H_2S adsorption from biogas with thermal treatment residues

GASQUET Valentine[1], KIM Boram[1], SIGOT Léa[2], BENBELKACEM Hassen[1]

[1] Univ Lyon, INSA Lyon, DEEP (Déchets Eaux Environnement Pollutions), EA 7429, 69621 Villeurbanne cedex, France

[2] Laboratoire Réactions et Génie des Procédés, CNRS, Université de Lorraine, 1 rue Grandville, F-54001 Nancy, France

Presenting author email: valentine.gasquet@insa-lyon.fr

Abstract

Biogas cleaning is a fundamental step before its exploitation, to take off potential pollutants in particular hydrogen sulfide (H_2S) from the gas and therefore to protect downstream facilities while reducing toxic emissions in the atmosphere. The use of alternative materials for biogas treatment, in addition of the positive impact on the environment, can reduce the costs of biogas as an energy vector and enable its development.

The aim of the presented work is to assess the H_2S adsorption efficiency with different types of thermal treatment residues: a biochar, a biomass ash, a municipal solid waste incineration (MSWI) bottom ash and an incinerated sewage sludge. H_2S-adsorption experiments were realized with a real landfill biogas. All materials were characterized before and after adsorption in order to evaluate their physicochemical properties related to their reactivity.

The results showed that biochar, biomass ash and MSWI bottom ash are efficient adsorbents as they can retain more than 120 mg of H_2S per gram of dry matter, despite these materials have very different features. Biomass and MSWI ashes are basic, humid and mineral materials, whereas biochar is dry, mainly organic and very porous. On the contrary, incinerated sewage sludge can adsorb only a small amount of H_2S under tested experimental conditions, underlining the importance of the porosity of materials for sufficient H_2S adsorption.

Key words:

Biogas purification – Hydrogen sulfide – Adsorption – Alternative materials

1. Introduction

Hydrogen sulfide (H_2S) removal from gaseous mixtures is an important and current issue; because being produced by several industries (paper-mill, petroleum refining, food processing, etc.). It is a corrosive and flammable compound, toxic for human being and the environment. It can also be found in renewable gases such as biogas. It is therefore mandatory to remove H_2S before the gas exploitation in order to avoid facilities corrosions and not lead to SOx production during combustion [1].

Currently, several solutions exist for H_2S removal from biogas, depending on the biogas origin, the pollutant concentration and the available spaces on site. It is indeed possible to oxidize H_2S into elemental sulfur using microorganisms [2], to wash the gas with a basic solution [3] or to adsorb H_2S on activated carbons [4] or ferric oxides [5]. Adsorption mechanisms seem promising for extracting H_2S from a gas phase as this solution is adjustable to H_2S variations and does not require long maintenance. However, adsorbents such as impregnated activated carbons have a relatively high cost and their use is not environmentally sustainable. Moreover, a life-cycle assessment of different desulphurization solutions showed that the worst scenario for the category “climate change” is the one using impregnated activated carbon because of its production and activation processes [6]. Besides, used carbons must be landfilled. In order to reduce the amount of landfilled waste, it was thought to use alternative materials (that is to say materials which were already used for another goal) as adsorbents. From a decade, many articles have dealt with H_2S adsorption on alternative materials. Attention was particularly focused on thermal treatment residues such as biochars [7], municipal solid waste incineration (MSWI) bottom ash [8] or pyrolyzed sewage sludge [9]. While H_2S adsorption mechanisms on activated carbons have been widely studied [10] [11], the mechanisms occurring with thermal treatment residues are not well understood yet .

Furthermore, most of experiments in the literature dealing with adsorbents used for biogas cleaning, were done in laboratory with synthetic biogas. It makes difficult extrapolation of the results for industrialization of the process. Otherwise, operational conditions, namely the gas flow, the reactor size, the experiment duration, etc., are very different depending on the studies. Therefore, removal capacities comparison does not make much sense with present studies.

This study aims to compare the H_2S adsorption efficiencies of different thermal treatment residues under the same experimental conditions, using a real landfill biogas as well as to understand H_2S retention mechanisms through a characterization of the materials before and after adsorption.

2. Method and material

2.1. Material selection

For this work, four materials have been selected a biomass ash (BA), a municipal solid waste incineration bottom ash (MSWI BA), a biochar (BCH) and an incinerated sewage sludge (ISS).

