



Deliverable 5.5: Final technical report

WP 5: Project management and dissemination

Task 5.1: project coordination and management

Version: 2021-01-22, Final

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Classification: Public



This Deliverable report is part of the ACT-3D-CAPS project.
This project has received funding from RVO (NL),
RCN (NO), UEFISCDI (RO), and is co-funded by the CO₂ Capture Project (CCP)
and the European Commission under the Horizon 2020 programme ACT,
Grant Agreement No 691712

Version Management	
Version	Description of updates
V20200920	Draft to be completed
v20201228	Draft for internal review
V20210122	Final

Identification of the project and report

Project Title	Three Dimensional Printed Capture Materials for Productivity Step Change, 
Project ID	271503
Coordinator	TNO / Robert de Boer
Project Website	www.3d-caps.eu
Reporting period	December 2017-September 2020

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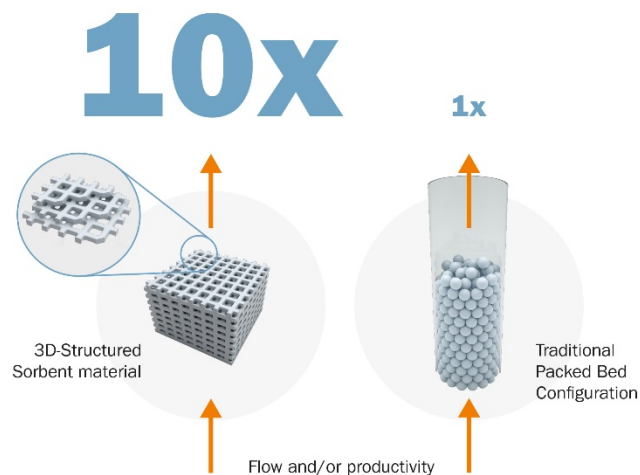
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1 Summary

Reducing the carbon footprint of many of the technologies associated with today's society is a challenge in terms of the size of equipment as well as the energy costs associated with the CO₂ abatement. The 3D-CAPS project aims to reduce the size of the equipment needed to remove and recover CO₂ from industrial gases, using two promising new adsorption-based technologies with an inherently small energy footprint. The 3D-CAPS project is carried out by an international consortium of end-users in the oil and gas industry, a technology provider, an SME, and European research and academic institutes.

The required adsorbents for these technologies are prepared using the latest innovations in additive manufacturing, commonly known as 3D-printing. This technology allows tailored materials with much improved heat and mass-transfer characteristics to be prepared, that are not available through traditional material preparation routes.



The objectives of the 3D-CAPS project are:

- To achieve a 10-fold productivity increase for two sorbent-based technologies in CCS
- To optimize sorbent shapes with Computational Fluid Dynamics (CFD) and other modelling tools, with direct realization in 3D-printed objects for testing under relevant conditions

Two development lines for structured sorbents are elaborated in the project:

- amine functionalised silica-supported sorbents (ImmoAmmo) for operation in post-combustion conditions in the 40-130 °C temperature range,
- hydrotalcites (HTC), suitable for operation under pre-combustion conditions at elevated pressure (up to 30 bar) in the 350-550 °C temperature range.

The Direct Light Processing (DLP) 3D-printing machine was successfully put in operation to produce 3D-printed structures of both silica and hydrotalcites. This printing technique requires slurries of the base materials, in a mixture with pre-polymers and photo-active additives to create the structured sorbents. Recipes for these slurries were developed, as well as the detailed procedures for printing the structures, and the post-processing steps to clean the printed structure and to sinter the solid material to achieve the final structures with the required mechanical strength.



After printing, the silica structures were functionalised with amine to create the CO₂ adsorption capacity, and bench-scale tests were carried out in a single column packed with 12 printed structures

stacked together. The model calculations to predict the structured sorbent performance in breakthrough testing were validated by the experimental results. The structured sorbents showed a stable performance during 50 repeated cycles.

The CO₂ sorption properties of the printed HTC were tested and showed equal capacity compared to the reference material. The pressure drop of 3D-structured HTC was much lower and the mass transfer rate was higher, compared to conventional packed beds. The experiments also showed that the regeneration step is more efficient using structured HTC compared to the packed bed. All these effects contribute to a productivity increase. Printed structures were tested during 225 repeated adsorption-desorption cycles and remained stable during these measurement, without changes in sorption capacity and catalytic activity.

A **Computational Fluid Dynamics** (CFD) model was developed to study the performances of 3D-structured sorbents. CFD models were built for conventional packed bed and for monolith shaped sorbents, and the comparison between models and experimental results showed a good correlation for both situations. Different channel geometries for the 3D-sorbents were studied and based on the calculated breakthrough curves and mass transfer zones, the cone shaped channel geometry shows the most efficient utilization of the adsorbent.

CO₂ capture system modelling was done for the ImmoAmmo system, analysing the post-combustion capture application. The modelling results on the 6-step vacuum swing adsorption (VSA) cycle, showed a productivity increase of a factor 2.5 for the structured sorbent, compared to a packed bed. Despite this productivity increase, without further optimization, the resulting total capture plant size for the structured solid sorbent is larger than a benchmark liquid amine post combustion CO₂ capture system, and its energy needs also remain higher than the benchmark. The study showed that it is very important to select sorbent materials having CO₂ sorption properties that closely match the applied sorption cycle conditions. With this precondition, the productivity can be optimised and energy needs minimised, to obtain improvements beyond the existing liquid amine-based capture systems.

The system modelling for HTC systems was done for application as pre-combustion capture system in a Steam Methane Reforming (SMR) based hydrogen plant. The productivity of the structured sorbent of 10 (mol/kg/hr) represents an increase by a factor 4 to 10 over packed bed configurations. The techno-economic analysis for this capture system in an SMR plant, showed that a strong advantage of the HTC system is the higher CO₂ avoidance rate of 60% vs 54% for the reference capture process. The cost of CO₂ avoided for of the structured HTC process is around 14% lower than the reference cost.

Market research on compact CO₂ capture applications was done to identify potential applications, the market needs, and end-user interests. The pre- and post-combustion CCU/S technologies and applications and the ecosystems of parties involved was mapped and interviews were held with end-users, technology and material suppliers, and engineers, scientists and experts. A business model based on the Business Model Canvas concept was developed to commercialize the technology. Most suitable at this time is the technology in a Standardised Unit (SU), or ISO container, for testing of the technology onshore, and possible future roll-out offshore.

The targeted productivity increase is substantiated through modelling and validated using the experimental test results, both for the ImmoAmmo and HTC development lines. The latter was only tested on small scale, as printing of HTC materials needs further efforts to improve the quality and reproducibility.

The results obtained from the experimental work, the model development and validation, and the system analysis, provided evidence that the targeted productivity increase of a factor 10 can be obtained and will lead to more compact capture technologies, and to reduced cost of CO₂ capture. To reach this productivity increase, a balanced combination of material sorption properties, structural details and the capture process design is required. Within the 3D-CAPS project, knowledge, experimental testing and modelling tools for structured sorbents were developed and can be applied in the further development of the concepts and in optimizing productivity, compactness and energy efficiency of future capture systems.

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2 Abbreviations and Units

BET	Brunauer-Emmett-Teller (adsorption theory)
BMC	Business Model Canvas
CCA	Cost of CO ₂ avoided
CCU/S	Carbon Capture and Utilisation /Storage
CCP	CO ₂ Capture Program
DEM	Demonstration
DIS	Dissemination activity
DLP	Digital Light Processing
FPSO	Floating Production Storage and Offloading
ImmoAmmo	Immobilised Amines -short for Amine functionalised Silica sorbent
IR-DRIFTS	InfraRed - Diffuse Reflectance Infrared Fourier Transform Spectroscopy
LCOH	Levelised cost of hydrogen
(K-) HTC	(potassium promoted) hydrotalcite
(a)MDEA	(activated) MethylDiEthanolAmine
MTZ	mass transfer zone
NMR	Nuclear Magnetic Resonance (spectroscopy)
SEM-EDS	Scanning Electron Microscopy with Energy Dispersive Spectroscopy
SEWGS	Sorption Enhanced Water Gas Shift
SMR	Steam Methane Reforming
SU	Standardised Unit
VSA	Vacuum Swing Adsorption
WGS	Water Gas Shift

3 Introduction

Step-changes are required to accelerate the introduction of CCS technologies, one of the overall goals of the ERA-NET ACT program. The project 3D-CAPS targets a productivity increase of an order of magnitude in two sorbent-based technologies for CCS. This is expected to lead to a substantial decrease in overall equipment size and costs of the capture systems.

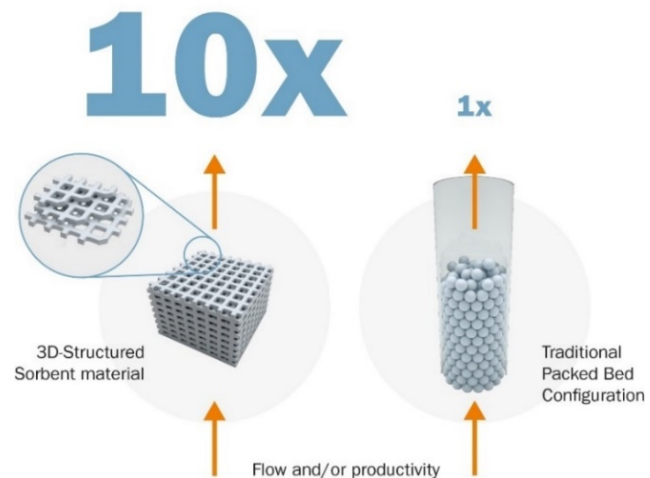
To achieve the productivity increase, the latest available technique is used for materials production: additive manufacturing, commonly known as 3D-printing. One bottleneck for traditional packed-bed solutions for sorbent-based CCS technologies is the trade-off between flowrate through the reactor, pressure drop and kinetics of the adsorption process. The use of 3D-printing allows material configuration solutions for solid sorbent-based CCS technologies, which are not available with current production technologies. 3D-Printing can deliver tailored materials with much improved heat and mass-transfer characteristics.

The two types of sorbent materials developed are hydrotalcites (HTC) for operation under high temperature and high pressure; and Amine functionalised Silica-Supported (ImmoAmmo) sorbents for operation in post-combustion conditions of low pressure and mildly elevated temperatures.

The objectives of the 3D-CAPS project are:

- To achieve a 10-fold productivity increase for two sorbent-based technologies in CCS
- To optimize sorbent shapes with Computational Fluid Dynamics (CFD) and other modelling tools, with direct realization in 3D-printed objects for testing under relevant conditions.

To achieve these objectives the first step in the project is to develop the 3D-printing technique and the supporting knowledge to enable the printing of structured materials based on HTC and on ImmoAmmo as active sorbents. Characterization and testing of the printed materials is performed to measure their properties and performances for application as CO₂ capture materials.



Simultaneously, a set of modelling tools is developed, which support the definition of structures with optimised geometries to improve the productivity, and will lead to CO₂ capture system designs that can utilise these improved productivities. The results of the characterization and testing of structured sorbents feed into the modelling activities and vice versa to generate optimised solutions.

The gains that can be made using the technologies of the 3D-CAPS project are benchmarked against reference and base-case technologies, following the methodology of the EU Benchmark Task for techno-economic evaluations of CO₂ capture systems.

As support and guidance to the technical developments, the market opportunities and business development for the innovative CO₂ capture technologies are systematically screened, assessed and elaborated using the Business Model Canvas methodology. This activity intends to lead to a direction and plan for the further development of the 3D-CAPS concepts.

The following chapters (4-7) describe the Work Package activities and key results achieved, followed by the discussion and conclusions. (chapter 8). The project impact and the collaboration within the consortium are described in chapter 9 and 10, respectively.

4 WP1: 3D Printing

Within the 3D-CAPS project, structured high surface area sorbent materials are prepared with Digital Light processing (DLP) 3D-printing. This technique uses an indirect slurry-based process that uses a photo-active material to initiate binding. The printer will form a layer of paste that will be illuminated layer by layer as a projected cross-section. After each layer, the structure will be moved up, and a new paste will be prepared.

To obtain the structured sorbents, slurries -mixtures of the sorbent, UV-sensitive monomer, photo-initiator and additives- are provided by Admatec. After printing, the objects are exposed to a debinding and sintering post- treatment to give them their final form and strength. For ImmoAmmo, the 3D-printed porous silica structures are sorbent supports that require functionalisation with amines after printing.

4.1 3D-printing hardware

Within the 3D-CAPS project a **state-of-the-art Admaflex 130 DLP unit for exclusive use within this project has been obtained by TNO**. At Milestone M1 in the beginning of 2018, the system (Figure 1) was ready to produce 3D-printed structures. During the project, scale-up of the production of the printer was established, by installing a larger printing area and light engine. With this upgrade, more structures can be printed at the same time, albeit with somewhat lower resolution (pixels from 50 μm to 75 μm) and light intensity (Figure 1). The resolution and intensity were still sufficient to print all the desired structure designs and materials in the 3D-CAPS project.

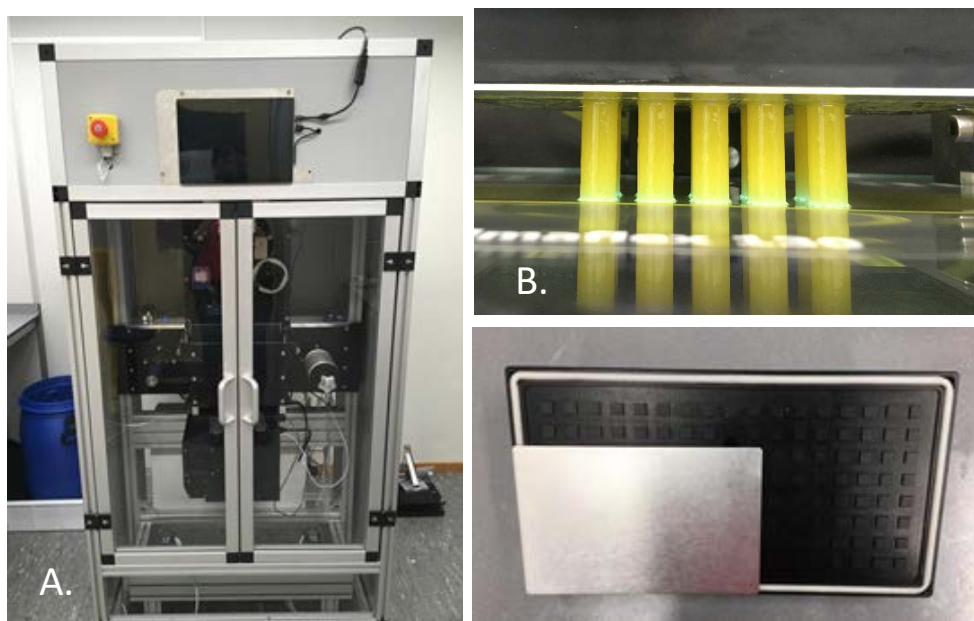


Figure 1: State-of-the art DLP printer. A: 3D-printing unit B: illustration of printing of monoliths and C. increased area for printing.

