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## Laboratory scale pilot CO<sub>2</sub> capture vacuum pressure swing adsorption using MIL-160(AI) with dry flue gas

Arnaud Henrotin<sup>a</sup>, Nicolas Heymans<sup>a</sup>, Jose Casaban<sup>b</sup>, Guy De Weireld<sup>a\*</sup>

<sup>a</sup>*Thermodynamics and Mathematical Physics Unit, University of Mons (UMONS), Place du parc 20, 7000 Mons, Belgium*

<sup>b</sup>*MOF Technologies Ltd, 63 University Road, Belfast BT7 1NF, United Kingdom*

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### Abstract

Carbon capture is a crucial technology for achieving net-zero anthropogenic CO<sub>2</sub> emissions by 2050. Among the various separation methods, adsorption stands out as one of the most promising. Vacuum Pressure Swing Adsorption (VPSA) cycles have been extensively studied for CO<sub>2</sub> capture. Metal-organic frameworks (MOFs), with their remarkable tunability, offer the potential to enhance adsorption processes. Despite this, experimental data for MOFs at pilot scale remain scarce. To bridge this gap, a versatile lab-scale VPSA pilot system (comprising three 1.1 L columns) was developed to evaluate adsorbents at the kilogram scale under diverse adsorption process configurations. The MOF MIL-160(AI), synthesized and shaped at a scale of 60 kg, was tested using a 3-bed 6-step VPSA cycle for separating a 15/85 %vol CO<sub>2</sub>/N<sub>2</sub> mixture at pressures ranging from 0.1 to 2 bar. Results show that MIL-160(AI) achieved 90% CO<sub>2</sub> purity and 92.7% recovery, highlighting its efficiency for CO<sub>2</sub> capture. These findings underscore the need for testing materials in VPSA cycles at the kilogram scale to accurately evaluate their performance, challenging conventional indicators.

*Keywords:* CO<sub>2</sub> capture; Vacuum pressure swing adsorption; Metal organic framework; Laboratory pilot.

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### 1. Introduction

Greenhouse gas emissions, and particularly anthropogenic CO<sub>2</sub> emissions since the Industrial Revolution, have been identified as the primary driver of climate change. Achieving net-zero emissions by 2050 and limiting global warming to 1.5°C, as stipulated in the Paris Agreement and COP26, requires a substantial reduction in CO<sub>2</sub> emissions to 20 GtCO<sub>2</sub>/year by 2035 [1,2]. Carbon capture utilization and storage (CCUS) offers a promising solution to mitigate CO<sub>2</sub> emissions during the transitional period from fossil fuels to renewable energy sources and for unavoidable emissions from industrial processes such as cement, lime or steel production. The International Energy Agency (IEA) projects that CCUS could capture 5.2 GtCO<sub>2</sub>/year by 2050 [3]. Among carbon capture methods, post-combustion capture is the most mature and widely studied due to its ability to retrofit existing facilities. To be competitive, a post-combustion capture technology must exhibit 90-95% CO<sub>2</sub> recovery and 95% CO<sub>2</sub> purity, while being able to treat flue gas CO<sub>2</sub> concentrations ranging from 4% in natural gas plants to over 30% in cement kilns [4–8].

Chemical absorption using amine-based solvents, such as monoethanolamine (MEA), is the most mature post-combustion capture technology. This method, already deployed at a commercial scale, offers energy consumption as low as 2.2 GJ/tCO<sub>2</sub> [4,5,9,10]. Despite its efficiency and its industrial scale development, challenges such as solvent

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\* Corresponding author. Tel.: +3265374203, E-mail address: guy.dewereld@umons.ac.be

toxicity, degradation, and sensitivity to contaminants like SO<sub>x</sub> and NO<sub>x</sub> limit its broader application [11]. To address these drawbacks, alternative technologies—including membranes, cryogenic processes, calcium looping, and adsorption—are under active development [4,7]. Adsorption has emerged as a competitive alternative due to its lower energy requirements, reduced costs, and environmental benefits. Gas purification by adsorption is already employed in industrial processes such as hydrogen purification and air separation [7,12,13]. The adsorption process typically operates in cyclic modes such as Temperature Swing Adsorption (TSA), Pressure Swing Adsorption (PSA), or Vacuum Pressure Swing Adsorption (VPSA). V(P)SA is particularly cost-effective for moderate CO<sub>2</sub> concentrations due to the lower amount of gas being compressed in the vacuum pump compared to the compression of the flue gas. Although VPSA offers advantages, challenges remain such as energy demands for pressure modification and potential reductions in CO<sub>2</sub> purity due to purge gas mixing with CO<sub>2</sub> [7,13,14].