Biomass ash came from a wood-fired heating plant, burning woodblocks. MSWI bottom ash came from a municipal solid waste incinerator[12]. The input waste of this incinerator were drop-off center refuse, common industrial waste and bulky waste. Biochar was obtained by pyrolysis of organic matter. This thermal treatment takes place in a pyrolyzer, without air. Carbon-rich residues are obtained, due to the absence of oxidation of organic matter. Incinerated sewage sludge came from a wastewater treatment plant. This ash was obtained by incineration of urban sewage sludge, previously partially dried, in a fluidized-bed and separated from smoke with an electrostatic precipitator or a bag filter.

2.2. Experimental set-up

Adsorption tests were performed with a real biogas, on a French landfill. The small-scale pilot was placed near the cleaning and exploitation platform, in a bungalow. In order to keep a controlled temperature during all experiments, an electric heating and an air conditioning system were present in the bungalow. Two stainless reactors, with a height of 24.5 cm and a diameter of 4 cm, are positioned in parallel (Figure 1~~Figure 1~~). Biogas is pumped from the main pipe and separated into two paths. For each one, biogas is humidified by bubbling it in a vial containing demineralized water. The gas flow was initially set at 1 L/min and continuously measured with a Yokogawa flowmeter. Biogas circulated through the bed from the bottom to the top. A good diffusion was ensured with Raschig rings present below the material. Metallic grids were positioned at each end of the reactor to keep the material particles inside the reactor. Before and after the reactor, biogas composition and temperature were measured continuously and saved on a data acquisition card EasyIO GC-32 connected to a computer. A software had been developed to monitor remotely all measured data. The biogas analyzer is a laser infrared spectrometer called ProCeas®. It uses an innovative technique based on Optical Feedback-Cavity Enhanced Adsorption Spectroscopy (OFCEAS). It had been calibrated CH_4, CO_2, O_2, H_2O and H_2S. The measurement range for this last compound is 0 to 10,000 ppm_v, with a relative accuracy of 4%.

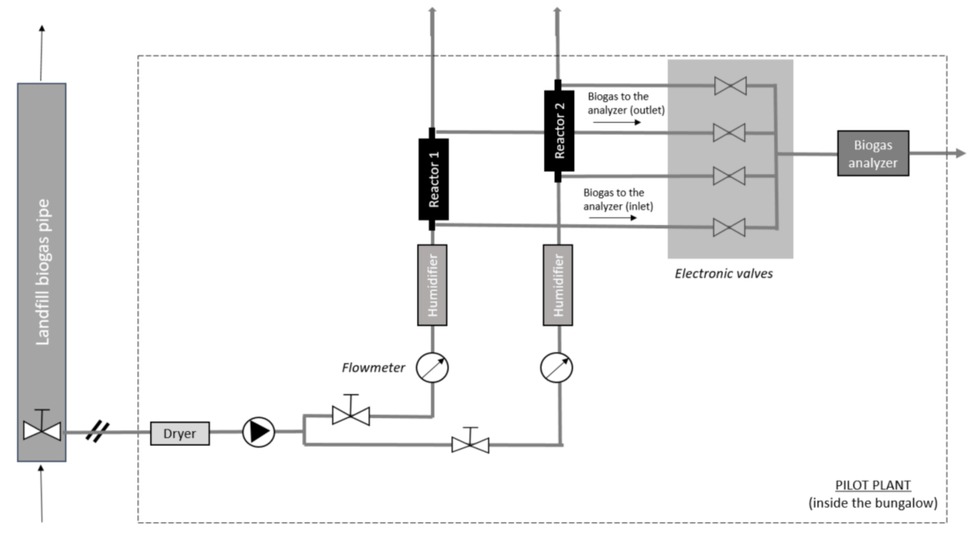

Figure 1: Pilot plant diagram

2.3.Removal capacity assessment

Using the collected data, some parameters can be calculated. The calculation formulas to assess removal capacities of studied materials are presented in Table 1~~Table 1~~.

Table 1 : Formulas used for adsorption parameters calculations

Retention rate (%)	$\boldsymbol{\tau_{ret} = \frac{[H_2S]_{inlet} - [H_2S]_{outlet}}{[H_2S]_{inlet}} \times 100}$
Removal efficiency (%)	$\varepsilon_{removal} = \frac{H_2S_{capt}}{H_2S_{pass}}$
Adsorption capacity (mg$_{H2S}$/g$_{MS}$)	$C_{ad} = \frac{H_2S_{capt}}{m_{dry\ material}}$

H_2S_{pass} and H_2S_{capt} are respectively the accumulated amount of H_2S passed through the reactor and the accumulated amount of H_2S captured by the material since the beginning of the experiment. These parameters are calculated for each temporal point and the calculations have been automatized with the software R in order to facilitate data processing. The final values of the parameters mentioned above are observed in order to compare the materials.

