drums and flanged derivations to connect the pipes to the well heads. The joints were made by welding and electro weld sleeve head to head, and this process was carried out by specialised operators.

2.1.3. Biogas suction, treatment, analysis and torch combustion section

The biogas conveyed from the substations passes through a first purification section made up of: a primary coalescer, where a first separation of the condensate takes place; a heat exchanger, with a glycol solution circuit connected to a chiller that brings the gas temperature down to approximately 0–4 °C; a secondary condensate separator; a dry filter to partially remove residual solids.

After this treatment, the biogas, which is now at a lower temperature and purified of macroscopic pollutants (particles and humidity), passes through a multistage centrifuge blower and is conveyed to the power generating sets. This section is also equipped with systems to analyze the biogas flow and control systems, e.g. oxygen and methane analyzers. Finally a biogas combustion torch, with a minimum combustion of 25% CH_4 has been installed for biogas combustion, in the case of engine malfunctioning.

2.1.4. Electric generation, transformation and distribution

The electric generation section consists of 6 Jenbacher engines with a nominal power of 1415 kWe, which are assembled in a soundproof container and cooling radiation masses. The engines are equipped with a GE thermal reactor for the abatement of CO and the emission of the hydrocarbons into the atmosphere. The emissions are continuously monitored by a Siemens Gas analyzer for CO, SO_2 and NO_x. The plant has a data acquisition and supervision system to collect parameters from the biogas suction and electric energy generation sections.

2.2. Gas adsorption section

The biogas is treated by means of an adsorption material bed installed at the engine inlet. Six different reactors have been installed at the Montescarpino power plant for gas desulfurization, in order to reduce the dimensions of the reactor in order to make loading and emptying operations easier.

The engine emissions are conveyed to three stacks, as shown in Fig. 1. As three infrared SO_2 gas-analyzers are connected to the three stacks, the same adsorption material was used in the reactors installed in the engines connected to the same stack during the experimental campaign.

The reactors used to assess the activated carbons adsorptive capacity have the following characteristics.

- Height: 2 m.
- Diameter: 1 m.
- Material: Stainless Steel AISI 304.
- A cochlea loading system.

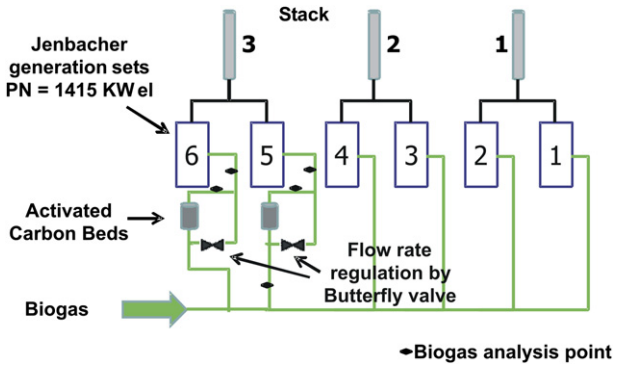

Fig. 1 – Power generation and adsorption section.

The biogas flows from the bottom of the reactor to the top. The system has a bypass line on which a manual butterfly valve has been installed to regulate the treated flow rate. The biogas flow rate was estimated on the basis of the total flow rate to the power generation plant. The total flow rate is measured by a Pitot tube sensor installed on the general purification section pipeline. The flow rates to the six engines were calculated on the basis of the instant power generation of each engine. These data are instantly registered and transmitted to the central office through a telemonitoring system.

Two different commercial activated carbons (ACs) for biogas purification were tested with different bed configurations. The ACs, which are among the most adopted commercial AC for this purpose [12], are commercialized by Norit and their characteristics are given in Table 1. Norit Roz 3, the most expensive material, is specifically used for the removal of sulfur compounds; instead, Norit RB4W, the cheapest one, is less selective and is used for the removal of halogenated hydrocarbons.

These two types of AC were tested in different solutions.

2.3. *Test monitoring*

The measurement of sulfur compounds in the field was limited to hydrogen sulphide. On the basis of periodical analysis conducted on biogas collected on site by the *Programma Ambiente s.r.l.* laboratory, this pollutant results to be the most abundant in the biogas (Table 2).

The mean concentration of H_2S is generally 100–200 ppmv, while the mercaptans content is 2–3 ppm. The periodic analyses at the Montescarpino landfill have shown that the measured mercaptan concentration over the last few years is in the 0.3 and 0.6 ppm range while the hydrogen sulphide is in the 180–200 ppm range. The effectiveness of the mercaptan removal treatment has been verified through gas chromatographic analysis.

