ADSORPTION OF H'S ON RESIDUAL BIOMASS GASIFICATION CHAR

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ABSTRACT: Char is the solid by-product of biomass gasification. Plant owners usually dispose of it at a cost, but its high carbon content and surface area could make it suitable for further applications. In this work, we studied its potential to adsorb hydrogen sulphide (H_2S), a common pollutant, e.g. present in the producer gas of gasification, as well as in biogas from anaerobic digestion. We tested different samples of char collected from commercial gasification plants in South Tyrol (Italy). The adsorption was reproduced in a lab-scale fixed bed reactor. The results highlights that all samples could capture hydrogen sulphide, showing different adsorption performance. The surface area of the char and the ash amount seem to affect the removal capacity, although other properties of the materials are probably important. The inlet concentration of hydrogen sulphide does not influence the adsorption capacity significantly.

Keywords: adsorbent, by-product valorization, fixed bed, gas cleaning, gaseous biofuel, hydrogen sulphide.

1 INTRODUCTION

The gasification of biomass is one of the most promising options to generate energy in a sustainable way. It takes place putting biomass in contact with an amount of an oxidising agent that is less than the one required for stoichiometric combustion, at high temperature. The main product of the process is producer gas, a gaseous mixture rich in hydrogen and carbon monoxide. The producer gas can be converted into energy or chemicals through various technologies.

Gasification is an established technology, with several commercial plants already built. In the alpine region of South Tyrol, 46 plants are currently operating (Fig. 1). Nonetheless, some aspects of the gasification process still need to be optimised [1]. Among these, char production greatly affects the economics of the process. Char is a solid carbonaceous material produced due to the incomplete carbon conversion. Its amount usually ranges between 2% and 5% of the fed biomass [2] and plant owners must dispose of it at a cost. In South Tyrol, about 1500 tons of char are produced every year [3], with an associated cost for the disposal of about 150 €/ton [4].

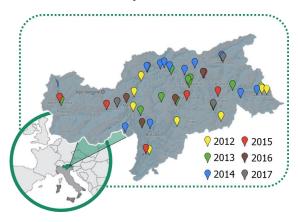


Figure 1: Gasification plants in South Tyrol

Several research groups have studied the properties of the residual char from biomass gasification [5–7], highlighting notable differences depending on the biomass feedstock, implemented technologies and operating conditions. Most samples exhibited a very

porous structure and high surface area, and these properties could make them suitable as adsorbents. In some cases, their features are similar to those of activated carbons, even without a further activation step.

Recent studies have investigated the use of char as adsorbent for air and water pollutants, as adsorbent for CO₂ sequestration, or as catalyst for tar cracking applications. However, most of the research has been carried out on pyrolytic char. Focusing on gasification char and establishing a way to further employ it, the whole gasification process would notably benefit, avoiding the cost and impact of the disposal of a waste.

Adsorbents are used in a wide variety of processes, usually with the aim of selectively removing harmful or valuable compounds from a gaseous stream. Adsorption is a common option in the clean-up of the producer gas and other biomass-derived gaseous fuels, such as the biogas from fermentation. In particular, adsorption can remove hydrogen sulphide (H2S), one of the most abundant detrimental compounds of these gases. In producer gas, it can be found in concentrations from 100 to 3000 ppm [8]. H₂S is highly toxic and corrosive, and all the devices producer gas is fed to, require a significant decrease of its concentration. The strictest case is represented by fuel cells: some of them are rapidly damaged if the fed fuel contains more than 0.5 ppmv of H₂S [9]. Such a purity in the gas is hardly achievable employing cleaning techniques different from adsorption, which is also easy to manage and thus particularly suitable for small-scale applications.

Different H₂S adsorbents have been studied in literature, including metal oxides [10–12], zeolites [13,14], graphene [15], and sepiolite [14]. Activated carbons have been the focus of several studies [14,16–20]: they can effectively capture high amounts of H₂S and they can also be easily produced from renewable sources. Finally, recent studies assessed the suitability of waste materials that feature similarities to the aforementioned adsorbents: this is the case of municipal solid waste incineration fly ashes [21] and waste pyrolysis char [22].

The aim of this work is to assess the efficacy of the residual char from biomass gasification to adsorb H_2S . This could thus represent a useful and simple way to valorise it within the process that produces it, or for other applications. To the knowledge of the authors, no study

of H₂S adsorption on gasification char exists in literature.