2.4. Materials characterization

Selected materials have very different features, discernible at macroscopic scale (color, particle size, density, etc.). Some of these parameters can directly influence H_2S removal capacities of the materials.

2.4.1. Physical characterization

Surface areas and porous volumes were measured at LRGP (Laboratory of Reactions and Chemical Engineering) using a device ASAP 2020. Beforehand, each sample, about 100 mg, was degassed under vacuum at a given temperature (usually around 100°C) during more than 24 hours in order to remove all the molecules adsorbed on the material surface. In order to characterize the material porosity, three parameters are observed: the surface area BET, the porous volume and the median diameter of the pores. With the device used for this study, only the porosity from the micropores (below 2 nm) and the mesopores (between 2 and 50 nm) can be measured.

Water content was measured in triplicate with 20-30 grams of sample each time by drying the material at 105°C during 24 hours.

The bulk specific gravities densities of the materials were calculated by measuring the volume of a given mass of compacted sample in a graduated cylinder.

2.4.2. Chemical characterization

pH was measured with a calibrated pH-meter (Consort C3431)after 48h leaching of materials with distilled water following the ANC 14429 norm. The L/S ratio was ten, with 15 g of dry sample and a total volume of 150 mL.

Elementary composition is determined with ICP-AES after sample fusion with $LiBO_2$ followed by dissolution using HNO_3. The major chemical compositions are given in oxides forms: SiO_2, Al_2O_3, Fe_2O_3, MnO, CaO, Na_2O, K_2O, TiO_2, P_2O_5 and Loss-on ignition.

3. Results and discussions

3.1. Raw materials characterization

Thermal treatment residues are complex and not well-known materials. Because of the wide variety of combustion inputs, ashes can have very different features. Moreover, the type of oven and the thermal treatment conditions (oxygen amount, temperature, turbulence and residence time) have an impact on physicochemical properties of ash. An incinerator can produce ash of different qualities over time if its management evolves or if new fuels are burnt. For these reasons, the selected materials were characterized and compared before adsorption.

Major chemical compositions of materials are presented in Figure 2~~Figure 2~~. At first glance, a material significantly stood out from the others, the biochar. Indeed, it was very organic, with a loss-on-ignition (the mass lost after heating at 1000°C during four hours under an oxidative atmosphere) of 97%. Biochar did not contain any silica and only a few amount of metals.

The other materials had more similar elemental compositions, with silica and calcite as main components. They are both inert components, not involved in H_2S adsorption. The materials also contained mineral oxides such as Al_2O_3 and Fe_2O_3. H_2S adsorption can be catalyzed by the presence of these oxides [13]. The high concentration of P_2O_5 in ISS is explained by the presence of phosphor in wastewater.

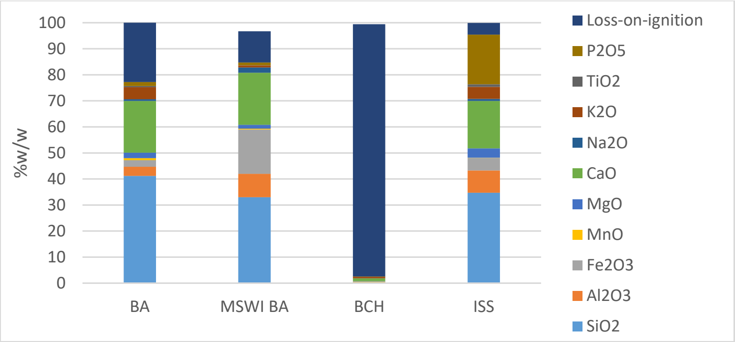

Figure 2: Major elements for the four raw materials: a biomass ash (BA), a MSWI bottom ash (MSWI BA), a biochar (BCH) and an incinerated sewage sludge (ISS)

All the studied materials were basic, with a pH range from 9.4 to 12.4 (**Error! Reference source not found.**~~Table 2~~). It is a common feature of thermal treatment residues due to the presence of lime, potash or carbonates. Based on literature, alkalinity is one of the key-factors for H_2S adsorption. pH must be between 7.2 and 12.9 based on the predominance diagram for H_2S [7]in order to transform dissolved H_2S into HS^- which can be easily oxidized.

