Advancing hydrogen carrier technologies

Results from testing a 16-reactor system using MCH as a LOHC for reliable screening and optimisation of catalysts for hydrogen release applications

Tiago Vilela, Graham Ormsby, and Lei Zhang
Avantium

Liquid Organic Hydrogen Carriers (LOHCs) have emerged as a promising solution for safe and efficient hydrogen storage and transport, addressing key challenges in the hydrogen economy such as volatility, low energy density, and infrastructure compatibility. Among various LOHC candidates, methylcyclohexane (MCH) stands out due to its favourable thermodynamic properties, high hydrogen content, and compatibility with existing fuel logistics. The reversible dehydrogenation of MCH to toluene enables hydrogen release on demand, making it a strategic component in the development of scalable hydrogen supply chains. The reaction scheme is shown in **Figure 1**.

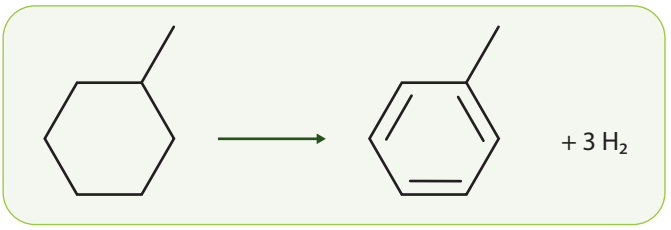

Figure 1 Reaction scheme for methylcyclohexane dehydrogenation

Chemical Factory Acceptance Testing (CFAT)

The CFAT is a critical phase in the commissioning of high-throughput reactor systems. It ensures that the equipment meets predefined specifications for mechanical construction, safety, automation, and analytical performance. CFAT typically includes mechanical integrity checks, software validation, safety interlock testing, and chemical validation using a representative reaction. This article shows the CFAT results of a Flowrence XR system. MCH dehydrogenation was selected as the model reaction due to its well-characterised kinetics and relevance to hydrogen carrier technologies. The unit itself was designed for a broader range of applications though.

Experimental

The Flowrence XR system is designed for catalytic process development under gas, liquid, or trickle flow conditions. It offers high-throughput, isothermal testing with precise control over reaction parameters and combines a high level of precision and automation. More information can be found in the five patents listed in the references. The key features of the unit are shown in **Table 1** and **Figure 2**.

Reactor setup 16 parallel reactors, arranged in 4*4 blocks, each block independent temperature control (50-600°C)
Reactor details 3mm OD, 2mm ID or 2.6mm ID, reactor length 300/560mm, SS316
Gas feed Passive distribution of reactant gas mixture using microfluidic distribution (±0.5% RSD reactor-to-reactor relative standard deviation)
Pressure control All reactors controlled with active pressure control (±0.1 barg) up to 190 barg
Downstream effluent control 4×16 parallel sample vials for liquid product collection (up to 85°C) and trace heating at up to 180°C for online analysis of gas phase
Analytics Online GC, with TCD and FID to quantify C1-C12 and permanent gases with other options of offline analytics available upon request (such as PONA analysis, alpha determination)

Table 1 Summary of standard Flowrence XR setup

The Flowrence XR system provided excellent control over temperature (±0.5°C), pressure (±0.1 bar), and flow rates (±1% of setpoint).

