

Monolithic MOFs for Carbon Capture

CCUS Innovation 2.0

Key Knowledge Deliverable 3.2

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WP3: Test Rig Adaptation and Experimentation

1. Introduction

Work package 3 (WP3) of the CCUS project focuses on characterization and performance evaluation of top performing materials that were synthesized during WP1. This work package allows the assessment of adsorption-desorption performance of the chosen materials through equilibrium uptake and dynamic column breakthrough (DCB) measurements. Moreover, WP3 provides critical information on structural stability of nominated adsorbents including their thermal, mechanical and chemical stabilities. Originally, the plan was to outsource this work package to Brunel and Swansea Universities. However, the construction of PVSA/PTSA rigs at these universities did not complete before the start of CCUS project. Consequently, WP3 was restructured and a new partnership with Cranfield University was formed. All changes were approved by DESNZ through a series of change requests submitted through the official channels. The updated scope of the work includes systematic and comprehensive characterization of IMM-16 and IMM-28 in their Gen 1 and Gen 2 forms.

2. Experimental Set-ups

Three distinct classes of experiments have been conducted as part of WP3: a) experiments associated with measurement of competitive adsorption of gases, which were mainly carried out using the DCB technique; b) investigation of materials stability including their thermo-mechanical, and chemical stabilities; c) measurement of MOFs physical properties such as heat capacity and thermal conductivity. IMM-16 and IMM-28 were both tested in these measurements. A complete list of all experiments carried out as part of WP3 is presented in Table 1 and Table 2.

Table 1. Complete list of DCB experiments for IMM-16 and IMM-28 in Gen 1 and Gen 2 MOFs (N₂ is the carrier gas in all tests)

Test Index	Test Title	MOF form/type	Particle Size (mm)	CO ₂ Concentration (%)	Flowrate (ml/min)	Total Pressure (bar)	Temperature (°C)	Relative Humidity (%)
1.1.	IMM-16_(2.8-3.35 mm)_15CO ₂ _N ₂ _200_1_25_0	Gen 1/IMM-16	2.8-3.35	15	200	1	25	0
1.2.	IMM-16_(2.8-3.35 mm)_15CO ₂ _N ₂ _400_1_25_0	Gen 1/IMM-16	2.8-3.35	15	400	1	25	0
1.3.	IMM-16_(2.8-3.35 mm)_15CO ₂ _N ₂ _600_1_25_0	Gen 1/IMM-16	2.8-3.35	15	600	1	25	0
1.4.	IMM-16_(2.8-3.35 mm)_15CO ₂ _N ₂ _800_1_25_0	Gen 1/IMM-16	2.8-3.35	15	800	1	25	0
1.5.	IMM-16_(2.8-3.35 mm)_15CO ₂ _N ₂ _400_5_25_0	Gen 1/IMM-16	2.8-3.35	15	400	5	25	0
1.6.	IMM-16_(0.25-0.5 mm)_15CO ₂ _N ₂ _400_1_25_0	Gen 1/IMM-16	0.25-0.5	15	400	1	25	0
1.7.	IMM-16_(2.8-3.35 mm)_15CO ₂ _N ₂ _400_1_45_0	Gen 1/IMM-16	2.8-3.35	15	400	1	45	0
2.1.	IMM-28_(2.8-3.35 mm)_15CO ₂ _N ₂ _200_1_25_0	Gen 1/IMM-28	2.8-3.35	15	200	1	25	0
2.2.	IMM-28_(2.8-3.35 mm)_15CO ₂ _N ₂ _400_1_25_0	Gen 1/IMM-28	2.8-3.35	15	400	1	25	0
2.3.	IMM-28_(2.8-3.35 mm)_15CO ₂ _N ₂ _600_1_25_0	Gen 1/IMM-28	2.8-3.35	15	600	1	25	0
2.4.	IMM-28_(2.8-3.35 mm)_15CO ₂ _N ₂ _800_1_25_0	Gen 1/IMM-28	2.8-3.35	15	800	1	25	0
2.5.	IMM-28_(2.8-3.35 mm)_15CO ₂ _N ₂ _400_5_25_0	Gen 1/IMM-28	2.8-3.35	15	400	5	25	0
2.6.	IMM-28_(0.25-0.5 mm)_15CO ₂ _N ₂ _400_1_25_0	Gen 1/IMM-28	0.25-0.5	15	400	1	25	0
2.7.	IMM-28_(2.8-3.35 mm)_15CO ₂ _N ₂ _400_1_45_0	Gen 1/IMM-28	2.8-3.35	15	400	1	45	0
3.1.	IMM-16_15CO ₂ _N ₂ _200_1_25_0	Gen 2/IMM-16	---	15	200	1	25	0
3.2.	IMM-16_15CO ₂ _N ₂ _400_1_25_0	Gen 2/IMM-16	---	15	400	1	25	0
3.3.	IMM-16_15CO ₂ _N ₂ _600_1_25_0	Gen 2/IMM-16	---	15	600	1	25	0
3.4.	IMM-16_15CO ₂ _N ₂ _1000_1_25_0	Gen 2/IMM-16	---	15	1000	1	25	0
3.5.	IMM-16_30CO ₂ _N ₂ _400_1_25_0	Gen 2/IMM-16	---	30	400	1	25	0
3.6.	IMM-16_15CO ₂ _N ₂ _400_1_45_0	Gen 2/IMM-16	---	15	400	1	45	0
3.7.	IMM-16_15CO ₂ _N ₂ _400_1_65_0	Gen 2/IMM-16	---	15	400	1	65	0
3.8.	IMM-16_15CO ₂ _N ₂ _1000_1_85_25	Gen 2/IMM-16	---	15	1000	1	85	25
3.9.	IMM-16_15CO ₂ _N ₂ _1000_1_85_50	Gen 2/IMM-16	---	15	1000	1	85	50
3.10.	IMM-16_15CO ₂ _N ₂ _1000_1_85_95	Gen 2/IMM-16	---	15	1000	1	85	95
4.1.	IMM-28_15CO ₂ _N ₂ _200_1_25_0	Gen 2/IMM-28	---	15	200	1	25	0
4.2.	IMM-28_15CO ₂ _N ₂ _400_1_25_0	Gen 2/IMM-28	---	15	400	1	25	0
4.3.	IMM-28_15CO ₂ _N ₂ _600_1_25_0	Gen 2/IMM-28	---	15	600	1	25	0
4.4.	IMM-28_15CO ₂ _N ₂ _1000_1_25_0	Gen 2/IMM-28	---	15	1000	1	25	0
4.5.	IMM-28_30CO ₂ _N ₂ _400_1_25_0	Gen 2/IMM-28	---	30	400	1	25	0
4.6.	IMM-28_15CO ₂ _N ₂ _400_1_45_0	Gen 2/IMM-28	---	15	400	1	45	0
4.7.	IMM-28_15CO ₂ _N ₂ _400_1_65_0	Gen 2/IMM-28	---	15	400	1	65	0