Material selection is critical to the performance of adsorption processes. Zeolites, such as 13X or NaY, are suitable for VPSA due to their high CO<sub>2</sub> adsorption capacities, selectivities, and fast kinetics. However, their hydrophilic nature reduces performance in humid conditions [15–17]. Activated carbons, with their hydrophobic properties and low regeneration energy requirements, are less sensitive to moisture but lack the high selectivity of zeolites. Advances in functionalization have improved their CO<sub>2</sub>/N<sub>2</sub> selectivity, making them increasingly viable [17]. Metal-organic frameworks (MOFs) are a promising class of adsorbents due to their tunable structures, high CO<sub>2</sub> capacity, and selectivity [18,19]. MOFs like UTSA-16, and CALF-20 outperform traditional adsorbents in CO<sub>2</sub> capture, particularly at low partial pressures [19–21]. Notably, CALF-20 exhibits stability in humid conditions which is an advantage over traditional adsorbents such as 13X and other MOFs [21].

Numerous newly developed materials are tested at grams scale allowing to quantify the properties of the materials, or by simulation to evaluate the performance in a cycle for CO<sub>2</sub> capture. Nevertheless, the evaluation of the adsorbent in a “real” cycle is rarely done to verify simulation predictions. In this study, a laboratory-scale VPSA pilot was developed to evaluate adsorbents for CO<sub>2</sub>/N<sub>2</sub> separation, using a 6-step cycle [22] adapted for three columns for continuous gas treatment. MIL-160(Al) [23], a microporous, bio-derived MOF, was tested at kilogram scale. It features a BET surface area of 1220 m<sup>2</sup>/g, a pore size of 4–6 Å, and high CO<sub>2</sub>/N<sub>2</sub> selectivity (34 by IAST) [24]. Produced under green conditions [25], it is stable in water, steam [26], and SO<sub>2</sub> [27], with low regeneration energy (heat of adsorption equal to -33 kJ/mol) and scalability for industrial applications, making it a promising candidate for CO<sub>2</sub> capture.

## 2. Equipment and methods

### 2.1. VPSA pilot

A versatile Vacuum Pressure Swing Adsorption (VPSA) pilot was developed to evaluate adsorbents at a flue gas flow rate from 0.5 to 3 Nm<sup>3</sup>/h. The pilot features three adsorption beds, a vacuum pump, a compressor, and a fully instrumented system to measure temperature, pressure, gas composition, and flow rates. This pilot is able to reproduce the most common step encountered in VPSA cycles: Adsorption, co-current and counter-current evacuation, light and heavy reflux, pressure equalization, light product pressurization, ... The pilot is composed of a gas generation system which uses CO<sub>2</sub> and N<sub>2</sub> cylinders to produce CO<sub>2</sub>/N<sub>2</sub> mixture. This mixture can be directly analysed to check the composition. Three adsorption columns can be used with a length of 30 cm and 7.01 cm diameter. The first column is equipped with two thermocouples for temperature monitoring inside. Six valves are connected to each column to send the gas in the different sections of the pilot depending on the cycle and the step ongoing.

The top of the column is connected to the waste section which is composed of a 500 L tank to store the depleted gas from the columns. Light reflux can be performed by directly connecting two columns or by reusing the gas from the tank. Pressure, temperature and gas composition are analysed with the gas coming from the top of the columns, and the gas inside the tank. Similar installation is present in the product section for CO<sub>2</sub> rich gas. Evacuation steps are carried out by a vacuum pump able to reach 2 Pa, using proportional valves for pressure control. A compressor is used for heavy reflux steps, using either gas coming from the vacuum pump or from the CO<sub>2</sub> tank.

