

A hydrodynamic study of oligomerization catalyst testing in a gas-liquid microreactor

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Oligomerization applications still raise numerous issues regarding process optimization as well as new catalyst development. This requires an important testing capacity, which cannot be reached with standard tools and methods (typically stirred tank reactors) in a short time. Using microreactors, which generally allow fast analysis times, is expected to minimize liquid hold-up and the duration of experiments.

The reaction of interest is the two-phase dimerization of ethylene using a liquid catalyst that is converted into butenes (70-80%), hexenes (20-30%) and heavier products up to C12 (5-10%). Performing this reaction in a microreactor can be challenging as the gas-liquid segmented flow will evolve as a function of the residence time in the reactor, starting from high gas-liquid ratios and long bubbles at the entrance, to a complete disappearance of the gas phase at the outlet when the reactant is totally converted and dissolved in the liquid phase.

In order to understand this evolving gas-liquid flow, a hydrodynamic study was conducted in a tubular microreactor (ID: 500 μm , length: 1 m) under the operating conditions of the oligomerization reaction (typically 45°C and 20 bar). A bubble train was generated by introducing ethylene (gas phase) and a solvent (liquid phase) in a T-junction. The injection of the liquid phase catalyst was mimicked by introducing the solvent in a second T-junction, downstream of the generation of the bubble train. The flow generation in the T-junction and the stability of the gas-liquid flow after the second liquid injection were firstly studied. Flow patterns and typical characteristics of the flow (bubble and slug length, bubble velocity, film thickness) were monitored using a camera and image processing techniques. Both the gas and liquid flow rates were varied in the range $Q_G = 0-500 \text{ NmL/h}$, $Q_L = 0.1-2 \text{ mL/h}$ (corresponding to $U_G =$

XXX and U_L xxx) in order to represent the evolution of gas-liquid ratio during the reaction and to cover the entire conversion range in our system.

This detailed characterization of the flow allows identification of the operating range that is favorable, in terms of hydrodynamics, for the reaction and of the limits of operability of the system.

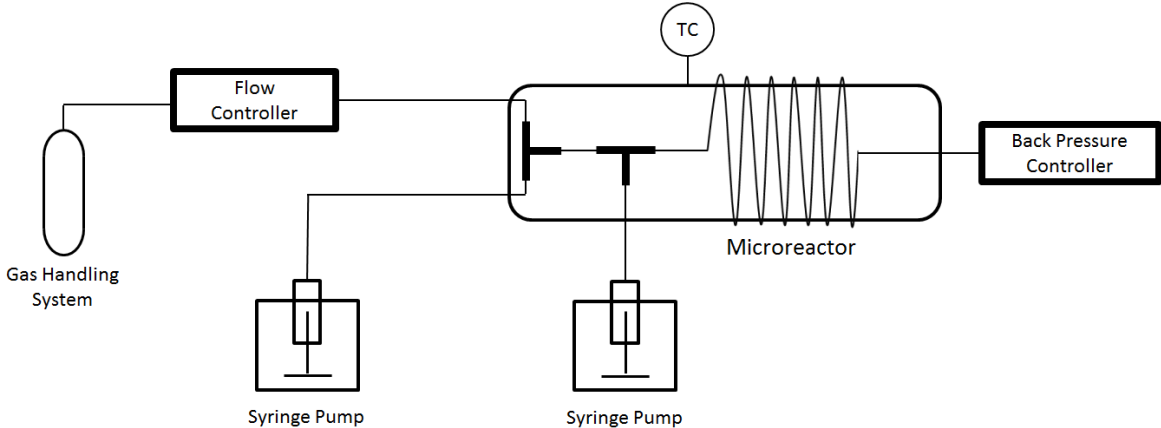


Figure 1 - Experimental set-up