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A versatile and compact experimental apparatus for the on-line spectroscopic study of liquid-phase heterogeneous catalytic systems

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Abstract

A versatile and compact experimental system is described for the study of fine-chemistry liquid-phase heterogeneous catalysis. The general experimental system consists of a stirred tank (25â€“100 mL), pump, tubular reactor, spectrometer(s), and injection block for liquid-phase perturbations, all in a closed-recycling configuration. The basic designs of the in-house-constructed components are provided. The system was characterized with respect to gasâ€“liquid mass transfer, mixing, liquidâ€–solid mass transfer, and intraparticle diffusion. The utility of the system is demonstrated with a heterogeneous catalytic reactionâ€”the racemic hydrogenation of acetophenone over Pt/Al_2O_3â€”using

on-line Fourier transform infrared (FTIR) analytics. Liquidâ€¨solid mass transfer and intraparticle diffusion were studied by varying the liquid hourly space velocity (LHSV) as well as the catalyst particle sizes used. The reaction rates based on the instantaneous reagent concentrations were precisely evaluated using on-line FTIR measurements. The rather novel inclusion of an injection/sampling block was particularly useful for performing multiple perturbations of reagents, a situation neither normally available nor convenient for the experimentalist, thus facilitating outstanding spectral deconvolution using band-target entropy minimization. The small total liquid-phase volume involved (â‰ˆ15 ml) would facilitate the frugal use of chiral reagents/auxiliaries as well as isotopically labeled components.

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Keywords

Recycle reactor; Liquid-phase catalysis; On-line FTIR spectroscopy; Multiple perturbations

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