2 MATERIAL AND METHODS

2.1 The employed chars

The experiments verified the H2S adsorption capacity five char samples. Four of them, previously characterised [5], were collected from commercial plants of the South Tyrol region, Italy. Another one [23] was obtained from a pilot plant that employs a spouted bed reactor as gasifier [24]. Finally, two different commercial activated carbons, already available in our laboratory, were also employed for comparison purposes. The first activated carbon is Norit GSX. It is obtained from the carbonisation of peat at 500 °C, then it is activated with steam at about 1000 °C and washed with hydrochloric acid [25]. This makes it particularly suitable for catalytic applications. The second activated carbon, Norit CA1, is obtained from wood and is chemically activated with phosphoric acid [26]. Norit CA1 is reportedly suitable for the purification of highly coloured and foaming liquids for foods and beverages. All the materials are fine powders.

The properties of these materials are summarised in Table I, II and III.

Table I: List of sampled materials

| Sample | Reactor technology | Feedstock | Gasifying agent | T [°C] |
|--------|--------------------|--------------|-----------------|--------|
| char-A | dual-stage | wood chips | air | 900 |
| char-B | downdraft | wood chips | air | 800 |
| char-C | rising co- | wood pellets | air | 700 |
| | current | | | |
| char-D | downdraft | wood chips | air | 800 |
| char-E | spouted bed | wood pellets | air | 880- |
| | | | | 900 |
| AC-1 | n/a | peat | n/a | n/a |
| AC-2 | n/a | wood | n/a | n/a |

Table II: Elemental composition of the sampled materials (dry, wt. %).

| Sample | C | Н | N | S | O | Ash |
|--------|-------|------|------|------|-------|-------|
| | [%] | [%] | [%] | [%] | [%] | [%] |
| char-A | 78.09 | 0.37 | 0.18 | 0.31 | 6.43 | 14.62 |
| char-B | 80.64 | 0.55 | 0.22 | 0.20 | 2.59 | 15.80 |
| char-C | 80.23 | 0.49 | 0.23 | 0.28 | 2.69 | 16.08 |
| char-D | 48.12 | 0.49 | 0.23 | 0.32 | 1.32 | 49.52 |
| char-E | 49.90 | 0.75 | 0.12 | 0.32 | 0.22 | 48.69 |
| AC-1 | 90.65 | 0.39 | 0.46 | 0.33 | 3.71 | 4.46 |
| AC-2 | 82.42 | 1.99 | 0.19 | 0.21 | 12.59 | 2.60 |

Table III: S_{BET} , pore volume and pore diameter of sampled chars and activated carbons.

| Sample | SBET | Pore volume | Pore diameter |
|------------|-----------|----------------------|---------------|
| ~ <u>F</u> | $[m^2/g]$ | [cm ³ /g] | [nm] |
| char-A | 586.72 | 0.30 | 3.88 |
| char-B | 281.23 | 0.23 | 5.22 |
| char-C | 127.67 | 0.28 | 7.08 |
| char-D | 77.90 | 0.08 | 8.58 |
| char-E | 103.97 | 0.14 | 5.18 |
| AC-1 | 1002 | 0.51 | 6.10 |
| AC-2 | 1269 | 0.91 | 5.20 |

2.2 Experimental setup

All the adsorption experiments were performed in the Biofuels and Bioenergy laboratories of the Free University of Bozen-Bolzano.

The adsorption took place into a quartz tubular fixed bed reactor, with length 600 mm and diameter 8 mm. To keep the sample stable, quartz wool was placed above and below it.

We prepared the gaseous feed with a tank of pure nitrogen and a tank containing a mixture of 1000 ppmv of H_2S in nitrogen, both connected to a mixer that can control and mix the two flows.

The composition of the gas at the exit of the reactor was measured by a micro GC system (Agilent 490 Micro GC).

All tubes and connections were in either quartz, PTFE or stainless steel treated with Sulfinert: these materials are inert towards H_2S , and hence did not adsorb it

The entire plant is schematised in Fig. 2 and the reactor is depicted in Fig 3.

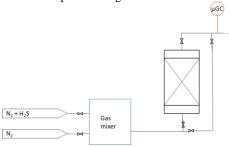


Figure 2: Scheme of the adsorption test rig



Figure 3: Char (above) and quartz wool (below) inside the quartz reactor

2.3 Experimental procedure

Prior to testing their adsorption capacity, char and activated carbons samples were degassed and dried at 105 °C overnight. Inside the reactor, we put a certain amount of material, so that the height of the fixed bed was approximately 2.5 cm. This corresponded to 150 to 200 mg of material, depending on the employed material.

