



# Sustainable activated carbons of macroalgae waste from the Agar–Agar industry. Prospects as adsorbent for gas storage at high pressures



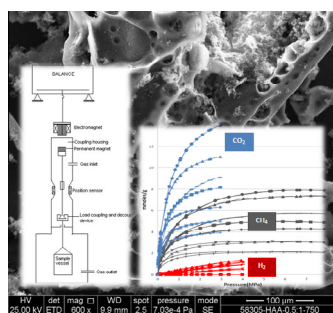
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## HIGHLIGHTS

- ACs are obtained by conventional and microwave activation using KOH and  $K_2CO_3$ .
- High-pressure adsorption of  $CO_2$ ,  $CH_4$  and  $H_2$  in ACs obtained was measured.
- The adsorption capacities of the gases on ACs obtained follow the order:  $CO_2 > CH_4 > H_2$ .
- ACs from macroalgae waste may be a new material for gas storage.

## GRAPHICAL ABSTRACT



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## ABSTRACT

$CO_2$  capture with solid sorbents is one of the most promising options for reducing the consequences of climate change when large amounts of  $CO_2$  are emitted into the atmosphere. The present study explores the possible use of macroalgae industrial waste, algae meal (AM), and its carbonised (AMP) to prepare activated carbons by means of KOH and  $K_2CO_3$  activation in two different devices (an electrical conventional and a multimode microwave furnace) for  $CO_2$  capture and for the separation or concentration of gas mixtures. The adsorbents obtained, at  $750\text{ }^\circ\text{C}$ , with a low weight ratio of KOH:AM (0.5:1), show in most cases a high specific surface area-BET (up to  $1982\text{ m}^2/\text{g}$ ). These materials are mainly microporous ( $V_{\text{micropore}} > 80\%$ ) with a certain degree of mesoporosity and contain a significant amount of nitrogen. The adsorbents were subjected to a  $CO_2$ ,  $CH_4$  and  $H_2$  adsorption process at high pressure, showing a better selectivity towards  $CO_2$ , than towards  $CH_4$  and  $H_2$ . This suggests they could be used for capturing  $CO_2$  in pre-combustion processes or separating/concentrating mixtures of  $CO_2/CH_4$  in natural gas feeds for use as fossil fuel. Some of the adsorbents proved to be more effective in adsorbing  $CO_2$  than one of the commercial activated carbons used as reference (Chemviron Filtrasorb F400).

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## 1. Introduction

The emission of greenhouse gases, such as  $CO_2$ , has been increased so rapidly that it is causing serious environmental problems via global warming and climate change. The greenhouse gas with the largest impact on climate change is carbon dioxide. It is

for this reason that  $CO_2$  capture is becoming an increasingly used technology.  $CO_2$  capture can be performed by different routes, such as post-combustion ( $CO_2$  capture from the flue gas stream) or pre-combustion (the  $CO_2$  can be selectively separated from the shifted-syngas ( $CO_2/H_2$ ) prior to the generation of electricity).

Dependence on fossil fuel and the energy shortage situation is increasing. As a result, biogas and other digester gases are being considered as possible attractive alternatives to natural gas due to their high methane content [1]. However, this alternative is

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excluded by the presence of impurities such as, CO<sub>2</sub>. Therefore, gas purification to remove CO<sub>2</sub> could be a possible solution.

Pressure swing adsorption technology (PSA) is considered as one of the potential options, not only for CO<sub>2</sub> capture in pre-combustion but also as a means of natural gas purification. However the success of this methodology depends on the development of a good adsorbent with a high CO<sub>2</sub> selectivity and adsorption capacity. Porous materials, like activated carbons (ACs), are suitable materials for CO<sub>2</sub> capture due to their highly developed porous structure. In order to reduce the production costs of these adsorbents, many research studies have been undertaken to find alternatives to the raw materials used in the activated carbon production, such as agricultural and industrial wastes, [2–4]. In this work, the precursor of the ACs is an industrial waste, algae meal (AM), generated in the production of Agar–Agar from the industrial processing of macroalgae (*Gelidium sesquipedale*). This residue (2000–2400 kg/day) comes from an industry located in the north of Spain which is one of the largest world producers of Agar–Agar. Currently, a portion of this algae residue is used for fodder and fertilizer, although most of it is disposed of by landfill or incineration. The use of this waste as a precursor of activated carbon would have a dual benefit: an economic reward because it would be possible to obtain ACs at a lower manufacturing cost and an environmental reward, since it would contribute to a more efficient management of wastes.

There are many works in the literature related to the preparation and characterisation of activated carbon from biomass using KOH and K<sub>2</sub>CO<sub>3</sub> as activating agent [4–7]. There are also papers directed at obtaining activated carbon by microwave activation [5,8,9]. However, there are only a few works focused on the preparation of ACs from macroalgae, [10–14] and no one related to the use of this type of macroalgae waste as precursor of ACs. It is for this reason that it is a novel precursor.