4.2 Silica

Silica containing slurries for printing were provided by Admatec. After a few iterations, a slurry that could be printed successfully was obtained. The post-processing method was optimised, finding a compromise between the strength and surface area of the resulting structures at 950 °C sintering. During the project various SiO₂ structures were dispatched to Sintef for testing. At the end of the project, some 50 foam structures (2,5 cm diameter and 4 cm height) were printed and sent to Sintef for final testing (Figure 2, A&B).

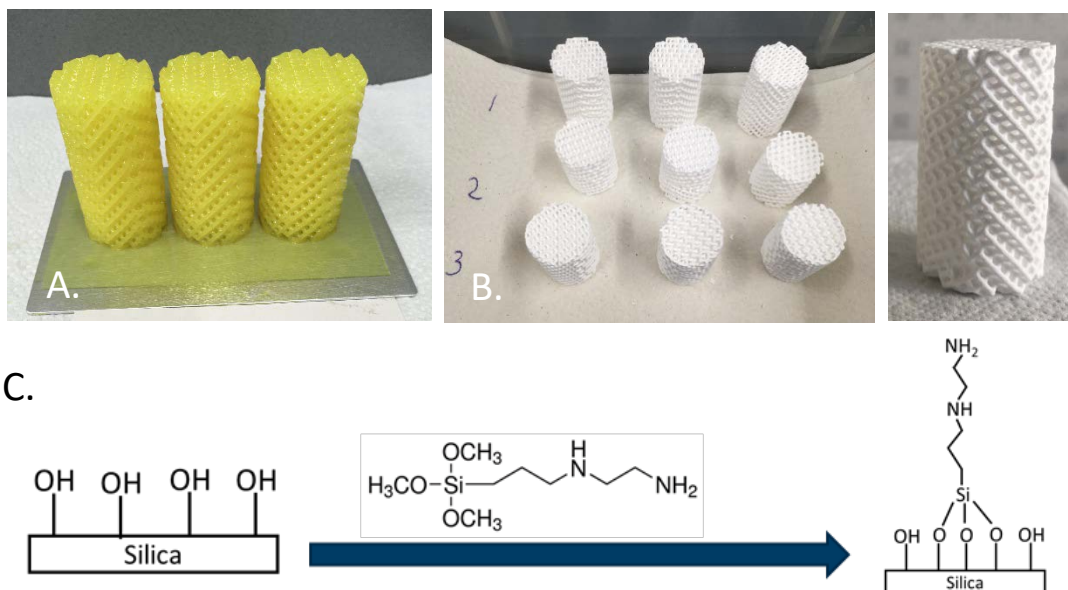


Figure 2: Silica printing and functionalisation. A: Final foam structures after printing B. Examples of foam structures after sintering. C: Functionalisation route for ImmoAmmo.

Finally, the functionalization of the structured SiO₂ materials with amines has been developed. Three different routes were investigated. The grafting procedure using trimethoxy silyl propyl ethylenediamine as an amine containing precursor (Figure 2, C), was found to lead to the highest CO₂ sorption capacity and was further optimised and used for testing.

4.3 Hydrotalcite

The printing and post-processing of the potassium promoted hydrotalcite (K-HTC) sorbent, proved to be more challenging than anticipated. Some issues (e.g. high viscosity of the slurry, reduced photo-activity over time) could be solved, but the recipes are still not completely reproducible. Especially, during post-processing, often cracks within the printed sample still appear. However, at the end of the project a potential solution was identified to strongly reduce this issue by adding an inorganic binder to the slurry.

Over the course of the project, sufficient K-HTC monolith structures could be obtained to perform testing at small scale, but unfortunately not enough good quality material could be produced for the testing at the scale that was anticipated in the project proposal (fully automated single column system of 2m high bed size). Still, essential elements of the performance improvement have been separately validated experimentally: maintaining adsorption capacity, pressure drop reduction, and lowering internal mass transfer resistance.

5 WP2: Test and characterization

This chapter describes the approaches and results obtained on testing the various 3D-printed materials and characterizing their properties.

5.1 Silica-ImmoAmmo

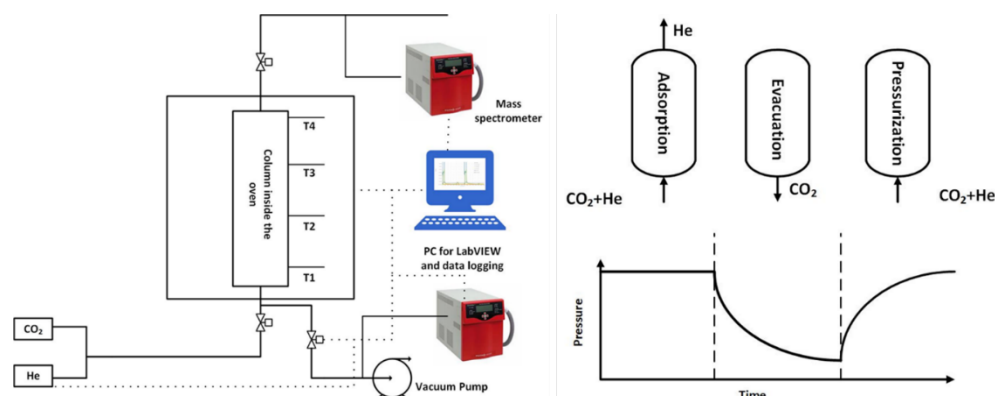
The 3D-printed and amine functionalised silica sorbents were characterised by BET, TG-MS, IR (DRIFTS), NMR, SEM/EDS, and CO₂ adsorption/desorption to study the surface area, degree of functionalization, the functionalization reactions, the surface morphology and composition, as well as the CO₂ capacity of the sorbents. The main results for the reference silica beads and the 3D-structured silica sorbents are summarised in Table 1 below.

Table 1. Summary of characterization results for the reference silica beads and the 3D-structured silica sorbents.

Silica support		BET (m ² /g) silica/granted sample	Grafted amine (mmol/g)	CO ₂ sorption (mol/kg at 10 kPa and 343K)	Crushing strength (N)
	Reference silica beads (Perikat, BASF)	328/118	3.8	0.93	99.3
	Final 3D printed foam	114/28	2.8	0.51	44.8

Bench scale tests were carried out with a single column packed with 12 printed structures. The schematic of the rig is shown in Figure 3. Breakthrough experiments were carried out with 15.5% CO₂ and rest Helium at 4 different temperatures from 70-100 °C (see Figure 4). Following breakthrough experiments, a simple 3-step PSA cycle comprising of adsorption, evacuation and pressurization was carried out. The breakthrough experiments were modelled with a 1D model and a good agreement was obtained between the experimental and simulated profiles. The CO₂ concentration during the evacuation step shows that it is possible to concentrate to about 90%. Further, the temperature swings reveal that the cyclic steady state condition is achieved after 15 cycles, see Figure 5. However, it should be remembered that the bench scale test does not have any provision for flow measurement and hence there was no provision to obtain CO₂ capture rate, productivity, and specific energy.

Figure 3: Schematic layout of the single column bench scale test-rig (left) and illustration of the pressure evolution of the 3-step PSA cycle.



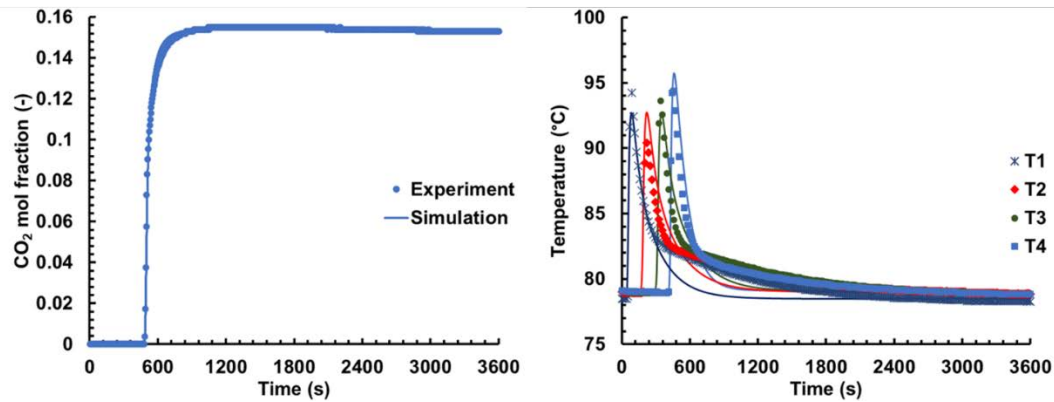


Figure 4: CO₂ concentration and temperature profiles in breakthrough testing at 80°C. Comparison of experiment and simulation

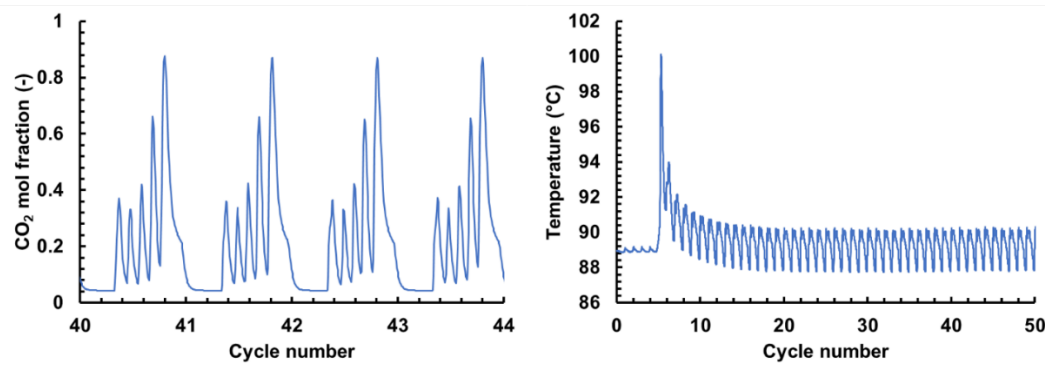


Figure 5: CO₂ concentration and temperature profiles in 3-step PSA cycle testing.

Preliminary simulations revealed that the 1D model was able to capture the breakthrough time and the temperature swings in the column during the breakthrough.

Figure 6 shows the temperature in the 3-Step VSA cycle for one thermocouple at the 50th cycle. The model is qualitatively able to predict the trend in temperature at various time durations. The minor differences could come from the use of an average temperature value and an arbitrary heat transfer coefficient value. Now, we are working towards improving the model predictions through calibration of sensors and the use of right heat and adsorption kinetics parameters from the breakthrough experiments and these results will be communicated through a scientific publication. It should be noted that the 1D model was able to capture the trends in the breakthrough experiments by only fitting the heat and the adsorption rate coefficient values. Preliminary PSA simulations were also able to capture the trends qualitatively. Therefore, this confidence in implementing the 1D model for the detailed process optimization done in WP3

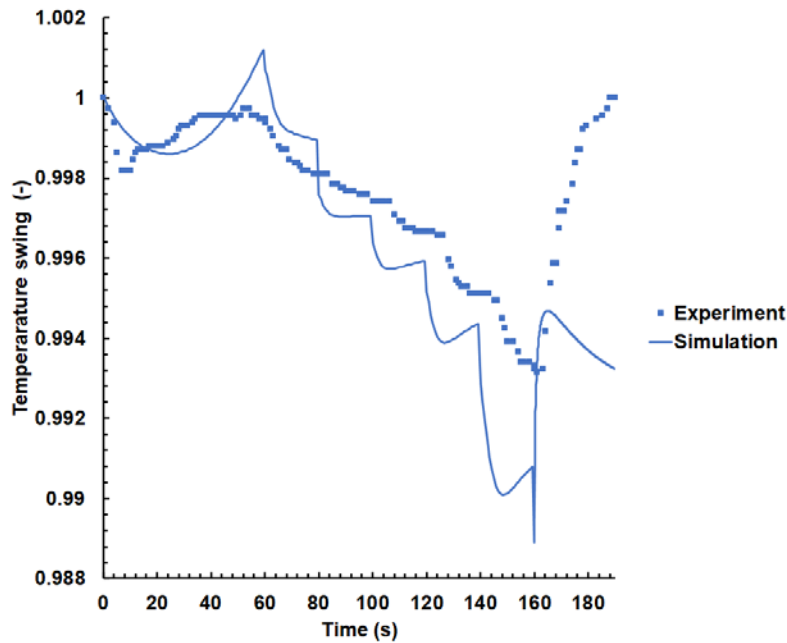


Figure 6: Comparison of simulated and experimental column temperature changes of the VSA process

After all the experiments we took out the column and studied the pressure drop across the columns. This data is compared with the measurements using a single structure of adsorbent. The pressure drop in the column is less than the one obtained using a single pellet. This implies two things, first, more sample is needed for accurate pressure drop measurements and second, the sample did not undergo significant attrition during testing.

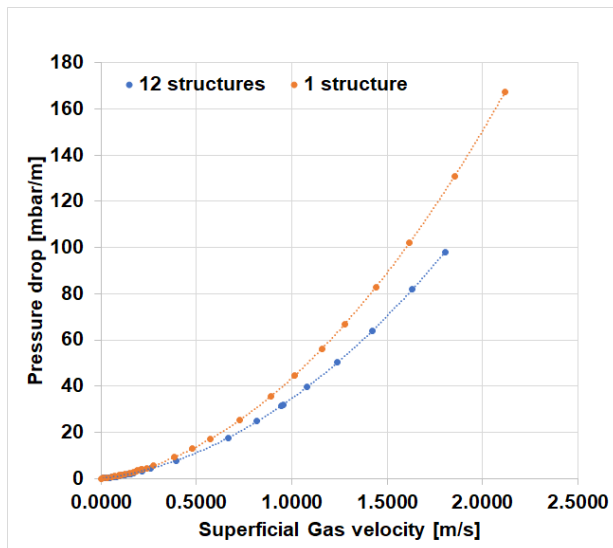


Figure 7: comparison of pressure drop for a single structured element and a column of 12 elements stacked on top of each other

5.2 Hydrotalcite

First, the effect of printing and post-processing on the properties of the printed K-HTC materials have been investigated. The printed sorbents were characterised by BET, SEM-EDS and capacity measurements. The capacity measurements of the materials after printing and post-processing at relevant temperature and pressure (400 °C and 20 bara) give **no indication of reduced capacity due to the printing process compared to the reference material**. Hereby, we meet the objective of having no significant loss of CO₂ capture capacity compared to the properties of the base powder material (<10% decrease of these intrinsic properties).