The total flow of the gas was set to 100 Nml/min, which corresponds to a velocity of 3.33 cm/s in empty column. This is inferior to the typical values of industrial-scale processes, which range between 14 and 17 cm/s [22]. This way, we avoided the formation of excessive pressure drops, given that the chars were composed of extremely fine particles.

Table IV summarises the operative conditions chosen for the experiments.

Table IV: Operative conditions of the experiments.

| Variable | Value | | |
|--|--|--|--|
| Height of the fixed bed | 2.5 cm | | |
| Mass of quartz wool | 400 mg | | |
| Total gas flow rate | 100 Nml/min | | |
| Inlet concentration of H ₂ S (in N ₂) | 250 ppmv to study the adsorption capacity 250 to 1000 ppmv to study the influence of the H₂S inlet concentration | | |
| Temperature | Ambient | | |
| Pressure | Atmospheric | | |

At the beginning of each test, we let pure nitrogen (N_2) flow into the reactor, in order to let all the oxygen out, and then we started feeding the H_2S-N_2 mixture. The composition of the outflow gas was continuously monitored, and the experiment was stopped when the measured concentration of H_2S exceeded a value equal to 95% of the inlet concentration. We performed each experiment at least twice.

After the analyses, we measured the final composition of the char sample with a Vario MACRO Cube Elementar Analyzer, in order to detect the increase in the sulphur content.

Thanks to the continuous monitoring of the outlet concentration, it was possible to calculate the quantity of H_2S that each char sample adsorbed in the experiments. We numerically calculated the following integral, based on the mass balance of the reactor and on the ideal gas law:

$$m_{ads} = \frac{M \cdot P \cdot Q}{R \cdot T \cdot m_{char}} \int_{o}^{t_{fin}} (c_{in} - c_{out}) dt$$

in which:

- m_{ads} is the adsorbed amount of H₂S per gram of adsorbent [mg/g];
- M is the molar mass of H₂S (34.0818 g/mol);
- P is the pressure $(1.0315 \cdot 10^5 \text{ Pa})$
- Q is the total gas flow rate, hypothesised to be constant throughout each analysis since the variation due to the H₂S adsorption is negligible [ml/min];
- R is the gas constant (8.314 J/(mol·K));
- T is the absolute temperature [K];
- m_{char} is the mass of char employed in the test
 [g]:
- t_{fin} is the duration of the experiment [min];
- c_{in} and c_{out} are the concentrations of H₂S at the inlet and outlet of the reactor, respectively [-].

3 RESULTS AND DISCUSSION

3.1 Adsorption capacity

The first set of experiments allowed us to calculate the maximum H_2S adsorption capacity of each sample. Table V reports the figures.

Table V: H_2S adsorption capacities of the studied chars and activated carbons.

| Sample | Adsorption capacity [mg/g] |
|--------|----------------------------|
| char-A | 6.88 ± 0.37 |
| char-B | 5.41 ± 0.26 |
| char-C | 5.38 ± 0.57 |
| char-D | 2.77 ± 0.08 |
| char-E | 1.61 ± 0.02 |
| AC-1 | 2.35 |
| AC-2 | 2.61 ± 0.05 |

These results confirm that all the studied chars were able to capture H_2S , with different efficiency depending on their properties.

The activated carbons (samples AC-1 and AC-2) performed poorly and adsorbed less H2S than most of the chars. This might be caused by the activation process, which makes these activated carbons particularly suitable for applications different from gas adsorption, as their specifications report. In fact, this is not in contrast with results obtained in a previous study [14], in which a type of activated carbon adsorbed only 1.71 mgH2S/g before the breakthrough, much less than the amount other types were able to remove (up to 27.15 mg_{H2S}/g). In that case, the reason was identified in the fact that the best performing activated carbons were impregnated with copper and chromium salts or potassium hydroxide. This indicated that the removal was not only promoted by micropores, but also by chemical reactions such as sulphide salts formation or acid-base neutralisation. As regards the experiments performed in the present study, we did not pre-treat the chars with chemicals. However, all the chars feature a much higher percentage of ash, and we speculate that they might contain compounds that enhance adsorption. Indeed, our research group already detected the presence of heavy metals in a type of char in a previous study [27].

Fig. 4 shows a plot in which the specific adsorption capacity of each sample is related to its surface area. For the chars, higher values of surface area seem to affect adsorption positively. However, the trend is not always increasing and it is likely that the adsorption capacity also depends on other factors. The two activated carbons feature a much higher surface area, but their adsorption capacity is lower than the best performing chars.