The concentration in the field was revealed using a portable infrared GA2000 analyzer equipped with an electrochemical cell. The instrument measures CH_4, CO_2, O_2, CO and H_2S. The pollutant concentration in the biogas was measured before and after the adsorption beds, and at the engine inlet. The analysis was conducted daily by the power station workers. The biogas was also collected in Tedlar bags and analyzed using an Agilent gas chromatograph (GC) equipped with a mass spectrometer (MS) and a specific sulfur chemiluminescence detector (SCD) for sulfur compounds identification at very low concentrations (0.1 ppb).

Table 1 – Commercial activated carbons datasheet.

Type	Principal characteristics
ROZ3	KI content: min 2% wt Moisture: max 5%wt Density: 0.47 kg/l Pressure drop at 25 cm/s: 2 KPa/m Diameter: 3 mm
RB4W	Steam activated extruded carbon Moisture: max 5%wt Density: 0.41–0.46 kg/l pressure drop at 25 cm/s: 1.5 KPa/m Diameter: 4 mm

Table 2 – Analysis campaign conducted by *Programma Ambiente s.r.l.*: Nov, 2008.

Parameter		Unit of measure	Analysis method
Temperature	26	°C	UNI EN 10169
Humidity	0.2	% v	UNI EN 10169
Oxygen	0.8	% v t.q.	GC-TCD
CO_2	35.7	% v t.q.	Volumetry (Orsat)
CO		% v t.q.	GC-TCD
N_2	8.6	% v t.q.	GC-TCD
H_2	0.1	% v t.q.	GC-TCD
Methane	52.5	% v t.q.	GC-TCD
H_2S	0.019	% v t.q.	UNICHIM
P.C.I.	18799	kJ/m^3_N	
P.C.I.	4490	kcal/m^3_N	
PM	0.1	mg/m^3_N	UNI EN 13284
Siloxanes	0.1	mg/m^3_N	NST Ah IV-2
NH_3	8.6	mg/m^3_N	UNICHIM 632
HCl	0.8	mg/m^3_N	D. 25/08/2000
Organic-Cl–	9.79	mgCl/m^3_N	IRSA/Q100/51
Total Cl + F	9.79	mgCl/m^3_N	
HF	4	mg/m^3_N	D. 25/08/2000
Organic-F	0.5	mgF/m^3_N	IRSA/Q100/51
Total F	3.8	mgF/m^3_N	
Total Cl + F	13.6	mg/m^3_N	
H_2S	291.0	mg/m^3_N	UNICHIM 634
H_2SO_4	1.3	mg/m^3_N	D. 25/08/2000
Mercaptans	0.6	mg/m^3_N	ASTM D-2913
SO_2	3.0	mg/m^3_N	UNI 10246/I
Zolfo totale	275.0	mgS/m^3_N	
HC > C5	207.3	mg/m^3_N	UNI EN 13649
Aromatic HC	54.6	mg/m^3_N	UNI EN 13649
TOC	256.5	mg/m^3_N	UNI EN 13649
NH_3	11.3	p.p.m.	
HCl	0.5	p.p.m.	
HF	4.4	p.p.m.	
H_2S	191.4	p.p.m.	
H_2SO_4	0.3	p.p.m.	
Mercaptans	0.2	p.p.m.	

3. Results and discussion

The experimental data were analyzed to evaluate the adsorption capacity, efficiency and treatment costs.

Table 3 – Adsorbent bed compositions.

Activated carbon	Weight percent (wt%)				
RB4W	0	100	40	60	70
ROZ3	100	0	60	40	30

3.1. Adsorption capacity of activated carbons

The tests were performed by treating the biogas with different beds which were loaded with the two activated carbons in different proportions, as shown in Table 3. The total quantity of adsorbent in the reactors was 700 kg and the RB4W AC was always positioned in the bottom part.

The total flow rate through the reactors was regulated using the manual butterfly valve installed on the bypass line, as shown in Fig. 1. The regulation was necessary to maintain a constant flow rate through the bed, even though there is a variation of the total flow to the engine which is caused by the need to vary the biogas production. The total flow rate was maintained at 500 Nm^3/h. The GSHV was about 335 h^{-1} during the tests. The gas velocity through the bed was calculated to be 17.6 cm/s, which, considering the characteristics of the activated carbons beds, causes a pressure drop of less than 3 mbar.

An initial series of tests was performed with the first three compositions of the adsorbent bed. The calculated adsorption capacities at breakthrough are presented in Table 4. The materials were considered to have broken through when an outlet H_2S concentration of 1 ppm was observed, compared to a mean measured inlet concentration of 245 ppm H_2S.