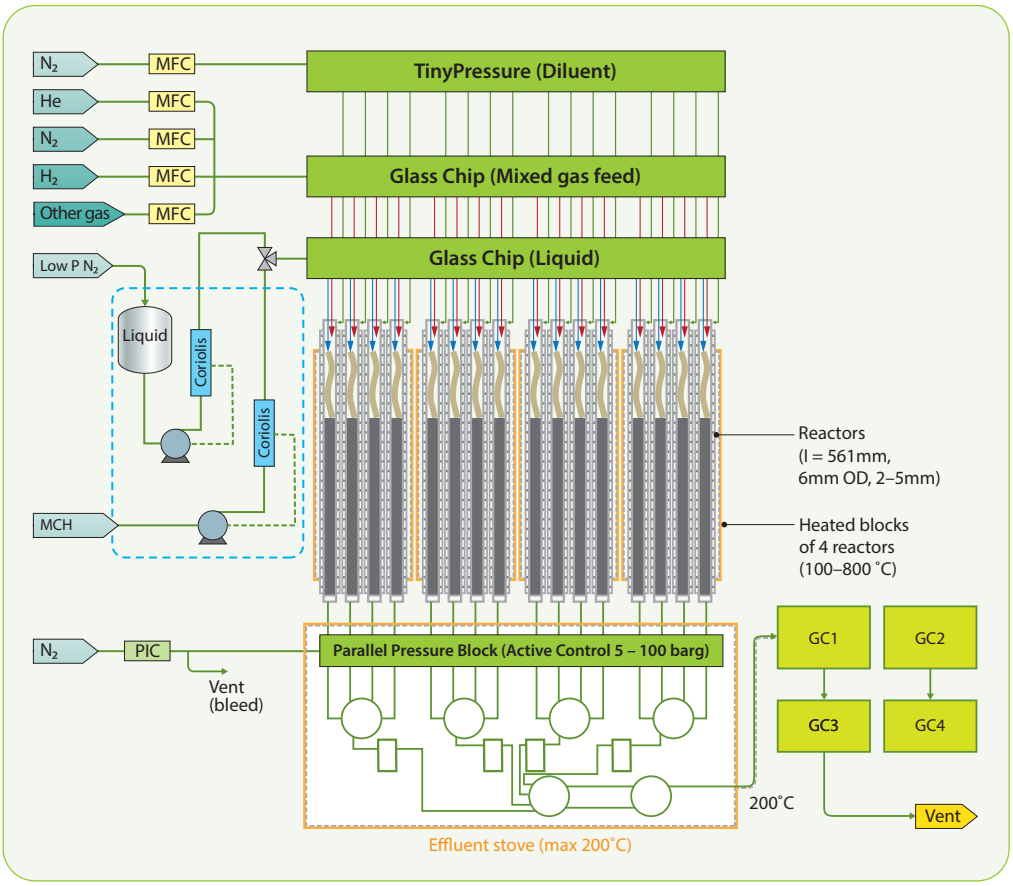

Figure 2 Schematic representation of a Flowrence XR. The gas and liquid supply system and unit specifications can be tailored to specific application

isothermal operation across all reactors.

- Utilising Avantium Microfluidics technology, coupled with a pump featuring Coriolis flow meter feedback, liquid MCH can be distributed evenly across all reactors with exceptional precision and accuracy. This guarantees that the system delivers the highest quality data for comparative testing of dehydrogenation catalysts.
- Furthermore, Avantium's patented parallel pressure regulator, integrated with reactor pressure control and Microfluidics technology, enables a single design to function with high accuracy across the entire pressure range. The reactor pressure control ensures uniform inlet pressure for all 16 reactors, even if pressure drop variations occur due to catalyst coking, for example. Additionally, the reactor pressure drop can be continuously monitored throughout the experiment, providing valuable supplementary process information.
- All process variables – temperature, pressure, flow, and valve positions – are managed through the Flowrence control software, which provides real-time monitoring, data logging, and automated safety interlocks.
- The unit contains 16 parallel fixed-bed reactors within four heating blocks, each heated independently. The design minimises axial and radial temperature gradients, allowing for
- Reproducibility: The system is designed to deliver <2% standard deviation in conversion and selectivity across all reactors.
- Effluent handling: Another key advantage of the Flowrence for this application is the design of the effluent section. After exiting the reactor,

	Run-01	Run-02
Catalyst	0.5%Pt/ alumina, sphere	0.5%Pt/ alumina, sphere
Total liquid feed, gram/h	6.7	6.7
Total gas feed, mL/min		
N_2 (carrier)	82.0	82.0
He (internal standard)	4.0	4.0
H_2	/	0 or 10
Temperature, °C	300-400	400
Pressure, barg	0	0 or 15
WHSV, h^{-1}	6	6
Time on stream, h	50	50