The objectives of this study are: (i) to evaluate the chemical and textural characteristics of the activated carbon obtained from this macroalgae industrial waste (algae meal) by means of two different heating methods (conventional and microwave activation) and two different activating agents (KOH and K<sub>2</sub>CO<sub>3</sub>), and (ii) to evaluate the quality of the adsorbents obtained as an alternative method for CO<sub>2</sub> capture (pre-combustion processes) and for the purification of various gases (CO<sub>2</sub>/CH<sub>4</sub> or CO<sub>2</sub>/H<sub>2</sub>).

## 2. Experimental

### 2.1. Materials

Algae meal waste (AM) is generated by an industrial process that consists of alkaline treatment of the macroalgae *Gelidium sesquipedale*, a washing process with cold water, followed by the baking and filtration of the seaweed to extract Agar–Agar. Details of this process are explained in a previous work [15]. The residue generated from this industrial process was dried and ground to obtain the AM for use in this work. The algae meal was sampled for a period of one month by the personnel of the plant in order to ensure their representativeness during the sampling period and following standard procedures. To obtain a representative sample of the total, it was quartered by division cone and partitions dividers or “riffles” until a representative sample of about 2 kg was obtained.

The activated carbons obtained were prepared by means of two different heating methods: conventional chemical activation in a Carbolite CTF12/65/550 horizontal electrical furnace and microwave chemical activation in a multimode microwave device. Details of the experimental arrangements are described in a previous work [16].

Two precursors were selected for the activation process: the algae meal (AM) and its char (AMP) obtained by pyrolysis at 750 °C in a conventional furnace as described in a previous work [15]. The precursors were physically mixed with two different activating agents: KOH and K<sub>2</sub>CO<sub>3</sub> in activating agent/precursor proportions of 0.5:1. The mixture was placed in an annealed alumina vessel “Alsint” that was introduced into the furnaces for chemical activation at 750 °C. In both devices there is a thermocouple, which is in continuous contact with the sample. The sample is connected to a PID controller, enabling the activation temperature to be constantly controlled and monitored. In the case of the microwave, it is known from previous works [17] that algae meal is transparent to microwave radiation. Therefore, initially only the activating agent absorbs the microwaves. As activation progresses, the waste pore structure develops, enabling the activated carbon thus formed to adsorb the microwave energy [18].

Once the activated materials were obtained, they were washed with HCl 5M and deionized water to remove the products blocking the pores. Finally the materials were dried in a conventional and a vacuum stove, respectively.

In order to conduct a comparative study on the capacity of the obtained activated carbon for capturing CO<sub>2</sub>, H<sub>2</sub> and CH<sub>4</sub>, a Filtrasorb F400 commercial activated carbon, was selected since this material is homogeneous and readily available. Furthermore, F400 is resistant to high temperatures. The F400 has been used in previous CO<sub>2</sub>-sorption studies [19–22]. On the other hand the commercial activated carbon “Maxsorb 3000” was selected since it is a material widely used in gas storage [23–26] provided with very good textural properties [27].

### 2.2. Chemical, morphology and textural analysis

The moisture and ash contents of the sample were obtained following the ISO 11722 and ISO 1171 norms, respectively. The carbon, hydrogen, nitrogen and sulphur contents were determined in automatic LECO CHN-2000 and LECO S-144-DR instruments.

The surface morphology of the AM, AMP and their activated carbons were studied using a scanning electron microscope (SEM), ZEISS Model DMS-942. For the textural characterisation of the samples an automatic Micromeritics (ASAP 2420) apparatus was employed. Nitrogen isotherms adsorption were used to calculate the specific surface area  $S_{\text{BET}}$ , by the standard method of Brunauer, Emmett and Teller (BET theory) [28–30] and the Total Pore Volume ( $V_{\text{TOT}}$ ) at a relative pressure of 0.95. The micropore and mesopore size distributions was calculated on the basis of the density functional theory (DFT). Before the experiments, the samples were outgassed under vacuum at 120 °C overnight to remove any adsorbed moisture and/or gases.

### 2.3. CO<sub>2</sub>, H<sub>2</sub> and CH<sub>4</sub> capture capacity

High pressure adsorption isotherms were obtained on a Rubotherm-VTI magnetic suspension balance at room temperature and under static conditions. CO<sub>2</sub>, H<sub>2</sub> and CH<sub>4</sub> adsorption isotherms were determined up to 3 MPa. Fig. 1 shows a diagram of the magnetic suspension balance used in this work:

To perform the analysis, 1 g of the adsorbent material was placed into an aluminium cell. The sample to be analysed is linked to a suspension magnet which consists of a permanent magnet, a sensor core and a device for decoupling the measuring load. An electromagnet, which is connected to the weighing hook, maintains a state of free magnet suspension through electronic control. By means of this magnetic suspension coupling device the measuring force is transmitted, contactless, from the measuring chamber to the microbalance, which is located outside the chamber under ambient atmospheric conditions.

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