



WCCE11 - 11th WORLD CONGRESS OF CHEMICAL ENGINEERING

IACCHE - XXX INTERAMERICAN CONGRESS OF CHEMICAL ENGINEERING
CAIQ2023 - XI ARGENTINIAN CONGRESS OF CHEMICAL ENGINEERING
CIBIQ2023 - II IBEROAMERICAN CONGRESS OF CHEMICAL ENGINEERING

Buenos Aires - Argentina - June 4-8, 2023

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CO-PrOx in a milireactor 3d-printed by FDM

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INTRODUCTION & OBJECTIVES: An attractive option towards process intensification is the introduction of milireactors, with increased heat and mass transfer coefficients and enhanced control of the flux pattern [1]. The manufacture of these units requires a detailed and meticulous procedure. The use of 3D printing technologies provides the desired manufacture precision, with a high degree of flexibility in the design and at reduced costs, specially when FDM-type printers are employed. The objective of this work is to study the design, manufacture and operation of a milireactor 3D-printed by FDM and profited towards the oxidation of CO in H₂-rich environments (CO-PrOx).

MATERIALS AND METHODS: Different prototypes of the milireactor were manufactured by means of a Creatbot F430 FDM 3D printer, using 1.75mm filaments including PLA, PETG, flex (TPU-based) and nylon (PA-12). The designs have been achieved using OnShape as a free CAD software whereas the Creatware slicing software was used to generate the correspondent GCode. A Au-Fe₂O₃/Al₂O₃ catalyst (pellets) is used to conduct the CO-PrOx reaction. Gases are fed from pressurized bottles via mass flow controllers. Analytics comprises a GC equipped with TCD, molsieve/porapak-Q columns and valve injector operating at 2psi manometric pressure. This last issue implies the milireactor operation at over-atmospheric pressures requiring enhanced sealing properties of the manufactured unit.

The milireactor design comprises two halves, bottom and top, the former containing the flow channels and the inlet/outlet conduits. Ca. 2.19g catalyst was loaded in the reaction channel. Two separate inlet channels to independently feed a CO/H₂/CO₂ mixture and O₂ were incorporated in the milireactor following the guidelines reported elsewhere [2]. The reactor output comprises a single channel. Cavities already prepared to couple 4mm racors to inlet/outlet channels are provided as well. A rubber self-made o-ring plus high-temperature silicon proved the better alternative to achieve the required seal between reactor halves. Nylon was finally selected as material for the reactor manufacture based on its thermal stability at the CO-PrOx reaction conditions under test (T ranging from 60°C to 120°C). The complete milireactor was disposed inside a GC oven to proceed with the experiences.

RESULTS AND MAIN CONCLUSIONS: Both PETG and nylon proved adequate in terms of printing definition to achieve the degree of detail required by the inlet channels. As mentioned, finally nylon was selected for the milireactor manufacture based on a superior thermal stability. On the other hand, flex filament showed poor results as sealing material between reactor halves. The milireactor prototype succeeded conducting sustained operation under COPROX conditions, with a feed of 215 Nml/(min.g_{cat}) ($\lambda=3$), at 3psi manometric pressure and temperatures up to 120°C.

References

1. Dautzenberg, F., Mukherjee, M. (2001). Process intensification using multifunctional reactors, *Chemical Engineering Science*, 56, 251–267.
2. Ehrfeld, W., Hessel, V., Lowe, H. (2000). *Microreactors: New Technology for Modern Chemistry*, Wiley.