Table 2 Testing conditions

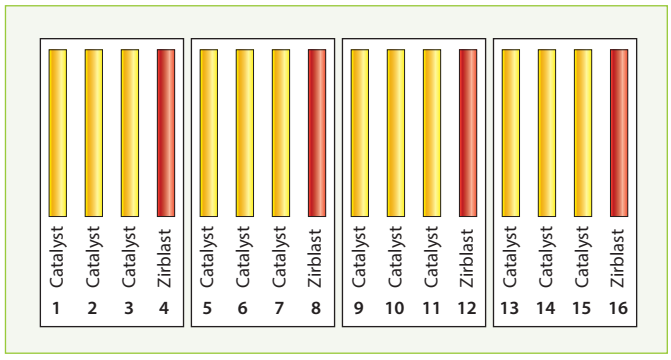

Figure 3 Reactor loading scheme for Run-01

the product stream is immediately diluted with an inert gas (typically nitrogen). The amount of dilution can be chosen freely and is independent of the flow of other gases or pressure.
The combination of dilution and elevated temperatures ensures that even high-boiling species, like hydrocarbons and oxygenates, can be maintained completely in the gas phase and can be directly analysed by online gas chromatography (GC). Using online GC for both gases and liquid products simplifies the analysis by eliminating the need for liquid sampling and allowing all product components to be assessed in one go. This enhances data point density and removes the need for manual liquid sample collection and offline analysis. It also reduces the handling of samples containing benzene and toluene, thereby improving operator safety.

Testing conditions

Two runs were conducted: Run-01 at varied reaction temperature and Run-02 at varied reaction pressure and atmosphere (see **Table 2**).

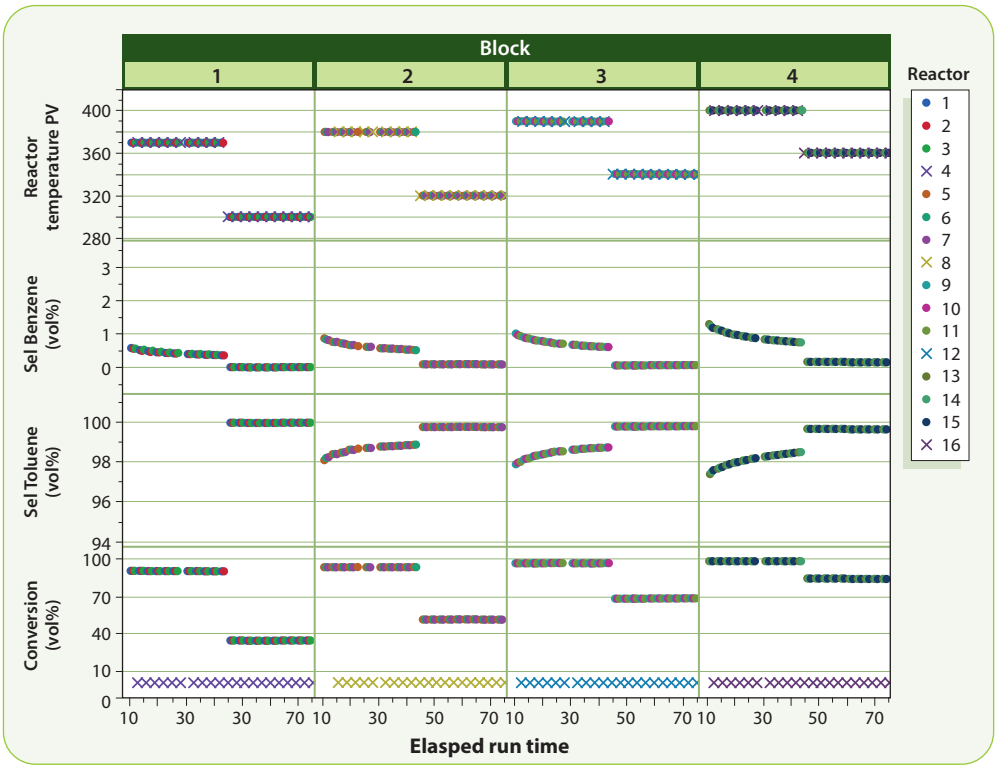

Figure 4 Testing results of Run-01 over time on stream (TOS)