In Table 1 each experiment is listed with a unique identifier (Index Number) and a Test Title with the latter having the following format:

Material name_CO₂ mole fraction_carrier
gas_flowrate_pressure_temperature_relative humidity

For example: IMM-28_15CO₂_N₂_400_1_65_0 refers to an experiment carried out on IMM-28 with 15% CO₂ concentration in the feed and with N₂ as carrier gas at 400 l/min flowrate, 1 bar pressure, 65 °C temperature, and 0% relative humidity.

Table 2. Overview of material stability and physical property measurements.

Test Index	Test Title	MOF Form/Type	Task Description
5.1.	Thermogravimetric Analysis (TGA)	Gen 1/IMM-16, IMM-28	Thermal and Cyclic Adsorption-Desorption Stability
5.2.	Attrition by Fluidization	Gen 1/IMM-16, IMM-28	Mechanical Stability
5.3.	Accelerated Ageing Test	Gen 1/IMM-28	Thermal and Mechanical Stability
5.4.	Differential Scanning Calorimetry (DSC)	Gen 1/IMM-16, IMM-28	Heat Capacity Measurement
5.5.	Laser Flash Analysis	Gen 1/IMM-16	Thermal Conductivity Measurement

3. Dynamic Column Breakthrough (DCB) Experiments

Two different experimental set-ups were developed to carry out DCB experiments on Gen.1 and Gen. 2 reactors as shown in Figure 1 and Figure 2.



Figure 1. Experimental DCB set-up built with either Gen 1 or Gen 2 reactors.

