Site Testing of Pilot-scale Unit

CCUS Innovation 2.0

Key Knowledge Deliverable 4.3

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Key Knowledge Deliverable Cover Sheet

This Key Knowledge Deliverable (KKD) has been produced by Promethean Particles Ltd. as part of the Department for Energy Security and Net Zero £1bn Net Zero Innovation Portfolio (NZIP) - CCUS Innovation 2.0 programme. The document is reflective of the status of the project at the time of writing. The material presented could have been subject to change as the project matured. These documents should not be considered a full representation of the final project.

Description of KKD 4.3 Rig Operation and Decommissioning

This deliverable report describes the testing completed and decommissioning of the MONET testing unit. The testing phase yielded valuable learnings that will lead to improvements on the next generation testing unit. Adsorption and desorption of Promethean's metal-organic framework (MOF) was conducted, and the CO2 breakthrough performance was determined. This allowed the capacity of the MOF to be determined under a test-site's flue gas emission conditions.

Once operation was complete, the rig was successfully decommissioned, removed from the test site and relocated to Promethean's facilities.

KKDs to be released in full:

D6.4 - Marketing Material Creation

D6.5 - Conference Presentations and Trade-Show Exhibitions

KKDs to be released after redaction:

D3.2 - Control and Safety System Manufacturing

D3.4 - Build of Capture Rig

D4.1 - Installation of Capture Rig

D4.3 - Rig Operation and Decommissioning

D5.1 - CAPEX Technoeconomic Analysis (TEA)

D5.2 - OPEX TEA

D5.3 - Life Cycle Analysis (LCA)

D6.6 - Stakeholder Analysis

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Introduction

The successful dry and wet commissioning of the testing unit, and subsequent training of operators during earlier work package tasks allowed the testing unit to proceed to the testing phase. D4.3 addresses the use of the testing unit under flue gas conditions with MOF loaded in the column. The learnings from the commissioning process will be applied to enable successful testing with MOF.

Deliverable Aims

This deliverable will demonstrate the operation of the MONET testing unit under flue gas conditions with a MOF. The design specification aims to achieve 0.5-1.0 t/day CO2 capture rate, with dual column operation (MOF in each column) and pre-determined cycle times.

After the completion of the commissioning process in an earlier deliverable, the scope of this deliverable was re-evaluated to reflect the operational constraints of the testing unit. The first phase will be in manual mode with single column testing. The aims of this phase are to evaluate the impact of vacuum regeneration during desorption, the impact of water on MOF performance during adsorption and finally the impact of heat and/or vacuum on super-regeneration of the MOF.

Subsequent aims of this deliverable are contingent on successful first phase testing. If successful, the main aim is to conduct adsorption-desorption cycles in automated mode.

The final aim of this deliverable is to decommission the testing unit, with removal of the MOF from the column, and disconnection of flue gas and power.

Results and Discussion

MOF Loading

Half of the MOF produced as part of work package 3 was heated overnight to reach a fully activated state at Promethean Particles. The material was then shipped to the testing facility. A procedure was followed which allowed the MOF to be loaded into bed 1. MOF was positioned between 2 layers of glass wool. Figure 1 shows an image of the bed after MOF loading. The purpose of the glass wool was to minimise the amount of MOF removed from the bed into the flue gas flow and limit movement of MOF within the bed during pressure changes (particularly while switching between vacuum operation and ambient operation).

Figure 1. Image of the complete arrangement in the adsorption bed, with glass wool on top of the MOF

Operation of the MONET Testing Unit with Single MOF Column

The amended operating conditions of the MONET testing unit determined during commissioning work were utilised for all testing discussed in further sections.

One change was a reduced flow rate as measured by the outlet flow sensor. This change was implemented due to the pressure limitations of a system part. Another change was the temperature control of the pipework and vessels within the unit. It was known that water would be present in the flue gas stream coming into contact with MOF, rather than being removed prior to MOF exposure, resulting from limitations in the engineered system.

During the testing phase, MOF samples were taken at significant points: after the first adsorption-desorption cycle, after various regeneration trials with vacuum and temperature swing, before and after heating with air purging, and at the end of the testing, in order to later analyse the individual effects of flue gas exposure, and repeated temperature and vacuum treatment.

First Flue Gas Trial with MOF

The baseline CO2 concentration was determined upon start-up of the unit and flow was diverted to bed 1 (containing MOF) and bed 2 (empty) was isolated from the flue gas flow. The outlet CO2 concentration decreased, demonstrating that the MOF was successful at removing the CO2 from the flue gas. Outlet flow rate was simultaneously observed to decrease during this period due to the lower volume of gas travelling through the system as CO2 was being stored within the MOF. The breakthrough of CO2 through the MOF bed was evidenced by an increase in CO2 concentration, indicating that the MOF was becoming saturated with CO2. Complete breakthrough was determined to have occurred when the original CO2 concentration was detected. The CO2 concentration was allowed to stabilise over ca. 10 minutes to confirm that no further CO2 adsorption was occurring. At this stage the adsorption phase ended. Focussing on the outlet flow rate, the flow rate first decreased, plateaued, and then slowly increased back to the original value. Water uptake was also recorded, indicated by a drop in outlet relative humidity during the adsorption phase. This decrease was stable throughout the adsorption.