These results show that RB4W has a very low adsorption capacity for hydrogen sulphide. As will be shown in the next section, this could be due to the capacity of this material to adsorb all the organic hydrocarbons in the biogas, and not only the sulfur species [13]. Moreover, beds with percentages of 60–40 and 0–100 of ACs have almost the same mean H_2S uptake capacity. Thus, considering that RB4W is cheaper than ROZ 3, the best cost-effective solution in this case seems to be a mixed bed with 60 wt% of the former material and 40wt% of the latter one.

However, in order to optimize the proportion of the two materials on the adsorption bed, a second series of tests was conducted with different compositions of the two ACs. The results presented in Table 5 shown that, with a reduction of 10% of the quantity of AC ROZ3, the total adsorption capacity

Table 4 – Breakthrough adsorption capacities of the first series of tests.

wt% of AC RB4W–wt% of AC ROZ3	H_2S_{ads} [kg]	H_2S_{ads}/kgAC (%)	Mean value (%)
60–40	40.62	8.12	8.01
60–40	39.45	7.89	
100–0	14.76	2.95	3.00
100–0	15.20	3.04	
0–100	43.24	8.65	7.96
0–100	36.31	7.26	

Table 5 – Breakthrough adsorption capacities in the second series of tests.

wt% of AC RB4W–wt% of AC ROZ3	H_2S_{ads} [kg]	H_2S_{ads}/kgAC (%)	Mean value (%)
70–30	45.69	9.14	10.08
70–30	55.10	11.02	
40–60	48.05	9.61	8.71
40–60	39.08	7.82	
60–40	47.65	9.53	10.88
60–40	61.14	12.23	

of the beds for H_2S remains almost constant. Instead, an increase in the quantity of AC ROZ3 in the bed does not cause a better performance of the treatment system. This effect could be explained by the presence of many other pollutants in the biogas in combination with the low selectivity of the ACs, as will be demonstrated in Section 3.2. In fact, the biogas is first treated by the RB4W AC, which probably also removes humidity [14,15] and other substance that are present and functions like a guard for the AC ROZ3 which uses all its capacity and selectivity to remove the sulfur species.

The different performances measured in the two tests with mixed bed reactors depend on the biogas quality. The adsorption capacity of hydrogen sulphide is influenced by its concentration, but also by the concentration of other pollutants that saturate the less selective part of the adsorption reactor [16].

3.2. Adsorption efficiency of activated carbons

The biogas collected in Tedlar bags during the on-site tests was then analyzed using the gas chromatographic technique. Fig. 2 shows the GC–MS analysis of the landfill biogas. It is evident the wide variety of sulfurated, halogenated but also aromatic and aliphatic hydrocarbons that are presents in the Genoa MSW landfill biogas. Moreover, Fig. 3 shows the GC–MS analysis of the biogas at the reactor inlet and after the treatment with ROZ3, RB4W and the mixed bed with 60–40 wt% proportions.

The collected biogas was also analyzed using an SCD detector, which is specific for sulfur compound detection; the chromatograms are presented in Fig. 4. In general, the three adsorption beds show high efficiency in the removal of all the sulfur compounds present in the biogas. However, AC RB4W is more efficient in the removal of other organic substances. The ROZ3 AC has been confirmed to be selective towards sulfur species, which could be attributed to its KI content [17,18].

3.3. Cost estimation

An estimation of the operative cost using the two AC tested has been made and the results are reported in Table 6. The operative costs for the activated carbon beds were calculated with reference to the experimental performances presented in this work and comprises costs pertaining to the purchase, transport and operation of the emptying and loading of the reactors. In particular, the operative cost of the desulfurization employing the bed of AC RB4W is higher than that of the single

