Laboratory Catalytic Reactors

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Novel or improved catalysts are often the essential feature of a new process development or process improvement. Laboratory testing to assess commercial suitability is thus a key process development skill. Scale up and design of the commercialreactor is another key consideration.

Laboratory reactors often look nothing like their commercial counterparts. For evaluation of catalyst kinetics, selectivity, and life, the lab reactor should operate in a well-defined operating regime, free of anomalous transport resistances that would mask scale-up performance.[1-3] For heterogeneous catalysts, intrinsic catalyst kinetics and life should be first assessed free of interphase (fluid-catalyst) and interparticle (catalyst-catalyst) transport resistances, followed by alternate experiments or numerical models to address potential nonideal, transported-limited conditions for the commercial-scale reactor. Tests at varying catalyst particle size including the proposed commercial catalyst should be conducted, to assess effectiveness factors or intraparticle transport resistances. Particulate catalysts for liquid phase systems are likely to operate in a regime where intraparticle resistances are significant, which can readily impact catalyst life. Low flowrates in laboratory experiments to obtain high conversions in small units can result in transport anomalies, relative to commercial reactors scaled to the same space velocity or contact time.

Trickle-bed reactors present unique challenges. Low liquid mass flowrates (less than 5 $kg/m^2/h$) associated with scaling via contact time or WHSV readily lead to incomplete wetting[4] of the catalyst surface for smaller reactors, which can result in either lower or higher conversions depending upon the volatility of the limiting reagent. Larger-scale reactors may be subject to nonisothermal conditions, with a tube / particle diameter ratio of < 4 recommended for highly exothermic reactions.[3] Continuous-flow stirred basket reactors should be considered as an alternative, or use of small particle diluent to improve wetting and reduce channeling for a laboratory trickle bed reactor.[5]

Homogeneous catalysts are less prone to masking of performance by lab reactor anomalies. However, impact of recycle impurities on catalyst activity is often important, which may require an integrated pilot plant with closed recycle loops for accurate assessment.

Vapor phase reactions are subject to kinetic masking due to channeling and axial dispersion. Poor interphase (vapor-catalyst) heat transfer at low laboratory flowrates may result in high intraparticle temperatures, and anomalous kinetics.

The stirred-reactor with gas-induction impeller is ideal for laboratory study of many liquid or gas-liquid reaction systems, with use of annular catalyst basket for particulate catalyst, or dispersed slurry catalyst with dip tube to effect sampling. Mixing smoothes concentration and temperature gradients, which can be confirmed via impact of shaft rotation speed on measured kinetics. Reliable kinetics can therefore be derived. Operation in batch mode allows rapid evaluation of kinetic parameters. This reactor type can readily be used to acquire kinetic and intraparticle transport information for design of a commercial trickle bed, slurry, fluidized bed, or bubble column reactor. There is no simple analog for vapor phase reactions, as recycle reactors with gas recirculation and low holdups are difficult to construct at small scale. Fortunately, once-through vapor phase reactions with solid catalyst are more readily implemented in continuous mode at small scale than their gas-liquid-solid counterparts, with flowrate and catalyst bed length variation to adjust contact time. Pulse microreactors entailing a catalyst bed followed by analytical GC column may be readily implemented for catalyst screening of vapor phase or gas-liquid reactions.

References

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