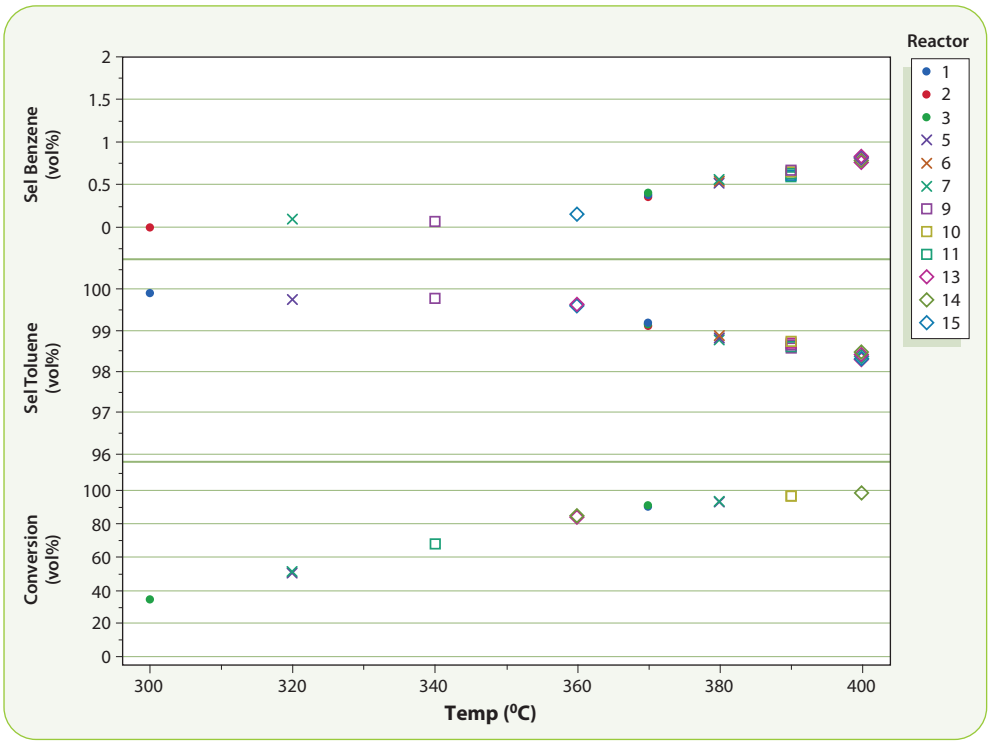

Figure 5 Effect of reaction temperature: testing results of Run-01 with TOS from 25 to 75 hours

Results and discussion

Run-01: Figure 3 shows the reactor loading diagram. The same catalyst was loaded in 12x reactors, with the remaining 4x reactors loaded only with the inert material Zirblast B120 to check the background conversion.

The 4x reactor blocks were heated to different temperatures to investigate the effect of temperature on the catalyst performance.

Figure 4 shows the testing results of Run-01. The run lasted 75 hours. Different reactor temperatures were applied to the different blocks to investigate the dependence

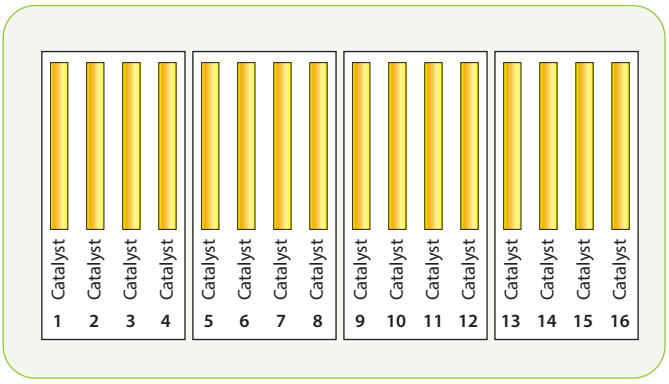

Figure 6 Reactor loading scheme for Run-02

of conversion on temperature. For each block, 2x temperatures were applied.

No conversion was found for the reactors filled only with inert material (R4, R8, R12, and R16), while the other 3x reactors within the same block showed almost identical results, demonstrating strong system stability and data reliability. The conversion for the reactors with the catalyst became stable already after 10 hours on stream, and the selectivity to toluene (major product) and benzene (side product) became stable at approximately 25 hours on stream.