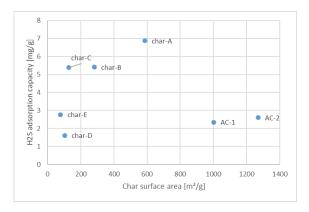


Figure 4: Dependency of the adsorption capacity of char and activated carbon samples on the BET surface area.

Looking at the graph, the fact char-C adsorbs about twice the amount of chars D and E and about the same as

chars A and B is hardly understandable by a simple comparison of the material properties that have been studied. The same can be said about the better performance of char-D compared to char-E, despite its lower surface area. Clearly, more in-depth analyses are required to identify the most relevant property affecting the adsorption capacity.

The specific adsorption capacity can also be related to the ashes content, as shown in Fig. 5. Even in this case, there is no overall trend, but it seems that a very high or very low amount of ash corresponds to a decrease in the adsorption capacity. Nonetheless, it is likely that the composition of the ash has a role, in addition to the amount.

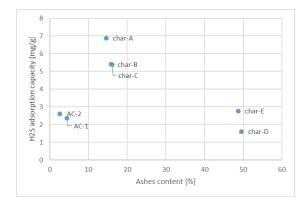


Figure 5: Dependency of the char adsorption capacity on the ash content.

The values of adsorption capacity are anyway interesting and promising for a real scale application of these materials, especially when compared with literature data, summarised in Table VI.

Table VI: Adsorption capacities of some materials from literature.

| Material | Adsorption | Reference |
|--|-----------------|-----------|
| | capacity [mg/g] | |
| AC RGM1 | 27.15 | [14] |
| AC RBAA1 | 20.43 | [14] |
| Alumina Galipur | 1.56 | [14] |
| AC RB1 | 1.71 | [14] |
| Zeolite ATZ | < 0.1 | [14] |
| Sepiolite | < 0.1 | [14] |
| Char from pyrolysis of used wood | 0.04 | [22] |
| pellets | 0.01 | [22] |
| same after activation | 12.92 | [22] |
| Char from pyrolysis of food waste and coagulation- | 0.22 | [22] |
| flocculation sludge - same after activation | 66.60 | [22] |
| AC Desotec Airpel Ultra DS | Up to 34.87 | [28] |
| Carbon fibers | 800 | [19] |
| Potassium hydroxide AC | 65 | [29] |
| Copper impregnated AC | 46.3 | [30] |
| | | |

Most of the reported activated carbons, either commercial or synthesised by the authors, can adsorb more H_2S than the studied chars. However, in the present moment, the gasification chars are a waste: not only they have no cost, but also they represent a cost for the plant owners. Moreover, several of the materials in Table VI underwent activation phases, in some cases even with chemical compounds, which increased their efficiency. Instead, the chars we studied were able to adsorb H_2S in the form at which they left the plant, only after a degassing phase, and their capacity was higher than the one reported for untreated pyrolysis chars [22]. Pyrolytic chars reportedly perform better only after activation with steam at 850 °C.

3.2 Influence of the H₂S inlet concentration

Char-A was the sample with the highest adsorption capacity, hence we chose it for further experiments.

We performed tests with different inlet concentrations of H_2S , to assess the influence on the process.

Fig. 6 depicts three representative breakthrough curves for the three studied inlet concentrations. As expected, a higher concentration saturates the adsorbent faster, so the outlet concentration becomes equal to the inlet one in a shorter time.

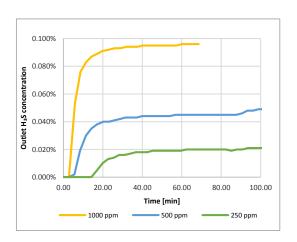


Figure 6: Breakthrough curves for different inlet concentrations of H_2S

Table VII reports the saturation adsorption capacities for the three inlet concentrations. It does not show a clear trend. It can be concluded that, in the studied interval, the inlet concentration of H_2S does not significantly affect the maximum adsorption capacity.

Table VII: Maximum adsorption capacity for different inlet concentrations of H₂S

| Inlet concentration of H ₂ S [ppmv] | Maximum H ₂ S adsorption capacity [mg/g] |
|--|---|
| 250 | 6.88 ± 0.37 |
| 500 | 7.87 ± 0.70 |
| 1000 | 6.98 ± 0.24 |

3.3 CHNS results

After the adsorption experiments, the used char underwent CHNS analyses, to detect any changes in their composition. Unfortunately, quartz wool used in the experiments heavily polluted the samples, so the results

were inaccurate and performing a mass balance was not possible. Despite this, the results permitted to draw some qualitative conclusions. Table VIII shows the results of the analyses.