A four-channel NDIR analyzer measures CO<sub>2</sub> concentrations at various sampling points during all stages of the VPSA

cycle. The system is automated via a programmable logic controller (PLC) integrated with custom software for real-time monitoring and control. Data on temperature, pressure, flow, and gas composition are recorded every second, and purity and recovery metrics are calculated to assess performance. The software interface enables manual or automated operation and provides visualization of process stability over multiple cycles. A complete description of the installation can be found in the reference [28].

## 2.2. VPSA cycle

The cycle performed in this work is the 6-step cycle from Khurana & Farooq [29] which has been adapted to work with 3 columns. The representation of the cycle with the state of each bed during each step is given by Figure 1. The different steps are:

- Adsorption: the  $\text{CO}_2/\text{N}_2$  mixture is sent by the bottom of the column,  $\text{CO}_2$  is adsorbed in the column and nitrogen flow out of the column.
- Heavy reflux (HR): the gas coming from a column in light reflux step is used to flush the column, replacing the nitrogen by  $\text{CO}_2$  in gaseous phase and increasing the amount of  $\text{CO}_2$  adsorbed.
- Co-current evacuation (co-evac): the pressure of the column is decreased by the top of the column to an intermediate pressure. This gas which is mainly nitrogen is not collected and sent to the atmosphere.
- Counter-current evacuation (cn-evac): the pressure is further reduced by the bottom of the adsorption bed. The gas mainly composed from  $\text{CO}_2$  is collected.
- Light reflux (LR): Nitrogen coming from a column in adsorption step is used to flush  $\text{CO}_2$  in the gas phase.
- Light product pressurization (LPP): The column is pressurized by the gas coming from the column in adsorption by the top.

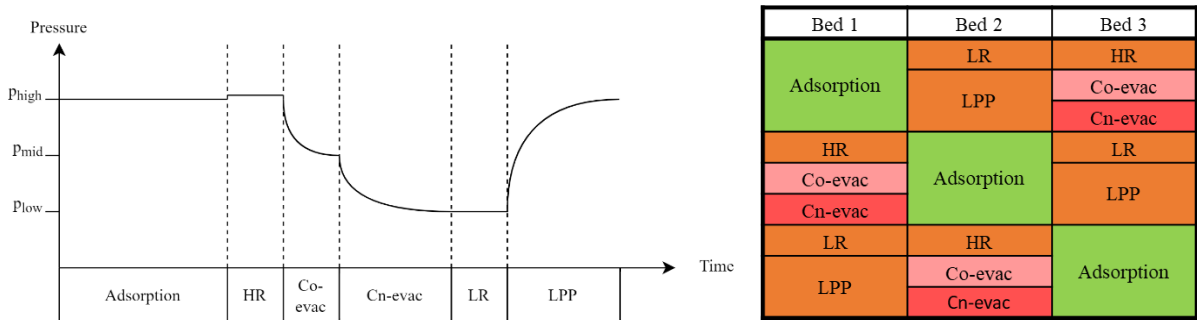


Figure 1: Pressure level representation of bed 1 during the 3-bed 6-step cycle (left), and configuration of the cycle with three adsorption bed (right) (HR: heavy reflux, co-evac: co-current evacuation, cn-evac: counter-current evacuation, LR: light reflux, LPP: light product pressurization). The size of the blocks is not representative of the duration of the steps.

Based on Figure 1, three relationships can be written between the different steps reducing the number of parameters defining the cycle to three:

$$t_{LR} = t_{HR} \quad (1)$$

$$t_{LPP} = t_{adsorption} - t_{LR} \quad (2)$$

$$t_{counter-current\ evacuation} = t_{adsorption} - t_{LR} - t_{co-current\ evacuation} \quad (3)$$

In addition to these three times, the pressure levels for adsorption, co and counter-current evacuation must be selected. Light reflux flow rate is also an additional parameter of the process which can be optimized. In this study, adsorption pressure was set to 2 bar and counter-current evacuation pressure to 0.1 bar for each experiment. In addition, the feed flow rate treated by the pilot is 1 Nm<sup>3</sup>/h with a 15/85  $\text{CO}_2/\text{N}_2$  mixture. The parameters studied with their upper and lower bounds are listed in the Table 1.

Table 1: Lower and upper bounds of the parameter studied with the VPSA pilot.