As shown in **Figure 5**, selectivity to toluene remained consistently above 95% across all test conditions, indicating minimal formation of undesired byproducts, with a slight decline at higher temperatures. Benzene formation was below 0.5 mol%, and heavier aromatics were negligible, confirming the catalyst's high specificity for the desired dehydrogenation pathway. Increasing the reaction temperature led to a higher conversion but lower selectivity towards toluene.

For the reactors in the same block, almost identical results were obtained, demonstrating high reactor-to-reactor repeatability.

Run-02: Based on the test results of Run-01, Run-02 was conducted with all the reactors filled with the same catalyst to check reactor-to-reactor variance (see **Figure 6**). The effect of reaction pressure and H_2 gas co-feeding was investigated. The testing results are shown in **Figure 7**.

The run consists of three parts:

- **Part-1**: Up to TOS of 23h: atmospheric pressure.
- **Part-2**: TOS 25 to 47h: 15 barg.
- **Part-3**: TOS 47 to 72h: hydrogen gas was introduced (H_2/MCH 0.40/1 mol/mol, 0.80/1 w/w).

Throughout the run, the mass balance remains within 100 +/-3%. For the first two parts, increasing the pressure from atmospheric pressure to 15 barg resulted in a low conversion and also slightly lower selectivity to toluene. The H_2 yield remained around 6.10 +/-0.10 wt% based on the liquid feed rate, which is close to the theoretical yield at full conversion (6.1 wt%).

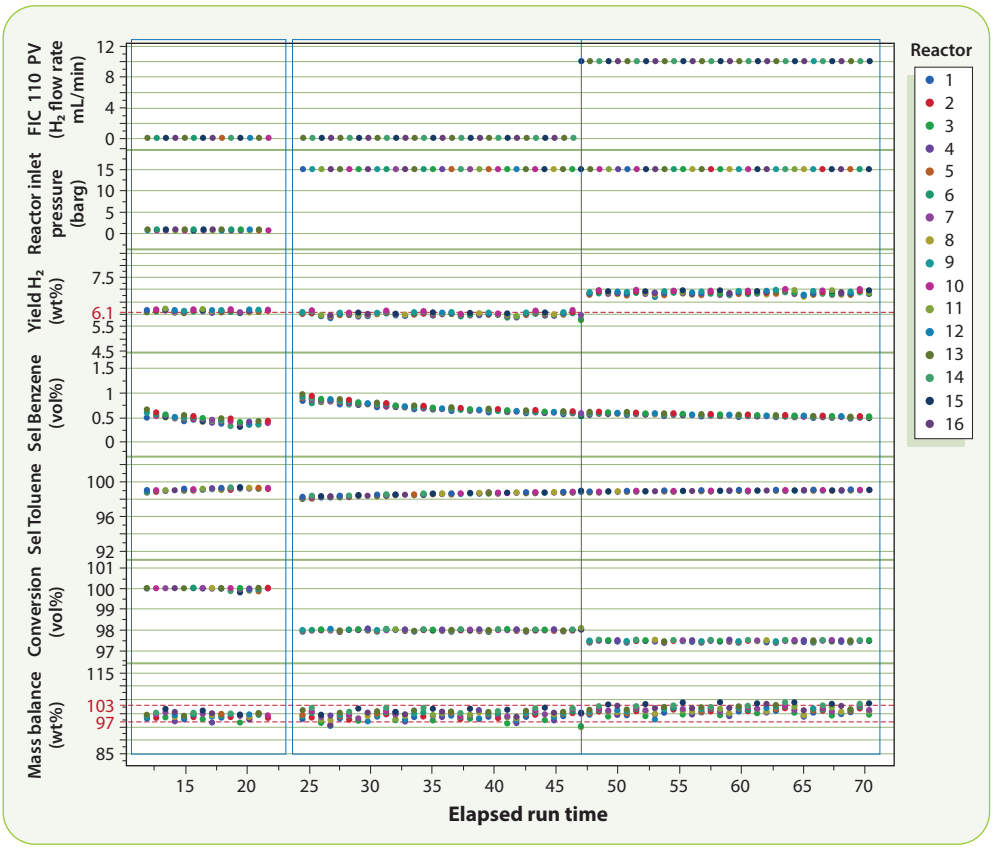

Figure 7 Test results of Run-02

Upon introducing H_2 into the gas stream, the conversion dropped, but the selectivity to toluene remained stable. The increase in the observed H_2 yield (ca 0.75 +/-0.15 wt%) is consistent with the H_2 added to the feed steam (0.80 wt%).

