THE GLOBAL CHALLENGES IN CHEMICALS AND ENERGY – HOW TO STANDARDIZE AND ACCELERATE ACADEMIC & INDUSTRIAL R+D WITHIN THE OIL AND GAS INDUSTRIES

Amira Abou-Hamdan[1*] and Michael Schneider[2]
[1, 2]Chemspeed Technologies AG, Wölferstrasse 8, CH-4414 Füllinsdorf, Switzerland

*amira.abou-hamdan@chemspeed.com

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INTRODUCTION

Standardization and acceleration of R+D within the oil and gas industries are vital for coping with all the dynamically increasing challenges in their broadest sense. Many industry leaders have recognized the need to act and successfully boosted their R+D outputs

The global challenges in chemicals and energy comprise:
• Increasing constraints in energy supply from fossils and recently nuclear power
• Increasing constraints by regulations (CO2 emission, global warming, …)
• Increasing energy demand e.g. spurred by the dynamic growths in the BRIC countries
• E.g. by 2020 China is projected to consume more than 50 % of the global energy.
• …

Corresponding R+D organizations need to cope with the challenges typically with the same amount of resources. The only way out of this “catch 22” situation is to standardize and accelerate R+D via enabling innovative technology. Real customer case studies on the applications of enabling, automated tools will address:
• enhanced oil recovery (EOR) surfactant and additives screening, e.g. ASP flooding
• catalyst development by incipient wetness/excessive liquid impregnation, precipitation, zeolite synthesis for e.g. hydrogenation, alkylation, isomerization, oxidation, cracking catalysts
• organic and organometallic catalyst synthesis and screening for e.g. polyolefins, rubbers, fine/specialty chemicals
• catalyst testing/optimization in batch, semi-continuous, continuous mode
• formulation and testing of e.g. fuels, oils, and lubricants
• sample preparation for e.g. quality control, waste/environmental analysis
• enhanced development of alternative energy solutions (e.g. solar cell, battery, and fuel cell materials) and renewable chemicals (e.g. from biomass and C1-chemistry)

DESCRIPTION OF THE WORK

In order to elucidate today’s achievements and approaches in High Throughput HT and High Output HO catalysis R+D, various case studies will be presented. These case studies provide examples about benefits, implementation scenarios, and state-of-the-art research platforms, in brief underlining the MUST HAVE in order to:
• Stay competitive
• Increase productivity
• Increase quality
• Spur systematics
• Spur serendipity

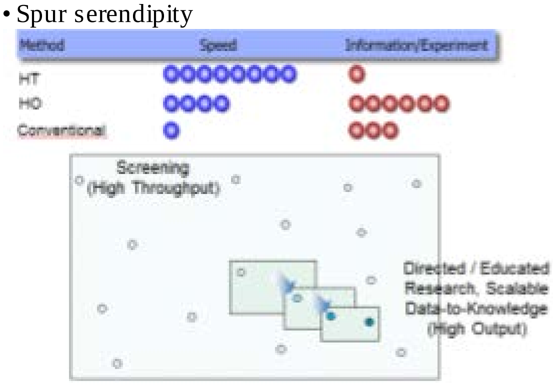


Figure 1: High Throughput and High Output experimentation

The presented case studies cover examples from homogenous and heterogeneous catalytic processes. Improving the conversion rate, the selectivity, yield, or the lifetime of a catalyst immediately affects the profitability of the process. Of similar importance are decreased energetic requirements like applied temperature and pressure. High-Output solutions serve the chemical industry and academia in their vital research not only by improving the number of experiments executed but also at yielding more useful information per experiment.

Heterogeneous catalysis examples include zeolite syntheses, pH controlled precipitation, incipient wetness impregnation and polymerization of propylene oxide among other examples.

The homogeneous catalysis case studies include ZN catalyst library synthesis, slurry copolymerization, FI (post MC) catalyst library synthesis, MAO supportation library synthesis, slurry-phase PE synthesis and scale-up from 100 to 1‘000mL, PP – metallocene quality control and many other examples.

One detailed case studies is presented here:

The incipient wetness impregnation of support materials is one of the most commonly used impregnation techniques in heterogeneous catalysis. Together with modern analytical facilities automated impregnation allows fast screening of different supports, metals, metal ratios, pH, drying conditions etc. In order to automate the incipient wetness impregnation in a High-Output fashion, 2 key requirements must be fulfilled:

1) Accurate automated solid dispensing of metal salts and solid supports, whether they are powders, round beads or whatever kind of extrudates. The first enables the user to automatically screen different metal/metal ratios. The second allows for a tedious, time-consuming and error-prone process to be automated.

2) The impregnation of the utilized solid supports has to yield homogeneous results, which requires a combination of vigorous, yet gentle mixing while controlled liquid dispense.

With our unique overhead gravimetric solid dispensing unit (SDU) and overhead gravimetric dosing units for powders and extrudates (GDU-P), Chemspeed Technologies made solid handling as easy and convenient as liquid dispensing (Figure 2).