Depending on their water contents, the materials could be split into two groups. Biomass ash and MSWI bottom ash were cooled with water quenching that is why they are humid, 38% for biomass ash and 12% for MSWI bottom ash (see Table 2~~Table 2~~). Indeed, at the outlet of the incinerator, the hot ash were placed in a water bath and then stored and drained. Incinerated sewage sludge were separated from dry fumes at the outlet of the fluidized bed with bag filters or electrostatic precipitators. Then, they were stored in a silo without any pre-cooling. It is the reason why this material remain dry. Biochar contained also a low amount of water, in the range of 3-6%.

Biochar was a light material with a bulk specific gravity of 0.1 while BA and ISS had close bulk specific gravity, about 0.7-0.8. MSWI BA was the densest material with a bulk specific gravity of 2.3. The difference of density change the mass of adsorbent in the reactor.

Regarding the results of porosity characterization of materials (Table 2~~Table 2~~), it appears that the biochar was by far the most porous material. Its surface area was close to the one of activated carbons (about 1000m²/g). Consequently, its porous volume was also high, with an influence of both the micropores (volume of 0.315 cm^3/g) and the mesopores (volume of 0.196 cm^3/g).

On the contrary, incinerated sewage sludge was a non-porous material: its surface area and porous volume were very low. Microporous volume was zero, only some mesopores were present. It could be observed with a mean pore diameter much larger compared with biochar, 26 nm for ISS and 1 nm for biochar. Biomass ash had a pore volume five time lower than biochar with a predominance of mesopores (for a volume of 0.090 cm^3/g against 0.026 for micropores). The surface area of MSWI BA was lower than biomass ash one but higher than ISS.

Table 2 : Raw material characterization

Material	**BA**	***MSWI BA***	***BCH***	***ISS***
pH	12.4	12.3	10.1	9.4
Water content (%)	38.0	12.0	4.4	0.3
Bulk specific gravity	0.7	2.3	0.1	0.8
BET Surface are (m^2/g)	32	17	919	3
Mesopore volume (cm^3/g)	0.090	N/A	0.196	0.0021
Micropore volume (cm^3/g)	0.026	N/A	0.315	0.000

3.2. H_2S adsorption

Adsorption tests on thermal treatment residues had been realized in almost same conditions (Table 3~~Table 3~~) for the four thermal treatment residues in order to assess the removal capacity of these materials.

Table 3 : Sum-up of the tests realized

Material	***BA***	***MSWI BA***	***BCH***	***ISS***
Duration (days)	14	11	9	14
Mass of sample (g)	122.6	357	26.7	168.8
Flow (L/min)	1.27	1.17	1	1
Final mg H_2S passed /g DM	233	80	752	222
Final retention rate (%)	40%	35%	11%	10%
Final removal efficiency	71%	50%	19%	9%
Adsorption capacity (mgH_2S/gDM)	173.6	39.9	144.6	20.9

To compare the materials, the mass-weighted amount of H_2S captured by the material was plotted as a function of the mass-weighted amount of H_2S passed in the reactor (**Error! Reference source not found.**~~Figure 3~~). Indeed, the experiments were performed within a constant volume reactor but the density of the materials were very different (see **Error! Reference source not found.**~~Table 3~~). Indeed, 122.6 g of BA, 26.7 g of biochar, 168.8 g of ISS and 357 g of MSWI BA were placed in the same volume of reactors.

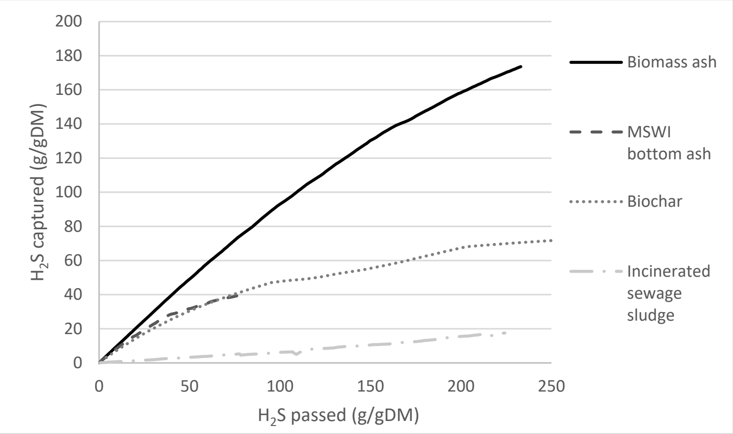

Figure 3: Comparison of alternative materials - Evolution of H2S captured per mass of dry matter as a function of H2S passed per mass of dry matter