The effect of the shape of printed K-HTC on their application in SEWGS was also studied. The underlying physical phenomenon that is at the heart of the desired productivity increase in this project, is that when flowrate in a standard packed-bed increases, the mass transfer zone in the bed starts to become disperse and pressure drop increases. This is illustrated in Figure 8 below: at higher flow rates the breakthrough curve flattens which means the column needs to switch to regeneration mode more quickly and the length of unused bed increases. The shorter time on stream in adsorption mode at constant regeneration times reduces the effective productivity. Similarly, cycling times can be limited by pressure drop over the column. By using optimised 3D-printed sorbents with good mass transfer and limited pressure drop, flow rates can be increased to increase the productivity.

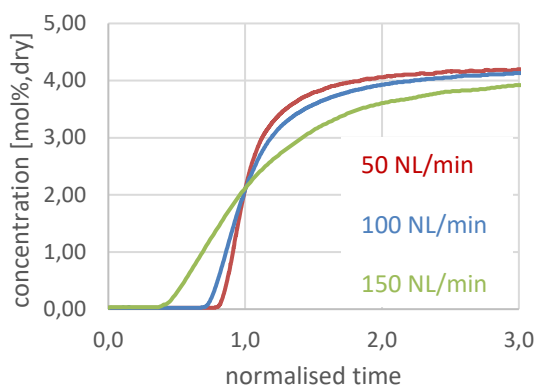


Figure 8. Graph showing how different flows influence the breakthrough curves in the packed bed as observed in previous experiments.

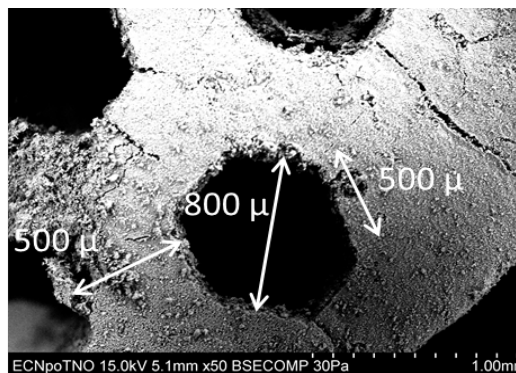


Figure 9. SEM picture of 3D printed K-HTC monolith with arrows showing the channel and strut sizes.

For SEWGS several monolithic structures were evaluated. The pressure drop over the monolithic structures was measured and proved to be significantly lower than for the pellets (Figure 10). As expected the size of the channels has a significant effect on the pressure drop. However, larger channels correspond to less material and thus the size of the channels needs to be optimised to obtain the highest productivity. Based on modelling results in WP3, the monolith with hexagonal channels of 0.5 mm and struts of 0.5 mm (Figure 9) was chosen to be investigated further, as this structure would potentially lead to a 8-fold productivity increase. A foam structure is also investigated to break the laminar flow in the channels.

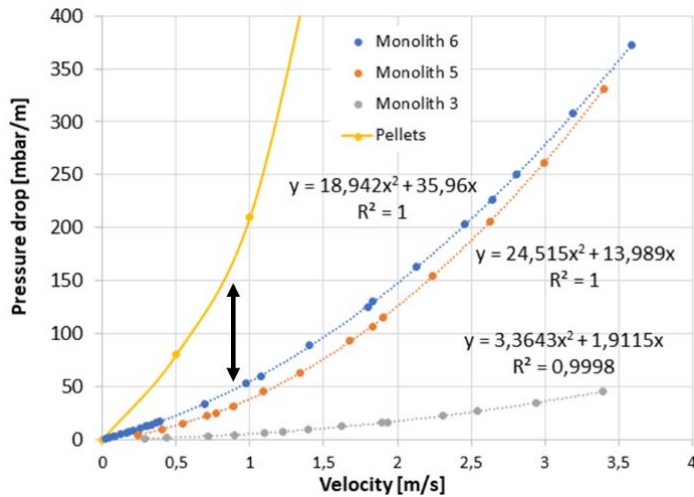


Figure 10: Pressure drop as a function of gas velocity in selected 3D-printed K-HTC monoliths with various channel sizes and pellets. Monolith 6 is the structure to be investigated further.

Due to technical issues with printing of K-HTC, described in 4.3, enough material could not be produced for the 2m high scale testing that was anticipated. Instead, smaller scale experiments were performed at ambient and pre-combustion conditions to prove the potential productivity increase (in combination with modelling).

The experiments at ambient pressure indicate **better mass transfer in the 3D-structured materials compared to the packed bed**, albeit more pronounced for the foam than for the monolith (Figure 11). This illustrates that more complicated structures (that cannot be produced by traditional shaping methods) can have added value compared to simple monolithic structures. The sieve fractions showed an even sharper break through as expected, due to smaller transport length. (Note that use of sieve fraction in a SEWGS cycle would lead to an unacceptably large pressure drop.)

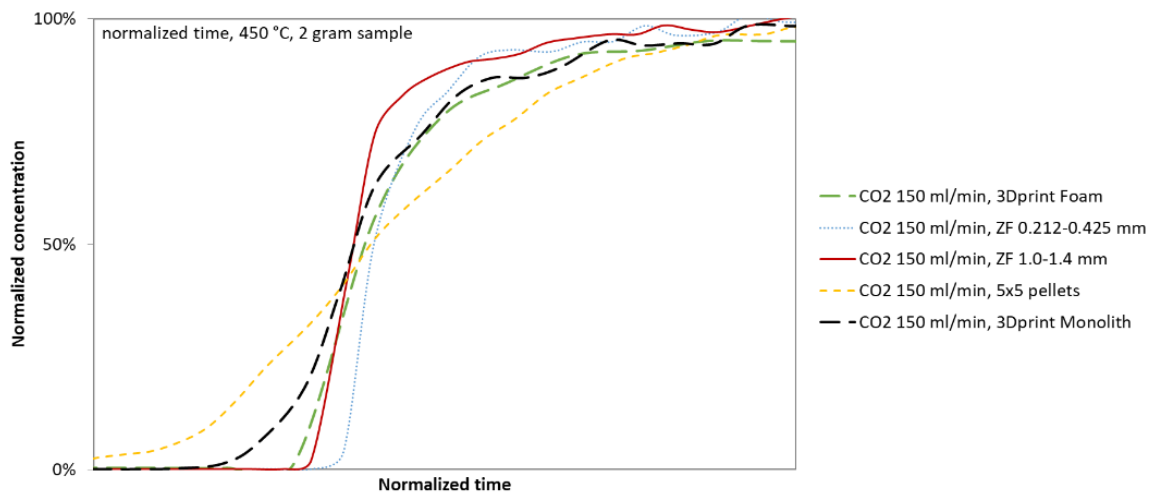


Figure 11: Breakthrough curves for 3D structured K-HTC versus pellets and sieve fractions at 150 mL/min.

At pre-combustion conditions, experiments showed that **regeneration is more efficient using structured K-HTC compared to the packed-bed, which will also result in a productivity increase**. Furthermore, **no decline in catalytic shift activity was observed for the printed sorbents when they were subjected to a syngas feed typical for H₂ production**. The obtained breakthrough times and curves feed into WP3 for model validation.

After all cyclic test (225 cycles) were performed the monolith structures were still intact and no significant capacity change was observed during the measurements. The foam structure, on the other hand, could not be recovered as a single piece from the reactor, instead K-HTC powder was collected. This underlines the need for further improving 3D-printing and post-processing procedures for K-HTC materials.

Overall, the combined results indicate that a productivity increase is possible by structuring (by maintaining sharp breakthrough curve at higher flowrates and more efficient regeneration in combination with low pressure drop). However, additional research is required to quantify the benefit, proof the technology on larger scale and improve the understanding of the effect of structuring on the productivity.

6 WP3 Modelling

This chapter describes the results of the modelling activities of the project. It contains CO₂ capture modelling on 3 levels; the very detailed modelling of the 3D-printed structures, and its impact on capture performance, the capture process modelling to develop designs for capture systems based on structured sorbents, and the calculations on the technical and economic feasibility of the innovative CO₂ capture systems.

6.1 CFD modelling

The main goal for using CFD modelling was the investigation of geometrical related effects and improvements on adsorption related mass transfer efficiency of CO₂ during the adsorption step of SEWGS, when using 3D-printed K-HTC structured bed reactors. The software used for model development and solving was COMSOL Multiphysics. The models solved equations for momentum, mass and heat transfer in time and space, with particular emphasis on CO₂ adsorption kinetics.

Initially, due to computational limitations, 2D geometric models were developed. Single channel models were chosen to further reduce the computational effort required; nevertheless, the models could provide valuable information on the entire reactor. Five different channel geometries (Figure 12) were considered: channels with a constant section (a), channels with reduced section at the inlet (b), channels with reduced section at the outlet (c), channels in a zig-zag configuration (d) and channels with angled sections (e). The comparison between channels was possible by considering the same operating conditions and the same mass of K-HTC adsorbent material. The normalized breakthrough curves in Figure 12 allow for a proper assessment of the mass transfer efficiency for each geometry. With the sharpest breakthrough curve profile, the cone shaped geometry with a smaller inlet section (c) showed the most efficient utilization of the adsorbent and least mass transfer resistance.

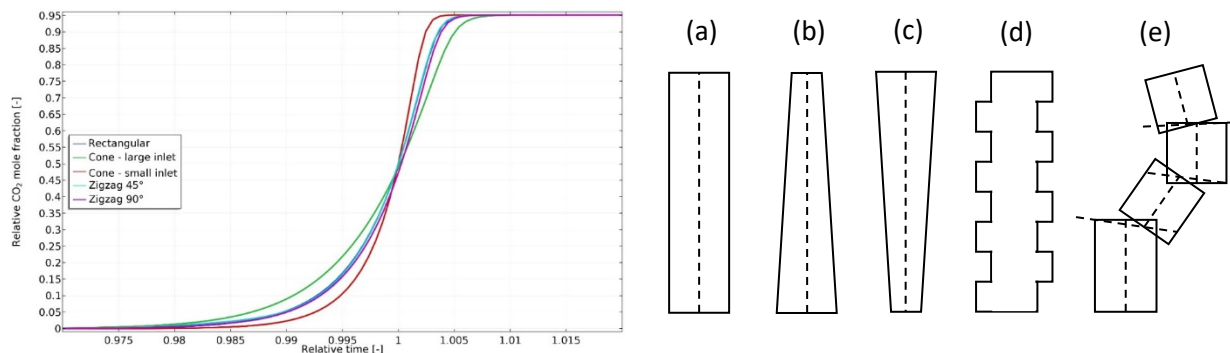


Figure 12: Normalized CO₂ breakthrough curves for 5 different geometries at 20 NL/min; (a)-(e) – simulated geometries.

In order to validate the developed CFD models, two validation scenarios were chosen: indirect validation using a packed bed configuration and direct validation of a bench-scale monolith configuration (for low- and high-pressure conditions). Validation was necessary before upscaling the model and moving on to a theoretical comparison of performance in SEWGS between packed bed and structured bed models on a pilot-scale.

When developing the packed bed model, the main challenge was that species transport and adsorption took place in dimensions of different orders of magnitude: macropores between the pellets and

micropores inside the pellets. A multiscale model was chosen for the packed bed reactor, where macroscale was represented by a 1D geometry, more specifically the bed height (Figure 13a), while microscale was also described by a 1D geometry, the pellet radius (Figure 13b). The packed bed was considered a homogenous domain and was modelled as porous media. Figure 12a shows a good agreement between experimental data provided by TNO and the model predicted breakthrough curves for the packed bed configuration.

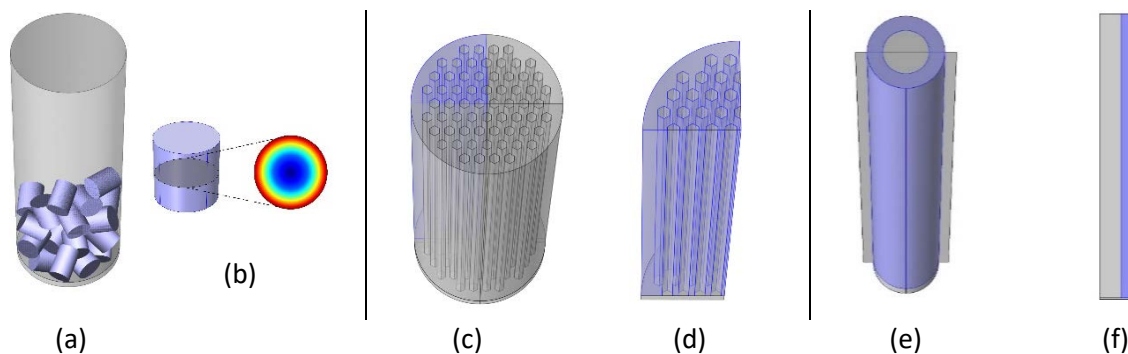


Figure 13: Schematic representations of the model geometries: (a) packed bed, (b) pellet, (c) 3D monolith, (d) 3D quarter monolith, (e) 3D single circular channel, (f) 2D-axisymmetric channel.

The model for the bench-scale monolith reactor was developed by taking into account the 3D-printed structure with hexagonal channels designed by TNO to undergo breakthrough experiments at low- and high-pressure values. 3D approach was chosen for the geometry of the bench-scale structured bed model, in order to properly highlight the complex nature of the process and phenomena involved. Instead of simulating the entire geometry (Figure 13c) and solving the full 3D equations, plane symmetry was used to split the structure into a quarter of the initial geometry (Figure 13d), and thus reducing the overall computation time by at least 4 times. Two computational domains were considered: the fluid domain and sorbent material domain modelled as porous media. Figure 14b illustrates a good fit between experimental results and model predictions for low- and high-pressure conditions.