Figure 2: overhead gravimetric dosing units for powders and extrudates (GDU-P)

The GDU-P is a combined tool of analytical balance with a dispensing system, which can choose from some 50 different solid containers. The GDU-P picks up one solid container, moves to its target reactor vessel(s), dispenses the desired amount, returns the container and picks up the next one.

For the preparation of the metal solution, the potential workflow is as follows: The Solid Dosing Unit (SDU) picks up metal salt 1 and dispenses i.e. 8 different amounts into 64 different vials. After returning the solid 1 a second metal salt 2 is added into the 64 different vials, also with 8 different amounts yielding 64 different ratios. After an utomatic tool exchange, a liquid handling unit fills water and an acid or base to create 64 mixtures. The mixtures are then heated at 120°C oil bath temperature under reflux to dissolve all the metal salts. After active cooling to 20°C, pH and density measurements of the 64 mixtures characterize the impregnation solutions with which the maximum catalyst load can be determined.

The impregnation procedure starts with the automatic dispense of the support(s). The precise dispensed amount of solids and their pore volume yield automatically the impregnation volume per vessel. The requested volumes of the impregnation solutions are aspirated and dispensed directly into the support while shaking. After the dispenses are completed, the impregnated supports are typically shaken for additional 30-60 min and then dried, for example under nitrogen flow and elevated temperature.

This example yielded heterogeneous catalysts prepared by incipient wetness impregnation with 64 different metal/metal ratios within one run. Similarly, excessive liquid and vacuum impregnations have been performed with confirmed homogeneity and comparability to manual results within 5%.

Note that multiple impregnation processes can be run on the same platform, namely:

- Excessive liquid impregnation

- Incipient wetness impregnation
- Vacuum impregnation
- Precipitation impregnation
- …

ISYNTH CATIMPREG

Figure 3: The CATIMPREG workstation

Automated metal salt solutions preparations, then impregantion:

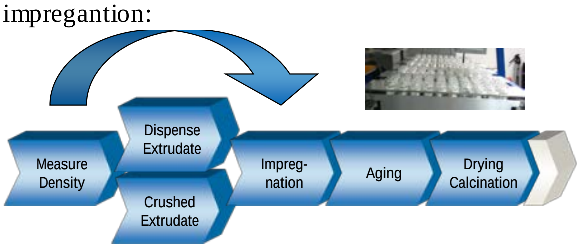
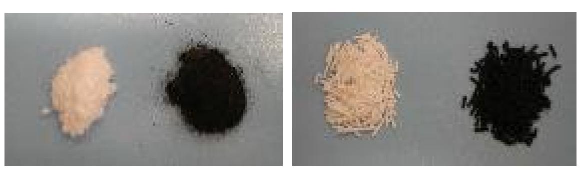


Figure 4: Incipient wetness impregnation workflow



Excellent homogeneity

Figure 5: Examples of untreated and impregnated powder supports and extrudates.

Similar to the described case study, different strategies were successfully carried out, namely:

For 1 solution / 8 impregnations

- Prepare solutions: 30 min
- Heat / cool solutions: 60 min
- Measure pH: 2.5 min
- Measure density: 2.5 min
- Dispense extrudates: 10 min
- Transfer solutions and impregnate: 45 min

Subtotal: 2.5 hours

- Aging: 1 hour
- Drying: 2 hours

Total: 5.5 hours

For 6 solutions / 48 impregnations

- Prepare solutions: 2 hours
- Heat / cool solutions: 1 hour
- Measure pH: 15 min
- Measure density: 15 min
- Dispense extrudates: 1 hour
- Transfer solutions and impregnate: 3.5 hours

Subtotal: 8 hours

- Aging: 1 hour
- Drying: 2 hours

Total: 11 hours

For 24 solutions / 48 impregnations

- Prepare solutions: 3 hours
- Heat / cool solutions: 1 hour
- Measure pH: 0.5 hour
- Measure density: 0.5 hour
- Dispense extrudates: 1 hour
- Transfer solutions and impregnate: 4 hours

Subtotal: 10 hours

- Aging: 1 hour
- Drying: 2 hours

Total: 13 hours

For 48 solutions / 48 impregnations

- Prepare solutions: 5 hours
- Heat / cool solutions: 1 hour
- Measure pH: 1 hour
- Measure density: 1 hour
- Dispense extrudates: 1 hour
- Transfer solutions and impregnate: 5 hours

Subtotal: 14 hours

- Aging: 1 hour
- Drying: 2 hours

Total: 17 hours

The various strategies carried out by various laboratories; confirm the flexibility and modularity of Chemspeed's automated workstations. Beside the increase in productivity, the quality and reproducibility of the experiments are optimal and a confirmed significant decrease of the cost per experiment can be reached, associated with the selected configuration of the workstation.

CONCLUSIONS

Scientists have proven: The preparation and testing of Homogeneous and Heterogeneous Catalysts can be executed utilizing Chemspeed's Array, ISYNTH, process reactors and formulation reactors technologies. Through parallelization of the reactors and automation of the chemical supplies, Chemspeed's highly modular and flexible automated workstations have enabled scientists worldwide performing High-Output Experimentation. High-Output Experimentation allowed researchers to explore the scientific space in a convenient, reproducible, and faster way without compromise leading to commercial products and applications in industrial processes.

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