Table VIII: Results of the CHNS analyses on the used chars after H_2S adsorption (wt. %).

| Sample | C [%] | H [%] | N [%] | S [%] |
|----------------------|-------|-------|-------|-------|
| char-A (250 ppm) | 44.63 | 0.05 | 0.09 | 0.53 |
| char-A (500 ppm) | 52.05 | 0.16 | 0.12 | 0.48 |
| char-A (1000 ppm) | 60.12 | 0.19 | 0.15 | 0.51 |
| char-B | 76.95 | 0.48 | 0.28 | 0.58 |
| char-C | 81.10 | 0.59 | 0.31 | 0.63 |
| char-D | 42.43 | 0.29 | 0.22 | 0.82 |
| char-E | 52.14 | 0.49 | 0.29 | 0.38 |
| AC-1 | 85.47 | 0.30 | 0.41 | 0.84 |
| AC-2 | 70.21 | 2.40 | 0.15 | 0.28 |
| | | | | |

Fig. 7 reports the variation in carbon and sulphur content for each material. It can be seen that the carbon content differs from the original value, mostly with a decreasing trend that is probably caused by the quartz wool. Instead, the sulphur content presents an opposite trend and increases in all cases. This can confirm that sulphur is adsorbed onto the char and activated carbon surface.

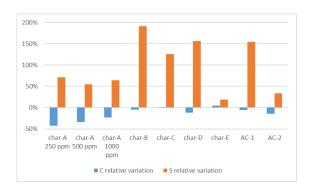


Figure 7: Relative variation of C and S after the experiments.

Finally, the increase is roughly constant in the three tests performed with char-A, confirming that the inlet concentration of H_2S did not affect the adsorption capacity.

3.4 Pressure drops

Char is a very fine material. A zeta-analysis on char-A reported an average diameter of about 50 µm. With a density of 480 g/l, the char belongs to the Group C (or "cohesive") of the Geldart classification [31].

With the aid of a manometer, we analysed the pressure drops that arose in the reactor containing a bed of char-A.

When the mass of char was 100 mg and the bed height about 1.25 cm, the pressure drop was lower than 10 kPa for a flow up to 650 Nml/min. Then, it increased to 30 kPa for a flow of 950 Nml/min.

With a double amount of char, the pressure drop was lower than 10 kPa for a flow up to 350 Nml/min. Then, it

increased to 32 kPa for a flow of 550 Nml/min. A further increased in the flow made the bed unstable.

These observations confirmed that the very fine nature of the studied chars might hinder their application. High pressure drops can arise, and channelling or dragging phenomena may take place. Increasing the size of these materials could enhance the feasibility of their use.

4 CONCLUSIONS

In this work, we studied the adsorption of hydrogen sulphide (H_2S) on residual char from biomass gasification and on activated carbon. We employed five different samples of biomass char, (collected from either commercial or pilot plants), and two commercial activated carbons available in our laboratory.

All the studied chars were able to adsorb H_2S , with better performances than the two activated carbons. The maximum adsorption capacity was 6.88 mg of H_2S per gram of char. However, a comparison with literature data showed that the adsorption capacity is usually lower than that of activated carbons, most of which are chemically activated. On the other hand, the adsorption capacity is higher than that of untreated pyrolysis chars.

The total adsorption capacity seems to be related to the specific surface area of the char, although the relation is not linear and other factors most likely affect the phenomenon. The ash content seems to influence the adsorption capacity, with very high or low percentages of ash hindering it. However, it is likely that the composition of the ash is relevant to the efficacy of the adsorption process.

For the studied range, the inlet concentration of H₂S affects the speed of the process, but not the saturation.

The adsorption was also confirmed through CHNS analyses, which highlighted an increase in the sulphur content even though the samples were heavily polluted by quartz wool.

Further studies might help to understand the phenomenon: it is still unclear which property of the material majorly affects the total adsorption capacity. Other operative conditions could be studied, including non-ambient temperatures, and the adsorption of H2S in a test or real producer gas (or biogas) instead of pure nitrogen. Furthermore, the performance of the char could be increased through either thermal or chemical activation, and its particle size might be increased to avoid great pressure drops and hence enhance its usability. Nonetheless, these preliminary results are promising and confirm that this by-product could be useful even within the process that produces it. Plant owners could avoid the costs for its disposal and those for the purchase of a commercial $H_2\hat{S}$ adsorbent, thus following the principles of the circular economy and reducing the environmental impact of the whole gasification process.

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6 ACKNOWLEDGEMENTS

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