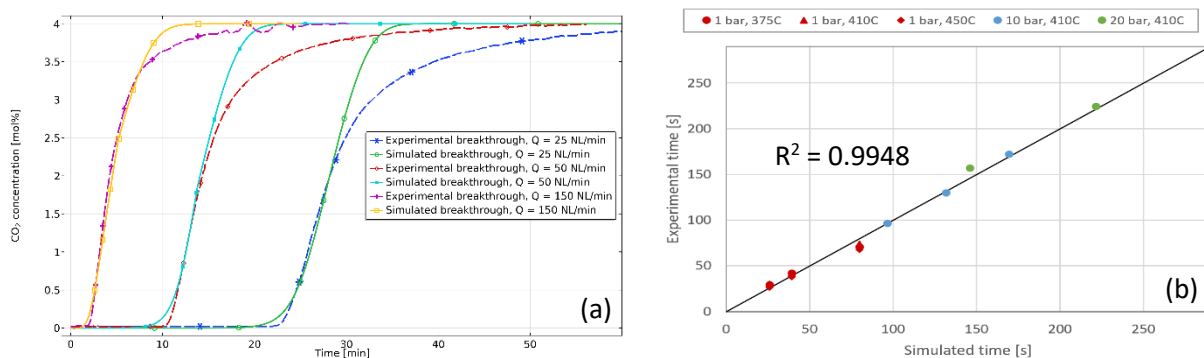


Figure 14: (a) Experimental vs. predicted CO_2 breakthrough curves for the packed bed; (b) parity plot showing the distribution of experimental vs. predicted values of CO_2 breakthrough times for the bench-scale structured bed.

To further evaluate the mass transfer efficiency of the bench-scale structured bed, three different channel geometry configurations (Figure 15a-c) were simulated at atmospheric pressure. Zigzag

configuration (a) showed the sharpest breakthrough curve and highest mass transfer rate, which can be attributed to mixing induced by geometric features.

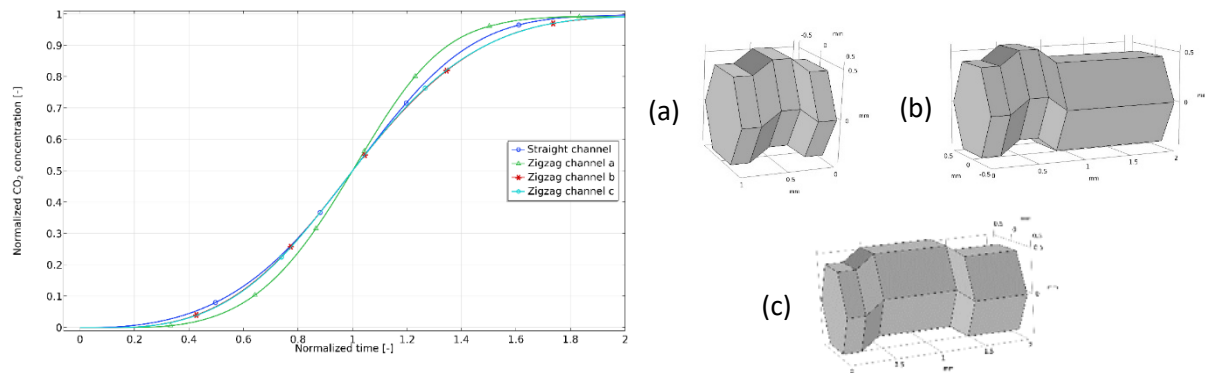


Figure 15: Normalized CO₂ breakthrough curves for 3 different geometries vs. straight channel predicted by the bench-scale monolith model at 100 NmL/min and atmospheric pressure; (a)-(c) – simulated geometries.

Finally, the bench-scale model was upscaled to a size comparable to the packed bed technology (~2 m). The adsorbent material mass was also identical, as well as operating conditions. With the assumption that a single channel (Figure 13e) could describe the entire reactor, a 2D-axisymmetric (Figure 13f) monolith CFD model was developed to study the fluid dynamics and mass transfer occurring in the adsorption stage of SEWGS, and thus enabling proper comparison of performance between packed bed and structured bed configurations in SEWGS. The sharpest curve profile (Figure 16a) was by far the curve predicted by the monolith model, with an almost ideal breakthrough curve profile by comparison, meaning the most efficient mass transfer was seen in the monolith configuration. Furthermore, the mass transfer zone (Figure 16b) calculated by the monolith model was considerably shorter than the packed bed one and the increase in CO₂ concentration after breakthrough was going to be substantially steeper for the monolith reactor, indicating a more efficient utilization of the bed.

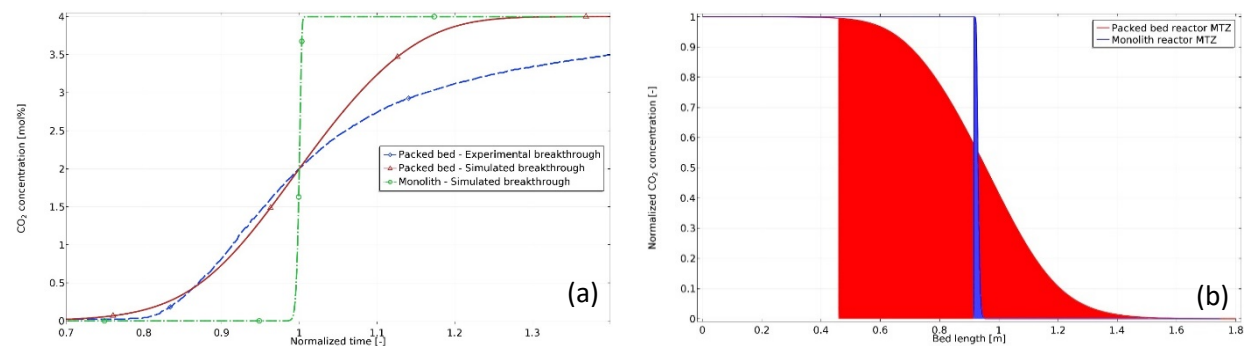


Figure 16: (a) Normalized CO₂ breakthrough curves predicted by the packed bed model vs. pilot-scale monolith model at 25 NL/min; (b) mass transfer zones calculated for packed bed (red) and monolith (blue) reactor models at 900 s simulation time.

6.2 Capture Process modelling

6.2.1 ImmoAmmo post-combustion capture

For the ImmoAmmo capture process, we have chosen a 6-step cycle with light product pressurization, light reflux and heavy reflux shown in Figure 17. The cycle consists of the following steps: Adsorption with feed, heavy reflux or rinse, co-current evacuation, counter-current evacuation, light reflux and light product pressurization. The light reflux and the light product pressurization steps are carried out with a part of the light product from the adsorption step. The entire stream from the light reflux step is refluxed to the rinse step to conserve the productivity.

1. Adsorption with feed, where preferential adsorption of CO₂ and H₂O occur. The remaining light product N₂ are collected as light products from the top of the column;
2. Heavy reflux or rinse, where a high concentration stream from the light reflux stream is introduced into the column to increase the CO₂ concentration in the bed and flush out any light product;
3. Co-current evacuation, to remove the nitrogen and oxygen from the column, the feed end is closed, and the column is evacuated from the product end and to enhance the purity;
4. Counter-current evacuation, where the column is evacuated from the feed end to remove the product CO₂;
5. Light reflux: The light reflux step is carried out with a part of the light product from the adsorption step to recover the remaining CO₂ adsorbed in the solid by pulling vacuum in the feed end. The entire stream from the light reflux step is refluxed to the rinse step to conserve the productivity. This stream will have a slightly higher concentration than the feed but less than that of the counter-current evacuation step;
6. Light product pressurization. The column is pressurised with the remaining light product from the adsorption step to prepare for subsequent feed step.

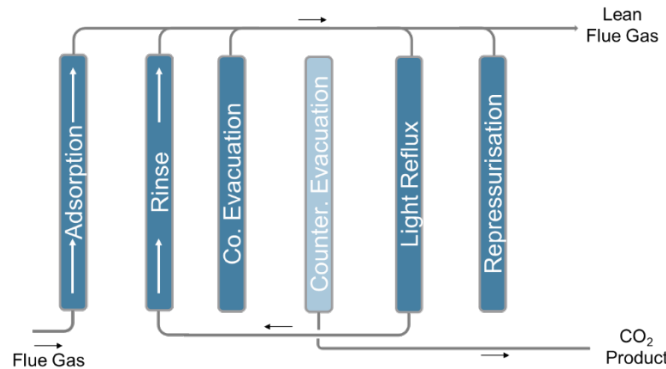


Figure 17: 6-Step VSA cycle for ImmoAmmo

Using the information from the lab scale tests in WP2, we have performed simulations for different flow rates for packed bed and a structured bed. The model equations are the same as those used in WP2 except for the following differences in the LDF coefficient and the absence of a reaction. The LDF coefficient is given by the following equation

$$k_{LDF} = \frac{3D}{w_c^2} \quad (1)$$

We have considered a coal fired power plant scenario with 15% CO₂ 5% H₂O and rest N₂ as a starting point for the hydrogen production case, since limited data of the system to be considered was available at the start of the optimization procedure. The flue gas is at ambient pressure and at 90 °C. The

performance of the pellet was compared to that of a monolith (equivalent 3D-printed adsorbent) with hexagonal channels of 0.5 mm width. The cycle times of the VSA cycles for the pellets and the structured adsorbent were 180 s and 72 s respectively. For the packed bed we assumed a length of 1m and 0.2 m in diameter and packed with 2mm particle size and the structured adsorbent was 1m in height and 0.2 m in diameter. The performance indicators are shown for the pellets and structures in the Figure 18 and Figure 19 below.

With the pellets, it was not possible to attain the desired capture rate (recovery) of 90% and purity of 95% beyond an inlet velocity of 0.6 m/s. In case of the structured adsorbent, it was possible to operate the cycle at 4 times the flowrate of 2.5 m/s. The 4 times improvement in feed flowrate resulted in 2.5 times improvement in productivity. It should be noted that the 4 times increase in velocity did not result in 4 times increase in productivity. This is due to the shorter cycle times for the structured adsorbent which meant that the moles of CO₂ treated is much smaller and therefore only 2.5 times increase in productivity.

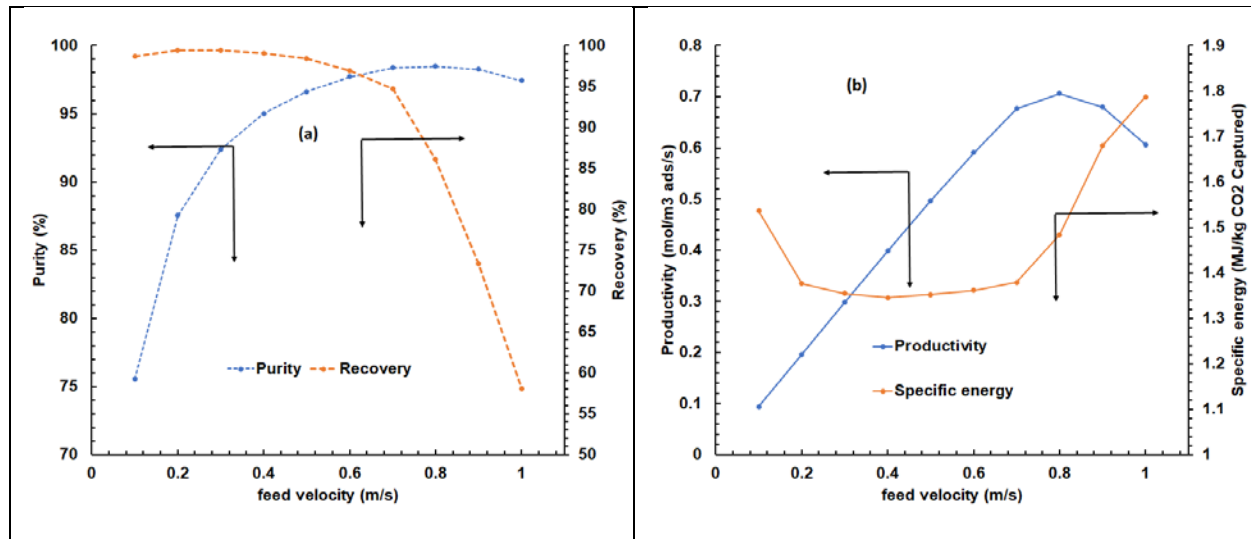


Figure 18. Effect of flowrate on (a) purity and recovery and (b) specific energy and productivity for a packed bed system.

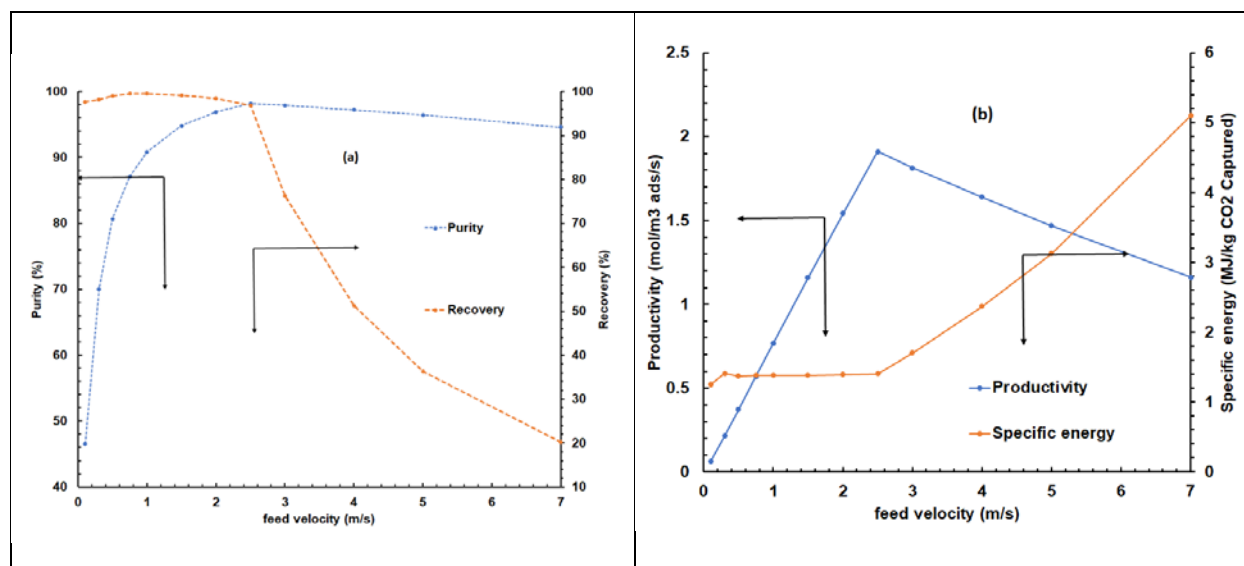


Figure 19. Effect of flowrate on (a) purity and recovery and (b) specific energy and productivity for a **structured adsorbent** system.