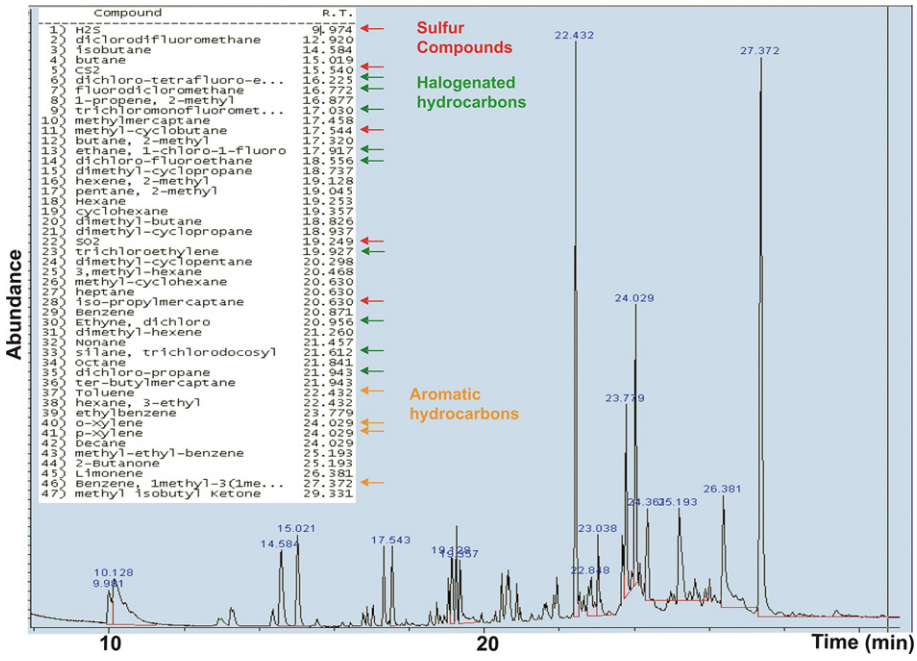

Fig. 2 – GC–MS landfill biogas analysis.

bed of AC ROZ3, most probably due to the low selectivity of the AC RB4W, even if this material is cheaper than the AC ROZ3. In fact, the results presented in the previous sections confirm the low selectivity of activated carbon towards the adsorption of only sulfur species. Such low selectivity implies a reduction of their adsorptive capacity respect the data declared by the manufacturer, which causes a reduction of their effective working time and an increment on the operative costs. This represents a drawback for the use of this type of system, however, the use of mixed beds has been demonstrated to be advantageous in improving the removal efficiency of H_2S. Therefore, the operation costs of the mixed adsorption beds (70–30) have also been estimated in comparison to the use of only one of the AC materials. This solution diminishes effectively the operation cost of the system in about 47% respect the use of the AC ROZ3 (see Table 6).

Between the currently available technologies for landfill biogas desulfurization, the most used in Italy are both adsorption and gas scrubbing. Hence, this second typology of sulfur removal system was also considered for the economic

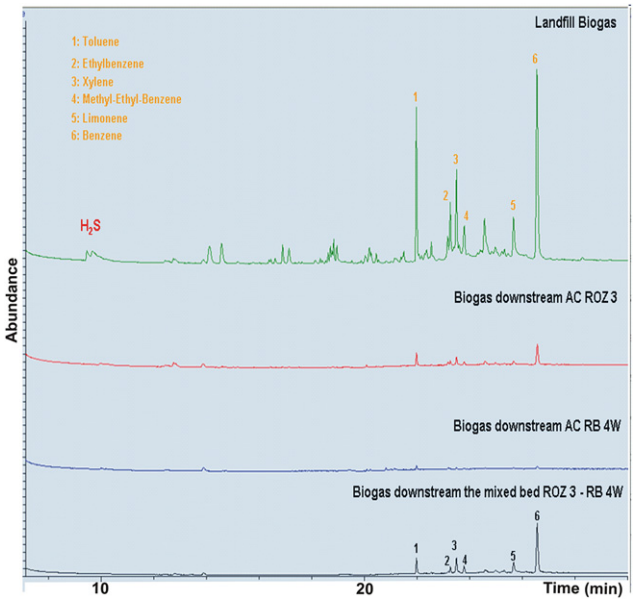

Fig. 3 – GC–MS landfill biogas analysis upstream and downstream the activated carbon beds.

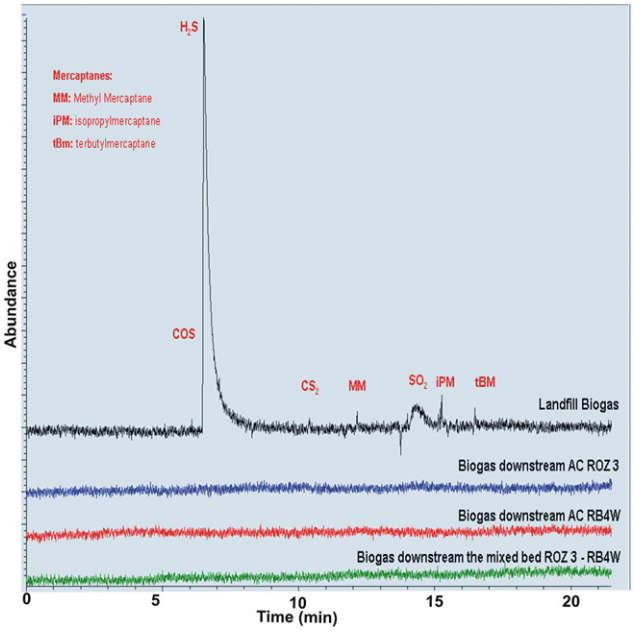

Fig. 4 – GC–SCD landfill biogas analysis of sulfurated compounds upstream and downstream the activated carbon beds.