The material removing the greatest mass-weighted amount of H_2S was the biomass ash with a final adsorption capacity of 173.6 mg_{H2S}/g_{DM}. Biochar and MSWI BA had very similar adsorption capacities on the first part of **Error! Reference source not found.**~~Figure 3~~ : until 75 mg of H_2S passed in the reactor they both removed 40 mg of H_2S per gram of dry matter. Indeed, the amount of H_2S passed per mass of dry material was far higher for biochar than for MSWI bottom ash although the experiments lasted almost the same time. It is explained by the very low density of biochar. ISS had the lowest adsorption capacity and was not an efficient adsorbent for H_2S removal.

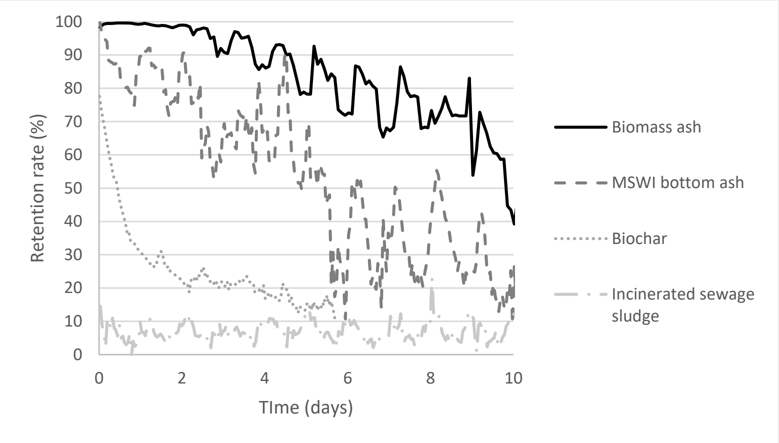

Figure 4: Comparison of alternative materials - Retention rate as a function of time

In order to compare temporal H_2S retention rate between different materials, the ratio between the H_2S concentration at the outlet and at the inlet of the reactor were plotted against time (**Error! Reference source not found.**~~Figure 4~~).

For biomass ash, H_2S retention rate was 100% during the first three days then slightly decreased until 40% at the end of the experiment. MSWI bottom ash had performances lower retention rate than biomass ash. The periodical variations observed for the H_2S retention rate of biomass ash and MSWI BA can be explained by daily temperature variations. Indeed, the biogas water content depends on the gas temperature as the biogas is water-saturated with the humidification vial. When the temperature is increasing, for a relative humidity of 100%, the absolute humidity is also increasing. Water is known as a key-factor for H_2S adsorption[4] [14].

On **Error! Reference source not found.**~~Figure 3~~, it seems that biochar and MSWI BA had similar efficiencies for H_2S adsorption. However, biochar's retention rate was different from BA and MSWI BA: it decreased quickly during the first days and then stabilizes around 20%. This material can therefore not be used as an adsorbent for H_2S removal because of the slump of retention rate. Furthermore, incinerated sewage sludge appears to be non-efficient adsorbent under these experimental conditions, as the retention rate was always below 15%.

3.3. Used materials characterization

After H_2S adsorption, pH had been decreased for all thermal treatment residues as expected. For biomass ash, MSWI BA and incinerated sewage sludge, the pH decreased to 7-8 while it reached to 2 for biochar. For the first group of materials, this acidification can be explained by the carbonatation: the adsorption of carbon dioxide leading to the formation of calcium carbonate within materials. As biochar contains only few amount of calcium, no carbonates could be produced. However, H_2S is an acid component. Its adsorption at the surface of a material can drastically reduce its pH because no component can act as a buffer.

The water contents of materials evolved in different ways depending on the initial value. Indeed, raw biomass ash and MSWI bottom ash are initially humid and they begin dryer after adsorption (from 38.0% to 27.1%, from 12.0% to 4.0%, respectively). It means that the biogas becomes dryer when it passes through the reactor. For the two other materials, they begin more humid after adsorption from 4.4 to 10.8% for BCH and from 0.3 to 0.8% for ISS; the biogas gives a part of its water to the material.