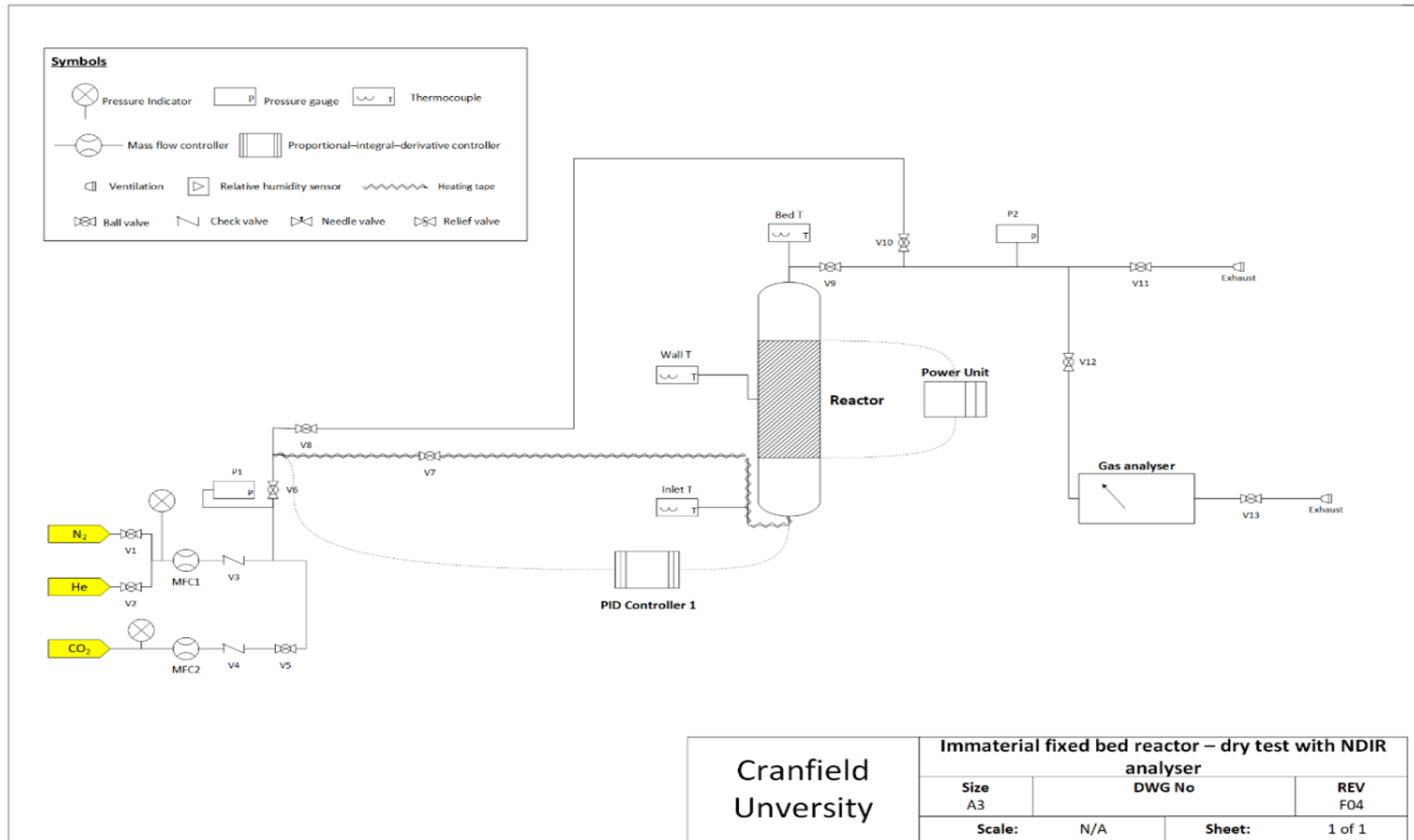


Figure 2. P&ID of the DCB set-up with either Gen 1 or Gen 2 reactors.

The DCB setup consists of a reactor loaded with either Gen 1 or Gen 2 adsorbent, where a gas mixture is continuously flowed into the bed while the concentration of each gas component is monitored at the outlet. This is used to study competitive adsorption dynamics under various conditions. The gas mixture was prepared by blending CO₂ with a carrier gas (typically N₂ or He), using two mass flow controllers (MFCs) to precisely regulate individual flow rates. The total flow was introduced to the reactor inlet, where the feed conditions, e.g. feed flowrate, feed pressure, feed temperature, feed CO₂ concentration, and feed relative humidity, were systematically varied during the experiments.

Prior to each breakthrough test, the MOF bed was activated using a dedicated heating system capable of preheating the gas stream before entry into the reactor. Activation was carried out under a flow of dry gas, i.e. N₂ or He, at elevated temperatures to ensure removal of residual moisture and adsorbed CO₂. Once activated, the reactor was cooled to the desired test temperature before introducing the wet or dry feed.

The outlet gas from the reactor was routed to a CO₂ analyser, which continuously monitored the effluent concentration over time, enabling breakthrough curves to be recorded. These curves were then used to quantify the dynamic adsorption capacity and to analyse the effects of process parameters on the performance of Gen 1 and Gen 2 materials.

Special care was taken to minimize dead volumes and to ensure uniform gas distribution across the bed. Challenges encountered during the setup and testing included maintaining consistent humidity levels at elevated temperatures, as well as thermal management of the reactor to avoid temperature gradients during activation and operation.

4. Material Stability Experiments

4.1. Thermogravimetric Analysis (Test 5.1)

We have employed TGA to investigate thermal stability and cyclic CO₂ capacity of our MOFs (Figure 3). By analysing mass changes of the MOF sample during heating and cooling cycles, we can obtain valuable information about their thermal decomposition patterns and structural stability. These data are crucial for understanding the

temperature range within which our MOFs maintain their structural integrity. Additionally, TGA was used to assess the reversibility and performance of MOFs in CO₂ adsorption-desorption cycles.