Parameter	Lower bound	Upper bound
Adsorption time [s]	100	230
Light reflux time [s]	40	150
Co-current evacuation time [s]	20	40
Co-current evacuation pressure [bar]	0.4	0.6
Light reflux flow rate [Nm <sup>3</sup> /h]	0.1	0.3

Two key indicators were used to determine the performance of the MIL-160(Al) in cycle: purity and recovery. Purity is the average CO<sub>2</sub> concentration obtained in the product stream. Purity is obtained by summing the flow coming from the product outlet of the pilot, multiplied by the CO<sub>2</sub> concentration of this stream, divided by the sum of the flow of the product stream (equation 5).

$$Purity = \frac{\int_{cycle} Q_{product} \cdot y_{CO_2 product}}{\int_{cycle} Q_{product}} \quad (4)$$

With  $Q_{product}$  the flow rate of the product stream, and  $y_{CO_2 product}$  the CO<sub>2</sub> concentration of the product stream. Recovery is the ratio of the amount of CO<sub>2</sub> retrieved in the product stream of the pilot by the amount of CO<sub>2</sub> in the stream feed (equation 6).

$$Recovery = \frac{\int_{cycle} Q_{product} \cdot y_{CO_2 product}}{\int_{cycle} Q_{feed} \cdot y_{CO_2 feed}} \quad (5)$$

With  $Q_{feed}$  the flow rate of the feed stream, and  $y_{CO_2 feed}$  the CO<sub>2</sub> concentration of the feed stream. A design of experiments based on the bounds of Table 1 was established to evaluate the performance of the pilot using MIL-160(Al). This method allows to study the impact of the different parameters of the cycle with a limited number of experiments. A feed flow rate of 1 Nm<sup>3</sup>/h with a 15/85 CO<sub>2</sub>/N<sub>2</sub> mixture was used as a feed gas for all experiments.

### 3. Results

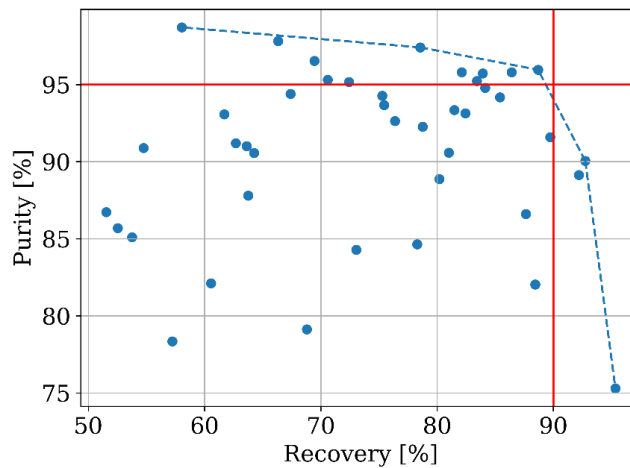


Figure 2: Experimental recoveries and purities obtained on the VPSA pilot for MIL-160(Al).

42 experiments were performed on the VPSA pilot. Results obtained are represented in the pareto plot on Figure 2 showing the recovery and purity obtained for each experimental run and the experimental pareto front represented by the dotted line. Several operating conditions yield a purity above 95% or a recovery exceeding 90%. However, achieving both 95% recovery and 95% purity simultaneously is not possible. When the purity exceeds 95%, the maximum recovery achieved is 88.7%, and when the recovery is at least 95%, the highest purity attained is 75.3%. Nevertheless, it is feasible to achieve both recovery and purity of at least 90% with MIL-160(Al) giving 92.7% of recovery for 90% purity.