An observation during the adsorption step was a temperature decrease at the outlet sensor from 21 °C to 15 °C, with an increase to 21 °C following. This may be due to the temperature difference between bed 2 and bed 1, as the MOF is known to be a poor thermal conductor and it is likely that the MOF was a lot colder than the incoming flue gas.

The desorption process was conducted by applying vacuum to bed 1. An immediate spike in the outlet CO2 concentration was observed before steadily declining. The flow rate rapidly increased when the vacuum pump was turned on. There is no measure of humidity in the vacuum pipework and so it is unclear whether the vacuum regeneration was sufficient to remove the water contained within the MOF.

Repeat Trials with MOF

Following the first flue gas trial whereby the MOF clearly demonstrated successful CO2 capture, further repeat trials were attempted. The MOF bed initially saw no further treatment beyond the vacuum desorption process carried out immediately after adsorption.

Subsequent trials occurred with a vacuum regeneration following. This outcome confirmed that vacuum regeneration alone is not sufficient to remove adsorbed water from the MOF, and as such the MOF had no available capacity to adsorb CO2 but would preferentially adsorb water.

With the outcome of the flue gas trials concluding that vacuum regeneration was not sufficient for the flue gas composition attainable in the current unit design, further work focussed on assessing the super regeneration methodology, which involves heating the adsorption vessels with the option of utilising vacuum throughout the process or not.

Multiple trials were undertaken in an iterative process. A combination of the variables were considered during the trials: heater set point, vacuum operation, air purging. After each super regeneration attempt, an adsorption was carried out to evaluate the degree of success. A key limitation of the test unit is that there is no integrated temperature monitoring within the vessel to ascertain the temperature of the MOF. This means that it was not possible to verify that the MOF had reached the target temperature during super regeneration.

Three distinct sets of super regeneration trials (SR1-3) were conducted utilising a combination of heating and vacuum application over a set period of time/steps. Due to challenges encountered with rig design limitations, and the known thermally insulative properties of the MOF itself, uniformly heating the MOF bed only achieved partial regeneration.

MOF Analysis

Upon completion of the site testing, a sample of MOF from the column in the MONET testing unit was separated for analysis. The purpose of this was to determine if repeated exposure to the flue gas, vacuum and elevated temperature had a noticeable impact on the quality of the MOF. The analysis undertaken was X-ray diffraction (XRD) to determine the crystal structure, nitrogen (N2) adsorption to determine the BET surface area and carbon dioxide adsorption to determine CO2 uptake capacity.

XRD results from the MOF exposed to flue gas, as compared to pristine MOF, showed that there were no significant changes to either the peak position or peak height, which indicates that the crystal structure of the MOF was not significantly degraded, and the material remains identifiable as the correct MOF structure after exposure to flue gas.

Further analysis will be required on the MOF to draw conclusions. In the future, elemental analysis, infrared spectroscopy, microscopy and pore size distribution will also be investigated to further probe any changes observed.

Decommissioning

The process of decommissioning the MONET testing unit involved multiple stages. Firstly, the MOF was removed from the column, prior to transport to Promethean Particles for in-depth analysis. Secondly, the connections between the MONET unit and the test-site flue gas outlet were removed to allow the unit to be removed.

The MOF was firstly removed from bed 1, utilising the operating procedure developed within the project. The MOF was then packaged prior to shipment back to Promethean for analysis.

Decommissioning involved removal of the flue gas hoses and the power supply. The hoses were decontaminated before being stored inside the unit. Removal from the test site was successfully completed and Figure 2 shows the delivery of the MONET rig at Promethean Particles after removal from the test site.

Figure 2. Image of the MONET rig arriving at Promethean Particles following the conclusion of site testing.

Concluding Remarks

This document summarises the testing of the MONET rig for capture of CO2 from flue gas streams using a metal-organic framework. A successful adsorption-desorption run was performed, achieving CO2 uptake. Due to the design limitations of the rig, and other technical factors, the initial planned testing approach had to be adapted in view of these challenges which provided valuable insights into necessary improvements in the rig's design for future studies.

The MOF was analysed both before and after being utilised in the MONET rig through a variety of analytical techniques. The crystal structure was demonstrated to be maintained, with further analytical work to be done beyond the project timeframe.