Table 6 – Estimation of the operative costs of two different desulfurization technologies.

Technology	Operative costs (c€/Nm^3)
AC ROZ 3	1.90
AC RB4W	1.99
AC RB4W–AC ROZ3 (70–30%)	0.89
Scrubber	0.20
Scrubber + AC ROZ3	0.22

evaluation, in order to compare the costs of the use of this approach respect the adsorption with activated carbons [19].

The gas-scrubbing technology was selected as secondary desulfurization system since it is in general the most commonly used in MSW Landfills. This is based on the absorption of the acid pollutants in an aqueous solution of water and soda. The operation costs of the scrubber system were calculated on the basis of literature data and from the theoretical efficiencies of a commercial apparatus. The soda consumption was considered to be 3.66 parts for the abatement of 1 part of H_2S. This efficiency was calculated on the basis of the performance of a commercial equipment produced by *Ecochimica System srl*. The reagent is a diluted 30% soda aqueous solution. The estimation of operative costs was then calculated considering principally the reagent cost, the wastewater treatment and the electric energy consumption. The performance of scrubber equipment was not experimentally verified.

From results in Table 6 it is evident that the scrubber system is the cheapest solution from the operative point of view. However, this system requires higher investment costs than the fixed beds of activated carbons. In addition, it is known that this technology does not permit the removal of sulfurated species until the low levels required to prevent the poisoning of the Fuel Cell catalyst [15]. Hence, on the basis of such considerations, we consider that an adsorption stage is essential to obtain the low sulfur concentration required for a MCFC. Thus, the operation cost of a combined treatment using both technologies: a preliminary scrubbing step followed by a treatment on an activated carbon bed, was also calculated. As a conservative approach it was considered the use of the most selective between the two tested activated carbons (ROZ3), even though it is the most expensive material. From the results reported in Table 6 is evident that for such dual approach the operative costs are equivalent to that of the single scrubbing system, which confirm the potentiality of this schema.

Biogas is extensively produced in Italy but at present its main source is from MSW landfills. Even though, political indications favour the adoption of organic waste treatments by anaerobic digestion. In addition, the operation costs of an activated carbon bed depend on the H_2S concentration in the biogas [20]. Hence, for low concentrations of sulfur compounds, the adsorption by AC (using a mixed bed reactor) is the optimal cost-effective solution, considering the low investment costs of this technology. This solution can be applied in anaerobic digestion plants where the outlet H_2S concentration is lower than 5 ppm, due to the uses of biological abatement systems. Instead, for the purification of landfill biogas, which is generally characterized by high pollutant concentrations (up to 400 ppm for H_2S), we believe that the best compromise between efficiency and operative costs can be obtained using a desulfurization system composed of a first scrubbing stage followed by an activated carbon fixed bed. However, some consideration about the life of the landfill should be made. The hydrogen sulfide concentration in a closed landfill decreases in time to less than 80 ppm. Thus, the high investment cost of a scrubber system would only be justified if this is implemented in a landfill in operation with high authorized waste disposal volume.

4. Conclusions

Two commercial activated carbons have been tested for the desulfurization of biogas extracted from the Montescarpino MSW Landfill in Genoa, Italy. The adsorbents were tested on their own and in series in different proportions and resulted in different sulfur absorption capacities.

The application of the two different activated carbons in series has demonstrated a cost-effective advantage, hence it has been determined that the optimal loading for the best treatment performance and operative costs of both materials is (70wt% AC RB4W–30wt% AC ROZ 3). This solution can be applied in anaerobic digestion plants where the outlet H_2S concentration is lower than 5 ppm, due to the uses of biological abatement systems.

Nevertheless, we believe that for the application of landfill biogas in fuel cells systems, a two stage purification system constituted by a first scrubbing stage followed by an activated carbon fixed bed would be the most economic and efficient solution, if it is implemented in landfills in operation with high authorized waste disposal volumes.

Studies are now in progress to analyze and compare the performances of other materials with the two AC considered in this paper. New insights are expected to be achieved by this mean.

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