Table 3 shows the statistical analysis of the conversion data for the TOS of 47 to 72 hours, specifically Part-3 of the run. For each reactor, seven or eight data points were used for analysis.

All the reactors showed stable performance,

	Yield toluene, wt%			Mass balance, wt%			Conversion, vol%		
Reactor	n	Mean	Std Dev	n	Mean	Std Dev	n	Mean	Std Dev
1	8	90.23	0.53	8	100.05	0.55	8	97.373	0.005
2	8	91.08	0.48	8	100.96	0.50	8	97.465	0.006
3	8	89.60	0.46	8	99.32	0.48	8	97.481	0.007
4	7	89.64	0.41	7	99.32	0.46	7	97.498	0.006
5	8	90.81	0.64	8	100.56	0.66	8	97.413	0.009
6	8	91.76	0.50	8	101.55	0.53	8	97.495	0.007
7	8	91.28	0.39	8	101.02	0.42	8	97.483	0.008
8	7	90.43	0.44	7	100.08	0.49	7	97.492	0.006
9	8	91.48	0.60	8	101.37	0.64	8	97.453	0.008
10	8	92.16	0.49	8	102.08	0.51	8	97.475	0.008
11	8	91.25	0.72	8	101.12	0.78	8	97.443	0.015
12	7	90.10	1.20	7	99.85	1.32	7	97.462	0.018
13	8	92.09	0.57	8	101.94	0.63	8	97.450	0.014
14	8	92.98	0.54	8	102.83	0.58	8	97.515	0.012
15	8	93.65	0.30	8	103.60	0.31	8	97.463	0.008
16	7	92.33	0.69	7	102.22	0.77	7	97.412	0.014
	Std Dev	**1.16**			**1.23**			**0.037**	

Table 3 Statistics of the testing data with TOS of 47 to 72 hours

with relative standard deviation (RSD) <1% for conversion, toluene yield, and mass balance. The data across all 16 reactors are tightly clustered, confirming excellent uniformity. Reactor-to-reactor variation in conversion and selectivity was consistently below 2%, validating the Flowrence XR's design for uniform flow distribution and thermal control. This is essential for reliable catalyst screening and scale-up predictions. This precision supports the use of the data for kinetic modelling and mechanistic studies.

Conclusion

This test campaign demonstrates the technical feasibility and operational reliability of MCH dehydrogenation as a hydrogen release pathway within LOHC systems. The use of the Flowrence XR platform enabled parallel testing under tightly controlled conditions, generating consistent and reproducible data across 16 reactors.

Conversion trends showed a predictable increase with temperature, reaching full conversion below 400°C while maintaining toluene selectivity above 99%. Reactor-to-reactor variation remained low across all runs, confirming both repeatability and uniformity of operation. These results support the suitability of the Flowrence platform for early-stage catalyst screening and kinetic studies. The campaign confirmed the importance of controlled operation and system design when handling reactive hydrocarbons and trace byproducts like benzene. Successful completion of both mechanical and chemical acceptance testing further confirms the system's readiness for reliable operation in a laboratory or pilot environment.

Overall, these results provide a solid basis for further development of MCH dehydrogenation processes. Avantium can support process optimisation, including extending catalyst stability. Additionally, integration with downstream hydrogen purification and evaluation of process economics to assess scale-up potential will be required.

References

de Ruiter, R. R., Bracht, M., Gruter, G. J. M. 2011 European Patent EP 2263790 A3.

Moonen, R. H. W., 2012 WO2012047095A1.

Moonen, R. H. W., 2016 US Patent 20,160,121,291 A1.

Smit, M., Gruter, G. J. M., 2011 US Patent 7,997,297 B2.

Van den Brink, P. J., Bracht, M., Harji, B. H., 2002 WO2002092219A1.

LINKS

More articles from the following categories:

Emissions Reduction

Hydrogen