Suspensions, slurry transfers and filtration on the Chemspeed MiniPlant

Jake Grace. Chemspeed Technologies, Caxton House, Northampton Science Park, Kings Park Road, Moulton Park, Northampton, NN3 6LG

Introduction

Many reactions used throughout the chemical industries are heterogeneous in nature. A great number of these involve the dispersal of solid particles in a liquid phase; for example a hydrogenation catalyst such as Pd/C in an organic solvent. Or where it is necessary to contact water-insoluble reactants with inorganic reagents; as in the halide exchange reaction between a chloroaromatic compound and KF, which has very low solubility in the solvents of choice for these reactions – typically DMF or DMA.

It is of paramount importance that an automated reaction system is able to cope with such solid-liquid systems; whether that manifests itself in the ability to remove a solid catalyst by filtering the liquid phase into a second reactor, or to transfer a slurry directly from one vessel to another – for example to perform a quenching step, or even to suspend a slurry in one vessel and carry out a controlled reagent addition (of that slurry) into a second reactor.

This application note describes how all three aforementioned procedures are possible with Chempseed's MiniPlant reactors (Figure 1).

Figure 1. Chemspeed's MiniPlant and reactors.

Experimental

The vessel-to-vessel slurry transfer and filtration were carried out in a very similar fashion. Two MiniPlant reactors were connected together by either a short length of PTFE tubing, or a combination of PTFE tubing and a filter-tip (Figure 2); and, assuming transfer from left to right, either a vacuum was applied to the right hand reactor or a positive pressure to the left hand side (or a combination of the two) to afford efficient transfer of the desired medium between the two reactors. Fine tuning of the pressure / vacuum and filter-tip were sometimes necessary to optimise the transfer.

For the suspension and transfer of a reagent slurry, a more quantitative treatment was required. The transfer of a slurry of Na_2CO_3 in MeOH into a solution of HCl, was chosen. The slurry was suspended in the left hand reactor of the MiniPlant and transferred into the right hand reactor using the MiniPlant's syringe pump / valve system. The progress of the transfer was monitored by measuring the temperature increase in the right hand reactor.

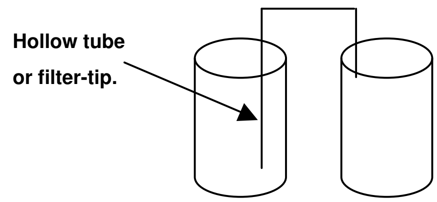

Figure 2. A schematic showing the connection of MiniPlant reactors for filtration or vessel to vessel transfer.

A control experiment was performed by adding Na_2CO_3 (250 mg) and MeOH (5 mL) (equivalent to a 50 g / L slurry) to a stirred solution of HCl (1.0 M, 20 mL) in a MiniPlant reactor and the temperature increase was recorded.

A slurry was made by adding Na_2CO_3 (0.5 g) to a stirred reactor containing MeOH (10 mL). A portion of the slurry (3 mL) was then transferred to a second stirred reactor (containing HCl (1.0 M, 20 mL)), through a loop in three portions using a 1 mL syringe.

Results

Both the vessel to vessel slurry transfer and filtration worked extremely well. Typically less than 1 % of solution remained after the initial cycle, which could easily be transferred by adding a step to wash the left hand reactor through with an additional quantity of solvent.

The control experiment gave a temperature rise of 5.0± 0.1 °C, which is in good agreement with the calculated value of 5.6 °C. The temperature profile of the reactor during the slurry addition is shown in Figure 3, the addition of 3 mL of slurry accounted for a 3.2 °C rise in temperature, in good agreement with both the control and calculated values.

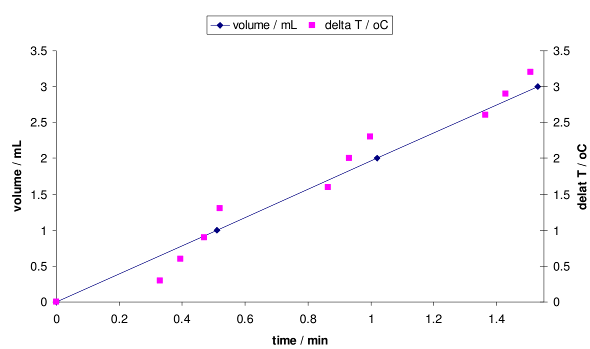

Figure 3. Temperature profile for the addition of 3 mL of 50 g / L Na_2CO_3 in MeOH to 20 mL HCl (1.0 M) over 1.5 min.

Conclusions

Chemspeed's MiniPlant technology affords two convenient methods for transferring slurries between reactors, and the capability to carry out multistage protocols by allowing filtration from vessel to vessel.