It should be noted that the specific energy consumption in Figure 19 is around 5 MJ/kg. This is a very high value and hence it re-iterates the importance of process optimization and **choosing the right material for CO₂ capture**. Therefore, detailed process optimization was carried out to identify the optimum operating conditions for two different adsorbents namely Amino Silane grafted SiO₂ and Lewatit OC. The objective was to minimize the specific energy and maximize the productivity of the 6-step VSA cycle. Detailed optimization was carried out using genetic algorithm and the output was plotted as Pareto front as shown in Figure 20. The results indicate that **an improvement in productivity from 20-90% was observed** and this improvement was more prominent in Lewatit OC than amino silane grafted sorbent. The minimum specific energy is 1MJ/kg on an electric basis (vacuum pumps) and this value is around 3-4 MJ/kg on a thermal basis. This is not an improvement over existing amine-based solvent and therefore a detailed study of mapping the best adsorbent to its best cycle is necessary.

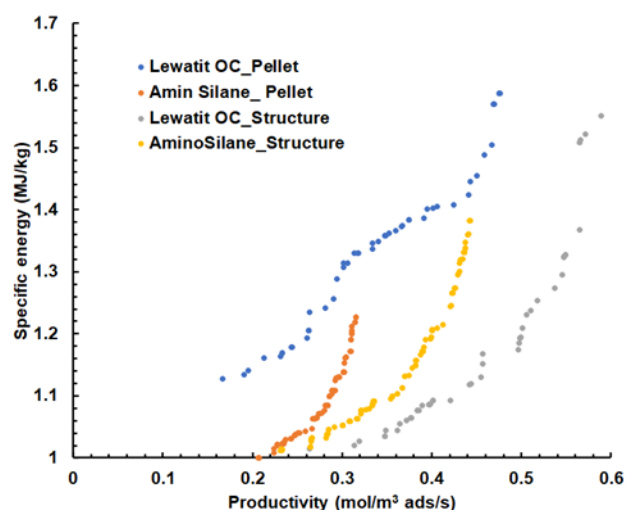


Figure 20 Comparison of productivity and specific energy consumption of amino silane and Lewatit adsorbents as pellet and as structured adsorbent

We have also explored zeolite 13X sorbent in pelletised form for CO₂ capture from a dry flue gas. In this case, Zeolite 13X was able to achieve much higher productivity than pelletised amine grafted sorbents at a lower specific energy. This would mean that with a structured 13X sorbent, significant improvement in productivity would be achieved. At ambient conditions, zeolite 13X can operate with 2-3% water in the stream and still achieve the desired performance targets as water would only be present in the 1st 10% of the bed.

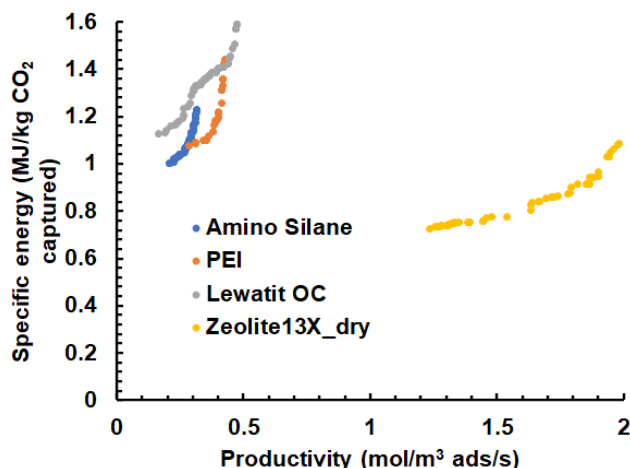


Figure 21: Productivity comparison between amines and zeolite 13X

6.2.2 Hydrotalcite pre-combustion capture

The pre-combustion option selected here is the Sorption Enhanced Water Gas Shift (SEWGS) process. The process functions by using potassium promoted hydrotalcite as CO₂ adsorbent that is catalytically active for WGS reaction. Due to the finite capacity of the sorbent for CO₂, multiple columns are required, such that when one column is saturated and enters regeneration, another column can be used for adsorption. There are a number of steps each column goes through to enable efficient conversion and separation. These are; adsorption, rinse, pressure equalisations (decreasing pressure), blowdown, purge, pressure equalisations (increasing pressure) and repressurisation, see Figure 22

During adsorption, feed gas enters the adsorption column at the top. The CO and H₂O in the feed gas is converted to CO₂ and H₂, while CO₂ is adsorbed. This simultaneous conversion and adsorption leads to a higher CO conversion. Subsequently, a high pressure co-current rinse is performed, in which part of the unconverted syngas in the column is replaced by H₂O. The use of rinse originates from PSA cycle design and is known to improve the CO₂ purity. After the rinse step, a number of pressure equalisation steps are carried out, to reduce the required compression energy. During the pressure equalisation, any CO₂ released should re-adsorb further downstream in the column rather than be transferred to another column. This ensures high carbon capture ratio during the next cycle. Additionally, the rinse gas is expanded causing syngas, that would otherwise become impurities in the CO₂ product, to be transported to another column that can use it in the upcoming adsorption step. During the blowdown step, relatively pure CO₂ product is collected. In order to further desorb CO₂, a low-pressure counter-current purge step follows. The purge steam effectively functions to free the bottom part of the column

from CO₂, thereby maintaining a high carbon capture ratio by decreasing the CO₂ slip to the H₂ product stream.

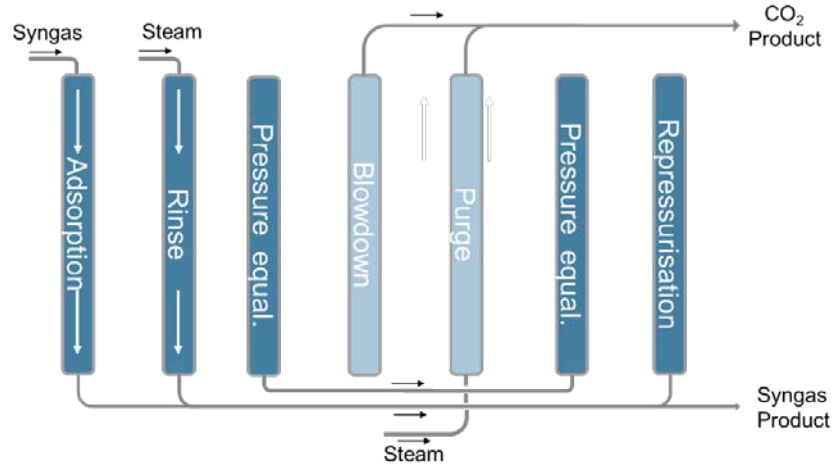


Figure 22 - Steps of a SEWGS cycle with co-current steam rinse

This batch process is transformed into a continuous process using multiple columns which are operated in pressure cycles, resembling the cycles of pressure swing adsorption process, allowing the constant production of separate H₂ and CO₂ streams. The purpose of this work is to evaluate the performance of the SEWGS cycle using structured reactors. Performance is defined as Carbon Capture Ratio (CCR), CO₂ purity (CP) and Productivity, formulated as shown in equation 2, 3 and 4. The evaluation is done based on the existing SEWGS reactor model developed for packed bed with modifications for a structured column.

$$CCR = \frac{CO_x \text{ in } CO_2 \text{ product}}{CO_x \text{ in feed}} \quad (2)$$

$$CP = \frac{CO_2 \text{ in } CO_2 \text{ product}}{\text{Total } CO_2 \text{ product} - H_2O \text{ in } CO_2 \text{ product}} \quad (3)$$

$$\text{Productivity} = \frac{CO_2 \text{ product flow } \left(\frac{\text{mol}}{\text{hr}} \right)}{\text{Total sorbent mass (kg)}} \quad (4)$$

Parametric Study

Cycle modelling of SEWGS is a complex undertaking given the high degrees of freedom in design of the process. The introduction of additional parameters in terms of the dimension of the microstructure (i.e. 3D-printed structure) adds further complexity. Therefore, the influence of microstructure dimensions is assessed, before embarking on a full cycle design. This is done by performing a sensitivity study on the parameters known to be affected by geometry to identify optimum microstructure dimension. With the target of 10-fold increase in productivity, then these parameters must also improve by an order of magnitude.

Productivity is defined with units of mol of CO₂ captured per kg of sorbent per hour, as expressed in Equation 1, which says that the productivity is proportional to the working capacity over the cycle time (Equation 2) meaning that the productivity is proportional to how quickly the working capacity can be used.

$$Productivity = \frac{n_{CO_2}}{m_{ads}} \cdot \frac{1}{t_{cycle}} \quad \text{Equation 1}$$

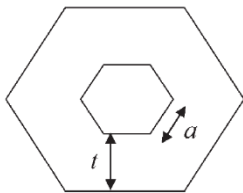
$$Productivity \propto \frac{WC}{t_{cycle}} \quad \text{Equation 2}$$

Based on the known sorption isotherm the maximum working capacity is estimated to be around 0.52 mol/kg. If this working capacity can be accessed in one hour, the productivity will be 0.52 mol/kg/hr, if accessed in 1 minute, it will be 31 mol/kg/hr.

The working capacity will only be used, if enough CO₂ is available for adsorption. This implies that to achieve higher productivity two things are required; (1) fast cycling and (2) higher throughput. The result of this is that the SEWGS system will experience higher gas velocity. Higher velocities influence the pressure drop and the mass transfer zone (MTZ). For the adsorption and regeneration, it is preferred to have a lower pressure drop and a shorter mass transfer zone. As the velocity increases, the pressure drop and length of the MTZ increases. The original definition of working capacity will need to account for the effect of the pressure drop and MTZ, expressed by Equation 3. In doing so, we link a limiting factor in productivity to the geometry of the structures used in adsorption. By exploring the functions f_1 and f_2 , we can establish more firmly the parameters for sensitivity study.

$$Productivity = k \frac{WC - f_1(dP) - f_2(MTZ)}{t_{cycle}} \quad \text{Equation 3}$$

f_1 is defined by pressure drop and the relation of the pressure drop to velocity and geometry. f_2 is more complex due to the multiple effects that result in the mass transfer zone. In principle, the mass transfer zone is influenced by the isotherm, the mass transfer mechanism and axial dispersion mechanism.



To achieve geometries for structured sorbents that combine a low pressure drop, with high mass transfer rates and show limited increase in the axial dispersion, a hexagonal channel (image left) is considered, and variations in the circumradii (a) and the thickness (t) are applied. This allows to study the impacts of geometry on the functions f_1 and f_2 , for a range of gas velocities, and provides guidance to identify geometries and dimensions for structured sorbents, that will give a productivity increase compared to packed beds. For the considered SMR application, this resulted in the selection of a hexagonal channel dimensions, combined with a gas feed velocity to be optimized for the SMR application. In order to test this, cycle modelling is performed with the selected structure parameters.

Cycle Modelling

The cycle modelling involved a sensitivity study with respect to total flowrate (as a proxy for velocity), total cycle time, steam consumption (purge and rinse) and purge duration. The final result and some key

observations are reported below. The final column dimensions selected to achieve the high productivity of 10 mol/kg/s based on modelling is shown in Table 2. The cycle design employs 2 pressure equalisations with a product repressurisation and has a total cycle time of 120s. This system achieves a CO₂ Purity of 95 mol% on a dry basis with a Carbon Capture Rate of and 84 %.

It must be noted that the design was optimised towards the high productivity and the cost of this is reflected by the high steam consumption. It is possible to optimize towards a lower productivity and decrease the steam consumption. This has not been considered in the current scope of the study. The overall carbon capture rate achieved is 84 % with respect to the CO₂ and CO entering the SEWGS system. It was observed that when performing fast cycling, the slip of CO increases as the kinetics of the shift reaction becomes limiting. This causes the overall capture rate to decrease, even if there is no slip of CO₂. This indicates that in order to achieve a high CCR at higher productivities, the kinetics of WGS reaction must be further explored.

Table 2 - Process Parameters for Modelling

	Reactor specifications		Mass flow	Number of columns	S/C rinse	S/C purge
	Height	Diameter				
Structured bed						
Hexagon: a=0.5mm t=0.25mm	10 m	1.5 m	30 kg/s	6	0.1	1.25

6.3 Flow-sheeting and Techno-Economic analysis

6.3.1 ImmoAmmo post combustion

The main objective of this activity was to evaluate the use of the 3D-CAPS technology for offshore post-combustion CO₂ capture. The chosen case was to place the capture system on an FPSO with two gas turbines running. The evaluated gas turbines are the standard LM2500+ gas turbines. The capture system was sized for one gas turbine, i.e. two modules would be needed. The process data for the adsorption process was received from SINTEF. The assumed available footprint on the FPSO was 18x20 m for one module. The footprint assumption is based on a study previously performed by Aker Solutions for Equinor for a liquid amine-based system sized for the same service.

The proposed 3D-CAPS system is a vacuum swing adsorption (VSA) process based on the solid amine on structured silica. The VSA requires several distinct cycle stages to operate, and the optimum system will therefore have several columns in parallel to ensure continuous operation. For the identified post-combustion capture process, 6 columns with a diameter of 8 m are needed. It is not possible to fit these columns into the assumed footprint as shown in the figure below. No further layout evaluations were performed, i.e. placement of additional required process equipment like vacuum pumps, compressors, intermediate storage vessel and pipe routing were not assessed.

In addition, a high-level evaluation of a mobile test container has been performed. Main equipment and piping have been sized to evaluate the fit into a container. This is further described in section 7.3.

6.3.2 Hydrotalcite pre-combustion capture in SMR

The integration of the 3D-SEWGS unit into the SMR flowsheet requires the balancing of heat demands across the plant and the capture unit. In the reference SMR system, excess heat is used for electricity generation and therefore, the integration will need to evaluate heat and electricity balance in tandem. The integrated process is shown in Figure 24

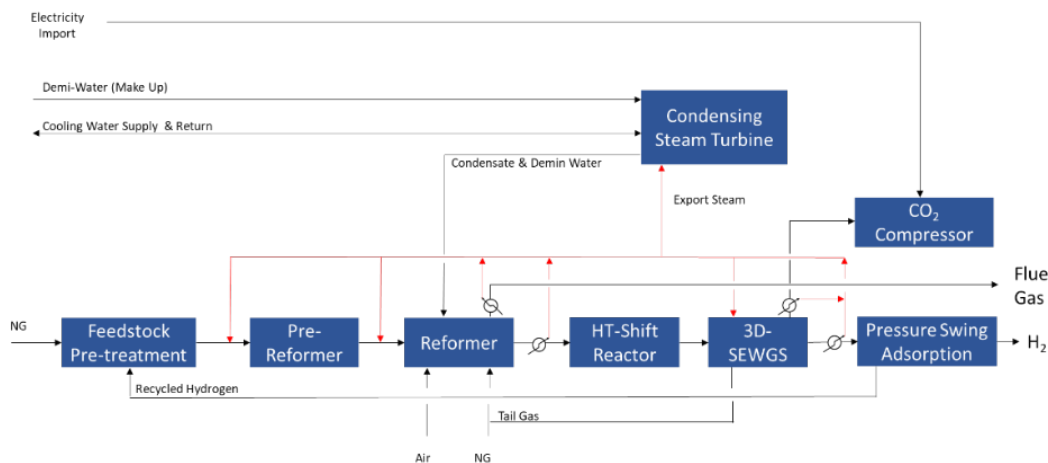


Figure 24 - Hydrogen production with carbon capture from natural gas using steam methane reforming

A preliminary technoeconomic evaluation was conducted on the 3D-CAPS SEWGS process based on the block flow diagram and the mass and energy balances from the process modelling. For the purpose of the technoeconomic evaluation, CO₂ capture from hydrogen production using activated MDEA (aMDEA) as the capture solvent is taken as the base case, to enable a direct comparison of the SEWGS process

with the more mature aMDEA. The aMDEA process is as presented in IEAGHG (2017). The reference process is the steam methane reformer (SMR) process without capture, presented in the same report.