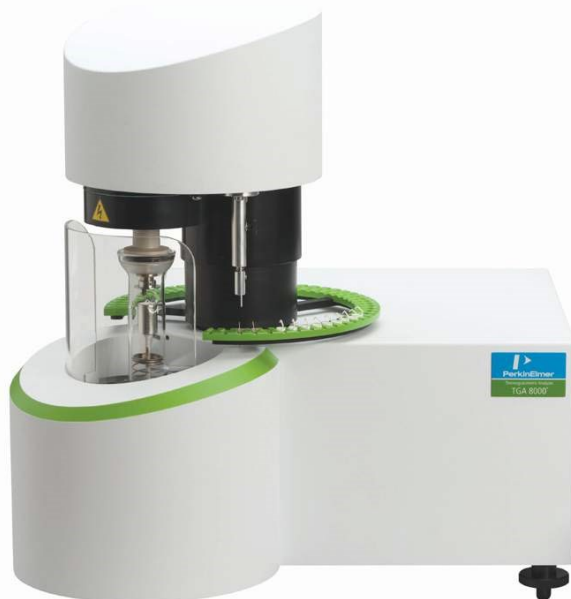


Figure 3. Photographic image of the thermogravimetric analyser 8000 (Perkin Elmer).

4.2. Attrition by Fluidization (Test 5.2)

Attrition by Fluidization test was used to assess stability and resistance of adsorbent materials against mechanical degradation that can happen in fluidized reactors. Here, adsorbents are fluidized at different velocities of the feed gas and particle size distribution (PSD) of collected samples are measured afterwards. Analysis of the PSD data will tell us if pellets have been crumbled during the fluidization process.

4.3. Accelerated Ageing Test (Test 5.3)

Accelerated ageing test is a crucial method employed to evaluate the long-term durability and performance of MOFs when exposed to specific operating conditions. Here, the primary goal is to simulate the conditions to which the material is exposed. By exposing the MOF to these conditions in an accelerated manner, potential

degradation or changes in adsorption capacity of MOFs can be assessed over a shorter period.

In our AAT tests, adsorbents were exposed to a simulated gas mixture with a composition similar to those of typical flue gas for duration of 2 weeks. After the test, CO₂ uptake of the adsorbents was measured in a DCB experiment and the results were compared against the original sample before any exposure test.

5. Physical Properties Measurements

5.1. Differential Scanning Calorimetry (DSC) (Test 5.4)

We employed modulated differential scanning calorimetry (DSC) using a thermal analysis (TA) instrument calorimeter to calculate specific heat capacity of our MOFs. This property is of particular importance in swing adsorption systems as it can control temperature fluctuations of the reactor during adsorption and desorption processes.

5.2. Thermal Conductivity Measurement (Test 5.5)

We employed the Laser Flash Analysis (LFA) using a TA Instrument model DLF 1200A to measure thermal conductivity of our MOFs. Thermal conductivity of an adsorbent plays a critical role in dissipation of thermal energy through the reactor potentially impacting adsorption/desorption uptake and kinetic of the process.

6. Concluding Remarks

Work package 3 of the CCUS project allowed extensive characterization of two of Immaterial's top performing m-MOFs (IMM-16 and IMM-28). As part of the work carried out during this work package, two different DCB rigs were developed and commissioned which were dedicated to testing of our Gen 1 and Gen 2 reactor designs. Suitability of each rig was carefully tested prior to the start of experiments and any necessary improvements were implemented. The actual experiments were divided into 3 categories: 1) breakthrough measurements, 2) aging and stability tests, 3) physical properties measurements.

As part of the above 3 categories, we tested sub-atmospheric and high-pressure uptake and kinetic of CO₂ adsorption process at different conditions of the feed

including feed temperature, flowrate, composition, and relative humidity. We also examined, thermal, mechanical, and chemical stabilities of our Gen 1 MOFs. Key physical properties such as heat capacity and thermal conductivity of our MOF adsorbents were also measured.

Key information obtained from WP3 was then used to validate and refine the models developed in WP2. The above experiments provided critical learning and insights into the ways that key performance indicators (purity, recovery, productivity, etc.) of our PVSA process can be further improved.

We believe the valuable information obtained during WP3 will benefit Immaterial's system and material development efforts that will continue beyond the end of CCUS project.

7. Glossary

MOF	Metal Organic Framework
DCB	Dynamic Column Breakthrough
BT	Breakthrough
MFC	Mass Flow Controller
P&ID	Piping and Instrumentation Diagram
RH	Relative Humidity
TGA	Thermogravimetric Analysis
DSC	Differential Scanning Calorimetry
TA	Thermal Analysis

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