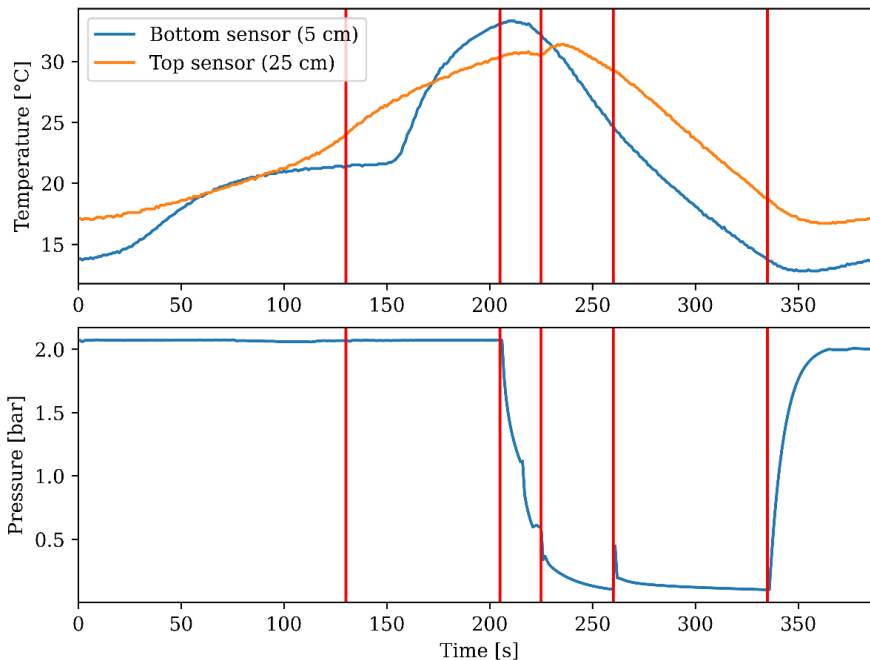


Figure 3: Temperature (upper) and pressure (bottom) profiles obtained for the experiment giving recovery and purity higher than 90%. Red lines are transition between two steps, first step is adsorption.

Temperature and pressure profiles measured during one cycle in steady state is represented on Figure 3 for experiment giving purity and recovery higher than 90%. The pressure profile closely follows the theoretical profile shown in Figure 1, with several fluctuations caused by the vacuum pump and the opening and closing of valves during transitions between steps. During co-current evacuation, the pressure is reduced in two stages to prevent exposing the vacuum pump to a pressure higher than atmospheric. During the adsorption step, the bottom part of the column experiences a temperature increase due to heat released by adsorption followed by a plateau, likely due to the saturation of the adsorbent with  $\text{CO}_2$ . Meanwhile, the top temperature shows a slight increase during this phase. During the heavy reflux phase, the bottom sensor records a sharp temperature rise, exceeding  $30^\circ\text{C}$ . In contrast, the temperature increase at the top sensor is less pronounced, reaching lower values. This indicates that most of the  $\text{CO}_2$  from the heavy reflux step is adsorbed at the bottom of the column, with only a small amount arriving at the top. During the evacuation and light reflux phases, the temperature decreases due to the desorption of  $\text{CO}_2$  and the pressure decrease. The bottom sensor records a minimum temperature of  $12.8^\circ\text{C}$ , indicating an effective  $\text{CO}_2$  desorption with this adsorbent.

#### 4. Conclusion

This study demonstrates the potential of MIL-160(Al) as a promising adsorbent for  $\text{CO}_2$  capture in a Vacuum Pressure Swing Adsorption process. By utilizing a laboratory-scale VPSA pilot performing the 3-bed 6-step cycle and an experimental design, MIL-160(Al) was evaluated for its performance in  $\text{CO}_2/\text{N}_2$  separation under realistic operating conditions. The results revealed that MIL-160(Al) can achieve both recovery and purity above 90%, with recovery and purity reaching 92.7% and 90%, respectively. However, achieving the simultaneous targets of 95% purity and 95% recovery remains unattainable, with trade-offs observed between these performance metrics.

The temperature and pressure profiles measured during the VPSA cycle reveals the material's excellent  $\text{CO}_2$  adsorption properties, especially during heavy reflux where the  $\text{CO}_2$  is retained in the first layers of the bed. Desorption performance of this MOF is also interesting, as indicated by the low temperature at the bottom of the adsorption bed during the evacuation phase. Additionally, the properties of the MIL-160(Al) to maintain stability under varying

process conditions and its bio-derived, environmentally friendly synthesis route make it a viable candidate for industrial-scale applications.

Overall, the results confirm that MIL-160(Al) exhibits competitive performance for CO<sub>2</sub> capture in post-combustion processes. Further optimization of the cycle parameters (pressures, feed flow rate, ...) must be done to reach the targets of 95% purity and recovery. Future work should focus on scaling up the process and evaluating the long-term stability of MOF particularly due to the presence of impurities in the flue gas to be treated.

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