A summary of the main process parameters is presented in Table 3, comparing the reference case, the base case and the 3D-CAPS SEWGS process. It can be seen from the table that when the feedstock natural gas input is kept the same as the reference case, the SEWGS process leads to slightly higher hydrogen production (about 1% higher) than the reference case and the base case. This is due to the extra water-gas shift reaction that takes place in the SEWGS reactors. This also leads to higher CO₂ being captured than in the base case.

Table 3 – A summary of the main parameters comparing the reference case, base case and the 3D-CAPS SEWGS process

		Reference (no capture)	Base case (capture aMDEA)	Capture with 3D-CAPS SEWGS
Natural gas (feedstock)	tonnes/h	26.2	26.3	26.2
Natural gas (fuel)	tonnes/h	4.3	5.3	5.1
Natural gas (total)	tonnes/h	30.6	31.6	31.3
H₂ production	Nm³/h	100,000	100,000	101,122
Gross power (cogen)	MW _e	11.5	6.7	3.6
Parasitic power load	MW _e	-1.6	-5.2	-5.1
Net power export	MW_e	9.9	1.5	-1.5
CO₂ emissions	Million tonnes/year	0.67	0.31	0.26
CO₂ avoidance	%	0%	54%	60%
Electricity penalty due to capture	GJ_e/tonne CO₂	0.00	0.70	0.83

A preliminary cost estimate for the 3D-CAPS SEWGS process was conducted using the main equipment items, based on the process mass and energy balances. A challenging aspect in the cost estimate is the rather high design temperatures required for the adsorption columns, which limits the choice of the material of construction. The total direct material cost for the 3D-CAPS SEWGS process add up to M€ 8,3 which is about 15% lower than that of the aMDEA process. The smaller column sizes and the absence of pumps to move fluids within the process, contribute to a lower overall cost for the major equipment items in the 3D-CAPS SEWGS process.

A breakdown of the major cost components and how these are combined to give the total plant cost for all the sections of the hydrogen plant and the 3D-CAPS CO₂ capture process is made. The total capture plant cost of M€ 18.9 for the 3D-CAPS SEWGS process is about 9% of the total cost for the whole SMR-Hydrogen plant of M€ 194.1.

Using these cost estimates the levelised cost of hydrogen production (LCOH) for the processes with and without CO₂ capture were estimated. The methodology and assumptions were kept the same as those

used in the IEAGHG 2017 report on steam methane reforming (SMR) process with CO₂ capture, to enable a direct comparison of the results generated here and those presented in that report.

The main LCOH and cost of CO₂ avoided (CCA) for the SEWGS process are presented in Table 4, in comparison with the numbers for the base case aMDEA process. The two processes have very similar LCOH, because the lower capital cost of the SEWGS process and the slightly higher hydrogen production are partly offset by the higher energy requirement of the SEWGS process. The strong advantage of the SEWGS process appears to be the higher CO₂ avoidance rate of 60% vs 54% for the aMDEA process. The CCA of the SEWGS process is around 14% lower than that of aMDEA.

The capital cost of the CO₂ capture section is only a small fraction of the total capital cost of the whole hydrogen production plant. This means that the ability to influence the CCA through (further) cost reduction in the SEWGS process is limited. An important uncertainty in the current analysis is the cost of the 3D-printed structures for the 3D-CAPS SEWGS process, which is currently assumed to be no more than the cost of the amines for the aMDEA process.

Table 4 – Levelised cost of hydrogen (LCOH) and the cost of the CO₂ avoided for the 3D-CAPS SEWGS process compared with the base case (aMDEA)

		Base Case (aMDEA)	3D-CAPS SEWGS
CAPEX (Total Plant Cost - CO ₂ Capture)	€million	22.1	18.9
CAPEX (Total Plant Cost - Whole SMR Complex)	€million	201.8	194.1
CAPEX (Total Capital Requirement)	€million	263.9	253.8
Levelised Cost of Hydrogen (LCOH)	€/Nm³	0.1352	0.1345
Cost of CO₂ Avoided	€/tonne	47.2	40.4
SEWGS Cost of CO ₂ Avoided vs aMDEA		-	86%

Given the results above, an important area of investigation for the future is a configuration of the plant that aims to maximise energy efficiency rather than CO₂ capture productivity. Further work should also be carried out on the assessment of the cost of the 3D-printed structures.

7 WP4 Business development

The 3D-CAPS project was based on the bold premise that through the use of 3D-printing technology, reactors of chemical plants and their physical footprint could be reduced by an order of magnitude, and consequently, costs of the reactor and the balance of plant could be reduced by the same order of magnitude, or more.

1. 3D-cat has mapped the ecosystem of players involved in the application of this technology, and have interviewed representatives in all areas including: Oil&Gas, chemical, power, medical (customers); Hardware, 3D-printer and paste suppliers (jobbers), reactor-designers, catalyst-manufacturers (technology suppliers); scientists and experts (Figure 26 in Chapter 7.1);
2. 3D-cat has completed a business model to commercialize the technology based on the Business Model Canvas concept. This business model has been improved during various iterations, after having completed interviews with suppliers, customers and partners in the eco-system of pre- and post-cumbustion CCU/S technologies and applications (Figure 27 in Chapter 7.2);
3. 3D-cat has identified spin-off/alternative applications making use of this technology, of which the most suitable at this time is the Technology in a Standardised Unit (SU), or ISO container, for testing of the technology on-shore, and possible future roll-out off-shore. (Figure 29 in Chapter 7.3);

3D-cat was tasked to make a plan for the commercialization of the technology developed in the project. Various companies and experts were interviewed, and the most important findings are discussed below.

7.1 Market analysis

4. Business Model: at the heart of the Ecosystem

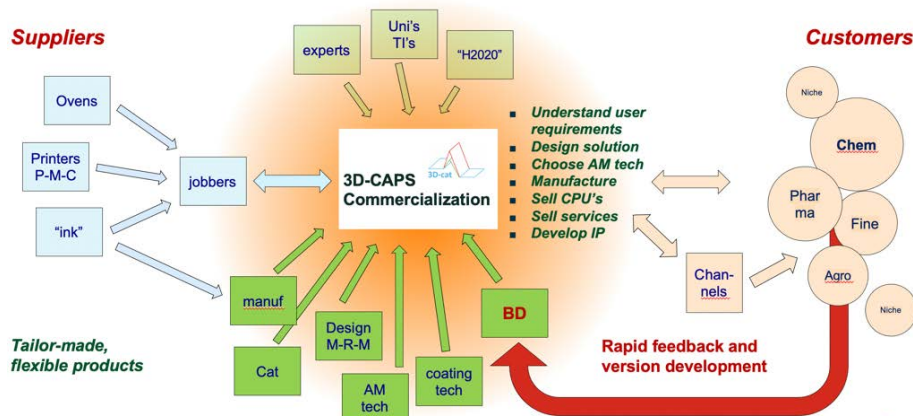


Figure 25 The eco-system covers multiple industries

Interviews indicated that customers with needs were quite unfamiliar with possible solutions available from the Additive Manufacturing (AM) industry. Similarly, AM players (Ceram, Lithoz, Admatec) had difficulty finding applications for their technology without the help of experts familiar with both their target markets and AM technology. Furthermore, large customers (Shell, Tata, BP) seeking technology for large industrial applications often did so via or with Engineering, Procurement and Construction (EPC) contractors (such as Aker, Technip, Wood). The EPC's were often stimulated by the end-customers to integrate new technologies in their solutions. The catalyst companies played a smaller role in the 3D-CAPS project, often simply as a supplier of standard catalyst or sorbent. This industry could play a larger role if they would let go of the current paradigm of supplying (small) catalyst parts and supply Chemical Processing Units (CPU's), or, (part-)reactors.

The lower cost of the new products made possible by this technology will replace proven products in existing markets, and in doing so create additional value. Customers (in O&G and Chemicals), where uptime of manufacturing is measured in tenths of percent, are amongst the most risk-averse in industry.

To consider replacement of current technology (and legacy assets) by new technology, such as 3D-CAPS, new technology must be *completely* de-risked before acquired and implemented. On the other hand, and consequently, gains of tenths of percent will be equally substantial. As 3D-CAPS technology promises a bold *order of magnitude* improvement, there is willingness to test the technology, initially at small scale.

More exciting from the perspective of implementing new technology is to look at applications where the smaller footprint enabled by this technology opens new markets for products that cannot be made today. Examples include the removal of CO₂, offshore (Figure 27), from exhaust gas for power generation, and, even more promising, from natural gas, reducing the need for pipeline investment, because current

technology requires a footprint only feasible onshore. The savings in pipe-line investment, and penalties for non-removal of CO₂ from exhaust gasses for power production dwarf the cost of the 3D-CAPS technology. Obviously, the technology will also be tested first onshore, in a SU. Interestingly, the SU has dimensions that make direct implementation on offshore structures, such as FPSO vessels, and O&G rigs realistic.

In the course of the project, the result of the system study for offshore post combustion capture (6.3.1), became available. The core learning from this study is that the vessels become large, take up a big footprint and the high degree of vacuum needed for the desorption dictates huge actual gas volumes with accompanying big dimensions of ducting, valves, actuators and other facilities. When the type of adsorbent material requires this vacuum in the desorption step, this results in capture system dimensions, as well as energy consumption, being larger than the benchmark liquid amine process.

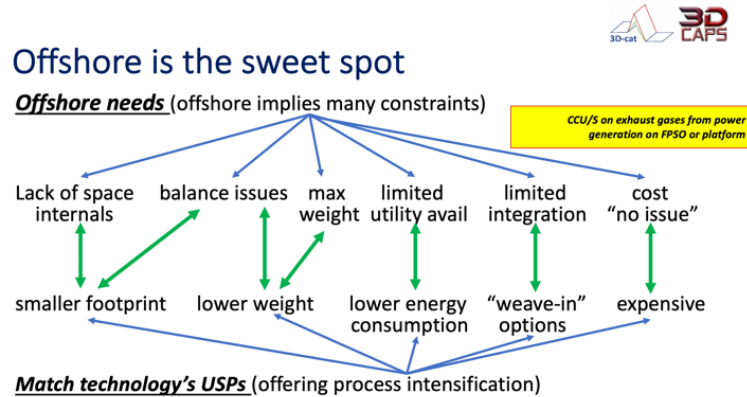


Figure 26 offshore application is the sweet-spot for compact systems

- Potential customers for the main (large-scale) applications have confirmed in interviews that such reduction of capex, opex and physical footprint is compelling, and the cost advantage of such process intensification (i.e. smaller footprint) in certain applications could be well over two orders of magnitude (e.g. CO₂ removal from offshore bad gas (followed by injection), reducing the need of pipelines to and from onshore alternative technology solutions, or CO₂ removal from offshore power generation, followed by injection);
- These customers have stated that they would want to see proof that such technology would work as advertised, and that they would want to test the technology themselves.

7.2 Business plan

- With Deliverable D4.1 we have explained and published the conceptual 10x reduction of size;
- For the pre-combustion application, TNO has theoretically verified that at least an 8x reduction of size is possible (SEWGS). For the post-combustion application, SINTEF has theoretically verified that at least an 3x reduction of size is possible (immo-ammo);
- Within the timeframe of the project, i) UBB was not able to verify this based on CFD calculations; ii) TNO was not able to verify the 8x improvement empirically; iii) SINTEF partially verified empirically that the 3x reduction was possible. Please refer to the technical section in this report.

Cost-effective structured sorbents and catalysts are enablers for designing more compact equipment in industrial plants, decrease steel usage and therefore they are key for cost competitive equipment design. This creates a clear opportunity. We have used the canvas model (Figure 27) to explore the exploitation strategy for a SU application, i.e. putting the technology in an ISO-container for testing.

ISO container product - New Business Model Canvas

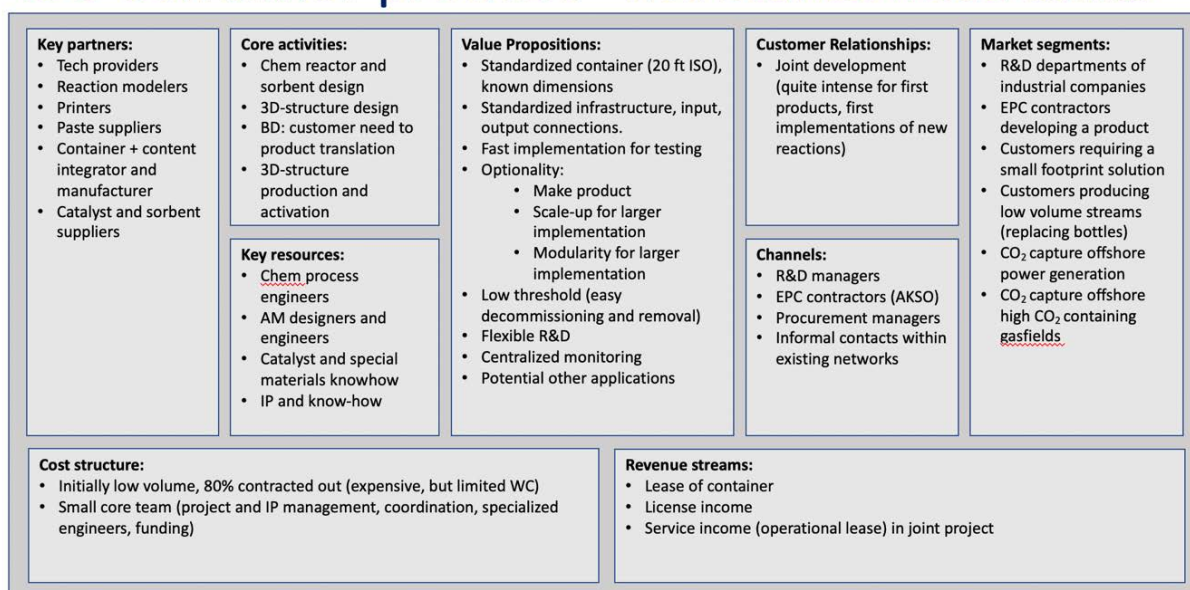


Figure 27 Business Model Canvas for the SU application

3D-cat has also identified spin-off/alternative applications making use of this technology, of which the most suitable at this time are small scale pilot reactors and applications for R&D labs at universities and technology institutes.