Table 4 : Used materials characterization

Material	***BA_H2S***	***MSWI-BA_H2S***	***BCH_H2S***	***ISS_H2S***
pH	8.4	8.1	2.1	7.4
Water content (%)	27.1	4.0	10.8	0.8
BET Surface are (m^2/g)	9	N/A	66	2
Mesopore volume (cm^3/g)	0.045	N/A	0.095	0.021
Micropore volume (cm^3/g)	0.000	N/A	0.007	0.000

After H_2S adsorption, microporous volume dramatically decreased for biochar (from 0.315 to 0.007), more slightly for BA and does not change for ISS, as observed in Table 4~~Table 4~~. Micropore volume is almost equal to zero for all the materials after adsorption. The micropores may be the ones most implied in H_2S adsorption. Surface area followed the same trend as porous volumes.

3.4. Discussions

From adsorption tests, it appeared that, in our experimental conditions, ISS is not a suitable adsorbent for H_2S removal. Indeed, the very low (but not null) retention rate remained quite constant during all the experiment. At first glance, it was surprising because ISS has an elemental composition close to the

BA and MSWI BA which can adsorb efficiently H_2S. Based on the physical characterization, this low performance can be explained by the absence of microporosity and the very low mesoporosity within ISS. The surface area of this material is equal to 2 m^2/g while an activated carbon (a widely used adsorbent) has surface area higher than 1000 m^2/g. Therefore, porosity can be the limiting factor.

As explained in erstwhile studies[10, 11], H_2S adsorption mechanism is a mixing between physisorption (i.e. physical adsorption due weak intermolecular interaction[15]) and chemisorption (chemical compounds formation). H_2S molecules must be first captured at the surface of the adsorbent (thanks to a large porosity) and dissociated into HS^-. The pH must be alkaline in order to dissociate H_2S into HS^- [7]. After that, this ion can react with oxygen (initially present in biogas), leading to the oxidation of HS^- and the formation of elemental sulfur. This oxidation can be favored by the presence of mineral oxides such as ferric oxides[13]. During this catalysis, metal sulfide is formed and then oxidized by oxygen to form elemental sulfur and regenerate metal oxide.

For ISS, the very low porosity decreases the surface for H_2S adsorption and dissociation and therefore the reaction between HS^- and oxygen can hardly occur. A way to overcome this problem can be to reduce the biogas flow in order to increase the retention time in the reactor and let more time for oxidation reaction to take place.

Concerning biochar, the high porosity enables a good physisorption of H_2S molecules in the material. However, the absence of mineral oxides strongly limits the chemisorption and the oxidation of the HS^- ions. Biochar has also a very low density and the amount of material in the reactor is more than five times lower than the other materials. It is the reason why the adsorption capacity in mg of H_2S per gram of dry matter is so high.

Biomass ash had the best removal capacity probably because of its porosity, even if not as high as traditional adsorbent, and the presence of mineral oxides for the catalysis of HS^- oxidation. The presence of a very large porosity is therefore not necessary for H_2S adsorption even if it appears that the highest the porosity, the best adsorption capacity for materials with almost the same composition (BA, MSWI BA and ISS). Otherwise, MSWI bottom ash was quite similar to biomass ash but was less efficient for H_2S removal. More investigations and adsorbent characterization will be necessary to identify the differences between these two materials and understand completely the adsorption mechanisms.

4. Conclusions

This work firstly enabled to assess H_2S adsorption capacities of different thermal treatment residues under comparable experimental conditions and correlate them to physicochemical properties of studied materials. Incinerated sewage sludge was a non-porous and dry material and it contained mineral oxides. It could not be used as an adsorbent for H_2S adsorption from biogas in our experimental conditions. Biochar was a very porous material but not, however, a better adsorbent because of the absence of catalyzer agents. Biomass ash could efficiently remove H_2S from biogas. The presence of mineral oxides associated with sufficient porosity in the thermal treatment residues seem to be mandatory for a good H_2S removal. MSWI bottom ash can adsorb H_2S but not as efficiently as biomass ash.

Acknowledgements

The authors are grateful for the financial support provided by INSA Lyon. They also thank Hervé Perier-Camby and Richard Poncet (DEEP laboratory– INSA Lyon, France) for their technical support.

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