3D-cat has also identified, and designed with the help of a patent attorney, the intellectual property equivalent of the SU, akin to a Technology Transfer Office (TTO). Commercial members of the consortium (i.e. AKSO) have suggested that without such TTO, the commercialization would suffer delays, as customers would have to negotiate with different entities individually to obtain the different licenses to the technology required for implementation, without getting assurance that the negotiated package would be complete. Also, the availability of jointly developed foreground IP would be facilitated

by such TTO, as the DESCA model does safeguard IP (inventor owns) but does not facilitate commercialization. A blueprint for such TTO has been developed by 3D-cat, but implementation was a bridge too far (TNO has decided to claim Inventorship of certain technology under its own name, and supply access under the DESCA rules).

7.3 Next phase development

It is clear that catalytically active materials, sorbents, heat-transfer and mixing elements produced with 3D-printing have a larger remit for use than just CCS technology. 3D-cat has produced a position paper on the use of these materials in catalytic reaction processes (e.g. steam reforming or other H₂ related technologies) captured in deliverable D4.1. AKSO has not produced a position paper on the use of these materials in offshore processing of hydrocarbon, as originally intended to be captured in deliverable D4.2, but instead has produced cost estimates supporting Task 4.3 (Blueprint for TRL6 demonstration plant), see Figure 29.

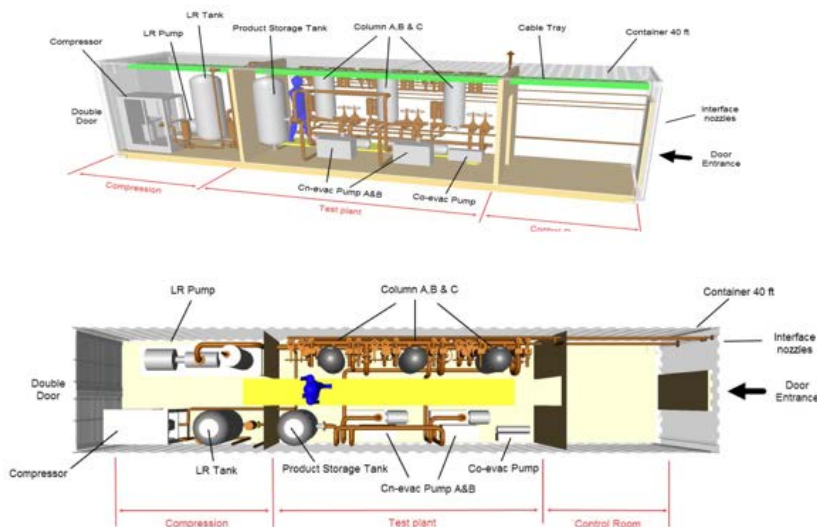


Figure 28 SU design for the ImmoAmmo capture process

In order to progress the 3D-CAPS technology, a plan for a technology demonstration at an industrially relevant environment has been defined. Based on the experiences of the experimental and calculative activities, a first preliminary basis of design for a TRL6 demonstration plant for carbon capture applications has been made and costed by AKSO.

Further a complete overview of all relevant

aspects, like footprint of plant, demands for electricity, cooling water and other utilities, total weight etc. have been identified. The total cost for a TRL6 demonstration plant is estimated to be between M€ 1 to M€ 2.

- The most relevant product definition, and the next step for the two main applications is to offer the technology in one or more Standardised Units (SUs), being 20' ISO-containers, which can be sold to potential customers to test the technology on customer's site. A first such sale would be supported through a follow-on project (a plan for TRL6 demonstration activities);
- SINTEF has supplied mass and energy balances for post-combustion technology in two SUs;
- TNO has supplied mass and energy balances for pre-combustion technology in two SUs;
- Aker has supplied 50% cost estimates for such products;
- Potential partners for such a follow-on project have been identified.

[slide of April 2nd 2020]

Product definition – standardised unit

"20' ISO container for testing"

Factor 4-10 productivity increase

Pre-combustion SEWGS with HTC

- Capacity 1 m³ nett capture volume
- 85% capture rate
- 4000 t/a CO₂ removed (10 mol/hr)
- Number of columns: 6 → 3
- Main constraints:
 - channel design of 3D-structure
 - lower water % in feed stream
 - < 95% CO₂ in effluent stream
- Other issues:
 - presence of catalyst to convert CO to CO₂ supports CO₂ removal
 - reproducibility of HTC printed structures (due to post-processing)
 - maximum possible diameter and height of 3D-structure ?

Factor 2.5 productivity increase

Post-combustion with immo-ammo

- Capacity 1 m³ nett capture volume
- 85% capture rate
- 5000 t/a CO₂ removed (12 mol/hr)
- Number of columns: 6 → 4/3
- Main constraints:
 - high level of vacuum for desorption consumes energy (steam purge helps ?)
 - different adsorbent (Zeolite X) or immo ammo sorbent
 - 400 ppm O₂ in effluent stream
- Other issues:
 - temperature control, sorbent stability
 - heat management between columns
 - Water removal if zeolites are used

12-10-2020

More detailed conversations coming weeks to further define 20 ft container contents, address constraints and confirm capacity

9

Figure 29: Outline for the further development of the 3D-CAPS technologies

SINTEF has taken the initiative to lead the next step and define such project. New partners, in addition to the core technology providers, should include companies that have competences and experience designing and building the technology within the constraints of a SU (i.e. one or more ISO-containers), potential customers, and an entity to commercialize the technology (both IP and the first SUs built).

The proposal for such a project is submitted to the ACT III call. In addition, it can be advertised to a potential customer directly, by the Business Development organisation of one of the current members of the 3D-CAPS project.

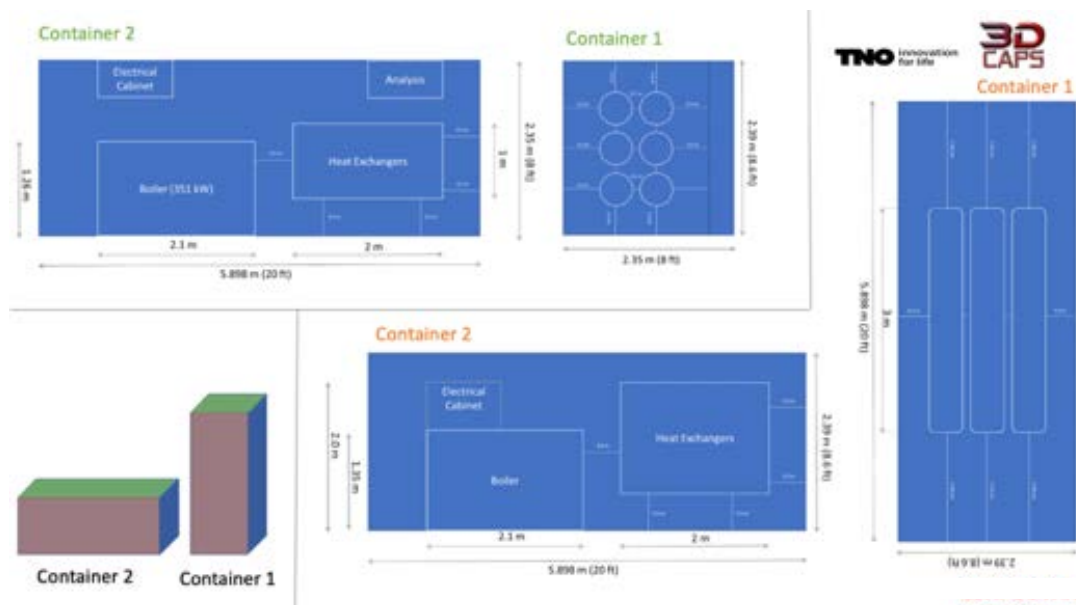


Figure 30: Basic lay-out of containerised 3D-CAPS SEWGS CO₂ capture system

The next step development would be to define a project to come to a basic design of an SU application (Figure 30) of either a pre-combustion test unit (see Figure 31), a post-combustion test unit, or both, as there are synergies in developing both at the same time.

8 Conclusions and recommendations

As a technological basis for the project, a Direct Light Processing (DLP) 3D-printing machine was successfully put in operation to produce 3D-printed structures of both silica and hydrotalcites. Good quality printed structures of silica as support for amines were repeatedly obtained for further characterization and testing in the project. 3D-printing of HTC materials was more challenging and printed structures could be prepared only for small scale testing. Printing of HTC materials needs further developments, e.g. to include inorganic additives, to improve the quality and reproducibility. In addition, other 3D-printing techniques can be explored, with a future view of printing on a larger scale.

Testing of the structured sorbents was performed to characterise their properties for CO₂ capture applications. The challenges in the testing of the structured materials were related to the available laboratory test-equipment, that was originally designed for pellets and powder testing in packed bed configurations, and needed adaptations to allow testing in higher gas velocities to achieve good quality testing and measuring of structured beds performances.

Modelling activities provided insight in the relationship between structure and capture system performance. The developed models for structured sorbents performance were validated with the results from lab-scale tests, and further used to predict the CO₂ capture system performances for HTC and ImmoAmmo. These models were useful in developing and analysing capture system designs. System modelling results also showed that it is very important to select sorbent materials having CO₂ sorption properties that closely match the targeted applications. Only then, the productivity can be optimised and energy needs minimised, and system improvements obtained beyond the existing liquid amine-based capture systems.

Market research on CO₂ capture applications mapped the market needs and end-user interests for compact capture solutions. A business model based on the Business Model Canvas concept was developed to commercialize the technology. Most suitable at this time is the technology in a Standardised Unit (SU), or ISO container, for testing of the technology onshore, and possible future roll-out offshore.

With the results obtained from the experimental work, the model development and validation and the systems analyses for ImmoAmmo and HTC the key objectives of the project were reached. Evidence was built that the targeted productivity increase of a factor 10 can be obtained, leading to more compact capture technologies, and to reduced cost of CO₂ capture. The optimised sorbent shapes, derived from modelling, were realised in actual 3D-printed structures that were tested under simulated process conditions.

The activities and results from the project indicated that to achieve this productivity increase, a balanced combination of material sorption properties, structural details and the capture process design for the targeted CO₂ capture application is required.

The knowledge, experimental testing and modelling tools for structured sorbents developed in 3D-CAPS can be applied in the further development of the structured concepts and in optimizing both productivity (compactness) and energy efficiency of future capture systems.

9 Project Impact

The results of the 3D-CAPS project have shown that a productivity increase of a factor 3 to 10 for solid sorbent CO₂ capture systems can be obtained. Implementing this will result in more compact capture systems, with reduced investment costs compared to conventional packed bed capture systems.

Although still at low TRL level, and further technology development is needed to proof the performance and reliability of these solutions at a larger scale, the results of this work demonstrate the **improvement potential** of both capture technologies, when optimizing the structures of a solid sorbent for specific process conditions.

Facilitate the emergence of CCS

This result contributes to facilitate the emergence of CCS, as it allows lowering the CAPEX and thus reducing financial barriers to invest, as well as reducing the cost of captured CO₂, as evidenced in the pre-combustion capture case in 6.3.2. Reducing this cost of CO₂ captured, leverages with the financial incentives (either taxations or penalties) to reduce emissions.

Reducing the required volume of capture systems, as a result of the productivity increase, also generates opportunities for CO₂ capture in applications where space/volume is limited and/or expensive. This holds specifically for offshore applications as well as for many existing industrial sites, where plot space for future capture systems were not included in the original plant design.

Strengthen the competitiveness and growth of EU companies

From end-user perspective, the competitiveness of EU companies, adopting CO₂ capture technology, can grow, once carbon neutral products and services are valued properly on the world market, related to political development. Potential growth of EU companies is possible for those investing in the novel capture technology development and introducing it to the market.

Other environmental impacts

The development and use of thermally stable solid sorbents for use in CO₂ capture systems, could replace some of the liquid amine capture systems, reducing the need for amine recovery and replacement.

Chances for commercialization

The 3D-CAPS technology needs further development before being commercially applied. In the development path to commercialization, opportunities exist to introduce small scale solutions to specific customers, also outside the field of the CO₂ capture. At the current TRL level, joint developments with targeted end-users are needed to progress the technology. These activities are included in the next phase development proposal, which is prepared for ACT.

10 Collaboration and coordination within the consortium

The 3D-CAPS project is carried out by a consortium of partners from industrial end-users, a technology provider, an SME, and research and academic institutes from across Europe. The team was built to include the appropriate skill sets, experience, expertise and interests to develop this technology further, such as:

- Solid background knowledge in advanced manufacturing technology for producing 3D-structured materials
- Existing infrastructure to test materials up to the TRL5 level
- CFD modelling and cycle design capabilities
- Understanding of factors and network to successfully implement the technology the field

The consortium members had distinct roles in carrying out their tasks, with well-defined areas, where results produced by one member were used as input for activities for other members. A specific **characteristic** of the project was the **broad scope of activities** to be covered, ranging from

- acquiring in depth knowledge on structure-properties relationships, covered by CFD modelling (UBB)
- developing the art and science of 3D-printing of structured sorbents by DLP technology (TNO)
- testing and characterising structured sorbents (SINTEF/TNO)
- capture system modelling and design (SINTEF/TNO)
- technical and economical assessments (CCP/AkerCC)
- Business development (3D CAT)

The collaboration between the partners was established in the face-2-face (f2f) consortium progress meetings, held every 6 months. In these meetings the progress and results were shared and openly discussed to understand their meaning and value for the project. Between the f2f meetings, online consortium progress updates were made. These meetings were well attended by all consortium partners, and ACT-representatives throughout the project. Within the work packages frequent working meetings between the partners involved in specific tasks were held. Fruitful collaborations (and exchange of students) were established between UBB and TNO on modelling activities and validations, between SINTEF and TNO on the development of 3D-printed structured sorbents and their characterization, between Aker and SINTEF on ImmoAmmo capture system integration and between CCP and TNO on SEWGS system integration and analysis.

A special role was assigned to 3D-CAT, to elaborate on the business development for the project, making them to involve all partners in combining the right technology with the right applications. Their role strengthened the collaboration between the partners during the project and resulted in identifying future business opportunities for innovative capture technologies, as well as the steps needed to reach these.

Carrying out the project in a European consortium, and with CCP partners Chevron and Petrobras also located in North- and South America was effective in identifying and extending the range of potential CO₂ capture opportunities, with a variety of techno-economical boundary conditions.

The coordination of activities of the project was addressed in the frequent working meetings between the partners. Specific coordinating efforts were needed once it became clear that delay occurred in delivery of the technical development steps and testing, effecting model validations as well as on the techno-economic analysis. By extending the project duration to accommodate the delay in technical developments, and also the delay caused by the COVID-19 pandemic, resolved this and valuable results could be generated and used in the last months of the project.

11 Signature

S. van Loo
Research Manager

R. de Boer
Author

Appendix

A: Project dissemination

A1: PUBLICATIONS

Hans Willemsen, Robert de Boer, *Additive Manufacturing of 3D ceramic structures for a step change in performance in industrial application as sorbent and catalyst*, Carbon Capture Journal, 73, February 2020

V. Sandu, I. Dumbrava, A.M. Cormos, A. Imre-Lucaci, C.C. Cormos, P. Cobden, R. de Boer, *Modelling of a rectangular channel monolith reactor for sorption-enhanced water-gas shift*, Environmental Engineering and Management Journal, 19, 2020, 2, Accession Number: WOS:000531733600003, ISSN: 1582-9596;

A. Soit, I. Dumbrava, V. Sandu, A.M. Cormos, *Modelling and Simulation of Water Gas Shift Reaction using COMSOL Multiphysics*, Studia Universitatis Seria Chemia, LXIV, 4, 2019, 19-29, DOI: 10.24193/subbchem.2019.4.02, ISSN: 1224-7154.

V. Sandu, I. Dumbrava, A.M. Cormos, A. Imre-Lucaci, C.C. Cormos, P. Cobden, R. de Boer, *Computational fluid dynamics of rectangular monolith reactor vs. packed-bed column for sorption-enhanced water-gas shift*, 29th European Symposium on Computer Aided Process Engineering, Eindhoven, The Netherlands, 16 - 19 June 2019, Book Series: Computer Aided Chemical Engineering, Volume: 46 Pages: 751-756, DOI: 10.1016/B978-0-12-818634-3.50126-0, ISSN: 1570-7946;

Shreenath Krishnamurthy, Anna Lind, Aud Bouzga, Joanna Pierchala and Richard Blom, *Post combustion carbon capture with supported amine sorbent*, Chemical Engineering journal 406 (2021) 127121.
Available online

Shreenath Krishnamurthy, Jurriaan Boon, Carlos Grande, Anna Lind, Richard Blom, Robert de Boer, Hans Willemsen and Gabriel de Scheemaker, *Screening supported amine sorbents in the context of post-combustion carbon capture by vacuum swing adsorption*, Chemie Ingenieur Technik, Manuscript under review by journal

Shreenath Krishnamurthy, Richard Blom, Anna Lind, Carlos Grande and Ibrahim Ali. *Potential improvement in productivity with structured sorbents* : Submitted to CCP for review (Same as CCP book chapter contribution)

Paper on SINTEF-PSA experiments with TNO yet to be prepared. Tentative date of submission : spring 2021.

Contributions to CCP4 Book: 3 Chapters in preparation.

A2: CONFERENCE PRESENTATIONS

Conference	Participant(s)	3D-CAPS Contribution
2020		
14th Mediterranean Congress of Chemical Engineering 2-5 June Barcelona, Spain https://www.mecce.org/	Sintef, TNO	Anna Lind, Soraya Sluijter, Shreenath Krishnamurthy, Carlos Grande, Robert de Boer, Richard Blom <i>3D printing of amine grafted silica sorbents for increased productivity in CO₂ capture processes</i> , cancelled due to COVID-19
The XXIV International Conference on Chemical Reactors CHEMREACTOR-24 30 August-4 September 2020 Milan, Italy	TNO, Sintef, UBB	S. N. Sluijter, J. Boon, J. James, S. Krishnamurthy, A. Lind, R. Blom, C.A. Grande, A. M. Cormos, V. C. R. Sandu, R. de Boer, <i>3D-Printing of Adsorbents for Increased Productivity in Carbon Capture Applications (3D-CAPS)</i> Accepted as oral presentation, but cancelled due to COVID-19
GHGT-15 5-8 October 2020 Abu Dhabi https://ghgt.info/	TNO, Sintef, UBB	S. N. Sluijter, S. Krishnamurthy, A. Lind, R. Blom, C.A. Grande, A.M. Cormos, A. Imre-Lucaci, R. de Boer, <i>3D-Printing of Adsorbents for Increased Productivity in Carbon Capture Applications (3D-CAPS)</i> Abstract accepted, postponed to 2021
The 15th Conference on Sustainable Development of Energy, Water and Environment Systems (SDEWES) 1-5 September 2020, Cologne, https://www.cologne2020.sdewes.org/	UBB	V. Sandu, A.M. Cormos, A. Imre-Lucaci, I. Dumbrava, C.C. Cormos, R. De Boer, S. Sluijter, <i>CFD Modeling of Sorption Enhanced Water-Gas Shift Process in Monolith Reactors</i> ,
2019		
Fundamentals of Adsorption (FOA13) Cairns – Australia 26-31 May 2019 https://foa2019.com/	Sintef, TNO	S.Krishnamurthy, A.Lind, R. Blom, C.A.Grande, R. De Boer, S.N.Sluijter, P.D.Cobden: <i>3D printed adsorbents for carbon capture applications: Characterization and process simulation</i> Poster presentation
ESCAPE29 29th European Symposium on Computer Aided Process Engineering, Eindhoven, The Netherlands, 16 - 19 June 2019, https://escape29.nl/	UBB, TNO	V. Sandu, I. Dumbrava, A.M. Cormos, A. Imre-Lucaci, C.C. Cormos, P. Cobden, R. de Boer, <i>Computational fluid dynamics of rectangular monolith reactor vs. packed-bed column for sorption-enhanced water-gas shift</i> , Book Series: Computer Aided Chemical Engineering, Volume: 46 Pages: 751-756, DOI: 10.1016/B978-0-12-818634-3.50126-0, ISSN: 1570-7946;
TCCS-10 Trondheim- Norway 17-19 June 2019	Sintef, TNO	Anna M. Lind, Soraya N. Sluijter, Bjørnar Arstad, Shreenath Krishnamurthy, Carlos A. Grande, Paul D. Cobden, Robert de Boer and Richard Blom <i>Development of 3D printed amine grafted silica adsorbents for CO₂ capture – adsorbent</i>

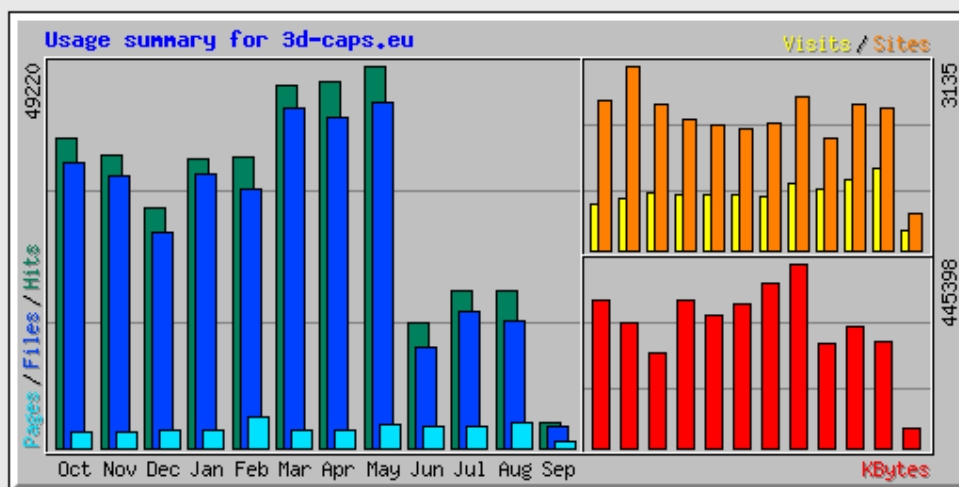
		<i>preparation, performance and potential applications</i> Oral presentation
Europacat Aachen, Germany, 18 – 23 August, 2019. http://europacat2019.eu/	TNO , Sintef, UBB	S. N. Sluijter, S. Krishnamurthy, A. Lind, R. Blom, C.A. Grande, A.M. Cormos, A. Imre-Lucaci R. de Boer <i>3D-printing of Hydrotalcite for Increased Productivity in Productivity in Sorption-Enhanced Water-Gas Shift Reaction</i> Poster presentation
12th European Congress of Chemical Engineering ECCE12 Florence, Italy 15-19 September 2019 http://www.ecce12-ecab5.org/	TNO , Sintef, UBB	Soraya Sluijter, S. Krishnamurthy, A. Lind, R. Blom, C.A. Grande, A.M. Cormos, A. Imre-Lucaci, P.D. Cobden, R. de Boer, <i>3D printing of Hydrotalcite and Amine Functionalised Silica Adsorbents for Increased Productivity in Carbon Capture Application</i> Poster presentation
ACT Knowledge Sharing Workshop 2019 Greece November 6-8, 2019 http://www.act-ccs.eu/events/	TNO	R. de Boer, 3D CAPS, Project overview presentation
5th Post Combustion Capture Conference Kyoto, 17-19 th September 2019	3D-CAT , Sintef, TNO	J.A.M. Willemsen, G.F. de Scheemaker, P.D.Cobden, R. Blom, R. de Boer, <i>Additive Manufacturing of ceramic 3D-structures for CO₂ capture. Poster presented:</i>
12th International Conference Processes in Isotopes and Molecules (T3: Energy Efficiency and High-Tech Engineering) Cluj-Napoca Romania, 25-27 September 2019 http://pim.itim-cj.ro/	UBB , TNO	V. Sandu, I. Dumbrava, A.M. Cormos, A. Imre-Lucaci, C.C. Cormos, S.N. Sluijter, R. de Boer, <i>CFD modelling of structured sorbent configurations in Sorption-Enhanced Water-Gas Shift,</i>
Americal institute of Chemical Engineers Annual meeting (AIChE 2019) Orlando Florida 11-15 Nov 2019	SINTEF	Shreenath Krishnamurthy, Richard Blom, Carlos Grande, Anna Lind, Soraya Sluijter, Paul Cobden and Robert De Boer, <i>Evaluation of structured adsorbents for carbon capture applications.</i> https://www.aiche.org/conferences/aiche-annual-meeting/2019/proceeding/session/psatsa
2018		
CHEMICAL ENGINEERING & 3D PRINTING Paris - France 7 September 2018 https://efce.info/European_Forum.html	Sintef , (Carlos Grande) 3D Cat TNO	SINTEF presented results from another project. Others attended symposium.
ICCE 2018 , Iasi, Romania October 31-November 2 http://www.ch.tuiasi.ro/ICCE2018/ 4th International Conference on Chemical Engineering (ICCE 2018) - Innovative materials and processes for a	UBB	V. Sandu, I. Dumbrava, A.M. Cormos, A. Imre-Lucaci, C.C. Cormos, <i>Modeling of a rectangular channel monolith reactor for sorption-enhanced water-gas shift,</i>

sustainable development		
GHGT14 Melbourne- Australia 21-24 October 2018 http://www.ghgt.info/	BP	3D-CAPS was presented as one of the CCP projects.
ACT Knowledge Sharing Workshop 2018 Niederaußem, Germany, Tuesday, October 13, 2018 http://www.act-ccs.eu/events/	TNO	Robert the Boer presented project progress and results
2017		
ACT Knowledge Sharing Workshop 2017 Bucharest, Romania, Tuesday, October 24, 2017 http://www.act-ccs.eu/events/	TNO	Jaap Vente presented project goals and plan

A3: DISSEMINATION AND COMMUNICATION ACTIONS

Action	Link to website or External Audience Sharepoint	Responsible
Development of ACT-3D-CAPS Logo		TNO
Project website *	https://3d-caps.eu/	TNO
Questionnaire	https://3d-caps.eu/sample-page/questionnaire/	3D-CAT
Peer reviewed publications	ACT-3D-CAPS Publication list	All partners
Press release	10X ADSORPTION PRODUCTIVITY INCREASE THE AIM AS CCP JOINS 3D PRINTING TECHNOLOGIES PROJECT	TNO
Internal TNO News		TNO
Flyers	ACT-3D-CAPS Information Flyer	TNO
Banner		TNO
Participation in workshops/conferences	ACT-3D-CAPS Conference list	All partners
Participate in joint activities H2020/ACT Projects	ACT progress meetings	
Position paper	3D CAT Position Paper	3D CAT
Time lapse	ACT-3D-CAPS Time Lapse	TNO
ACT-3D-CAPS activities in CCP Annual Report 2019	https://www.co2captureproject.org/reports/Annual_report_2019.pdf 3 chapters to be submitted to CCP booklet	
CCP Factsheet	CCP project Factsheet 3D-CAPS	BP, Chevron, Petrobras
ACT-3D-CAT Presentation for Dissemination	ACT-3D-CAPS Presentation for Dissemination	TNO

* Usage Statistics for website 3d-caps.eu d.d. 8 September 2020



Summary by Month										
Month	Daily Avg				Monthly Totals					
	Hits	Files	Pages	Visits	Sites	KBytes	Visits	Pages	Files	Hits
Sep 2020	420	334	100	42	615	49816	342	800	2673	3365
Aug 2020	650	525	106	44	2419	258028	1393	3290	16297	20172
Jul 2020	653	569	93	38	2466	292327	1189	2885	17669	20251
Jun 2020	539	433	91	34	1896	250278	1043	2748	13006	16189
May 2020	1587	1432	98	36	2623	445398	1138	3048	44420	49220
Apr 2020	1572	1421	76	30	2156	399394	909	2294	42650	47167
Mar 2020	1503	1413	74	30	2065	348399	953	2296	43815	46599
Feb 2020	1289	1148	134	32	2119	318383	933	3904	33304	37386
Jan 2020	1202	1136	71	30	2229	357901	954	2213	35216	37276
Dec 2019	997	898	71	31	2466	231459	971	2224	27854	30922
Nov 2019	1260	1167	65	29	3135	300761	870	1955	35019	37804
Oct 2019	1283	1187	65	25	2557	355980	793	2018	36818	39777
Totals						3608124	11488	29675	348741	386128