Batch-to-continuous case study 1: Quaternary Ammonium Monomer

The Process

Synthesis

$R_2NH + R'\text{-}Cl \longrightarrow R_2NR'.HCl$

$R_2NR' + R'\text{-}Cl \longrightarrow R_2NR'_2.Cl$ (product)

Neutralization

$HCl + NaOH \longrightarrow NaCl + H_2O$

$R_2NH + HCl \longrightarrow R_2NH.HCl$

Side-reaction

$R'\text{-}Cl + NaOH \longrightarrow R'\text{-}OH + NaCl$

Customer Requirements

- Company B were interested in developing a continuous reactor system for production of an important quaternary ammonium monomer

Project Aims

- To determine suitability for different reactor modes (continuous and batch).
- To obtain calorimetry data + other information for use during batch scale-up

Approach

- A combination of reaction calorimetry and process simulation was used
- A chemical model for the system was constructed from the process chemistry network
- Reaction kinetics were determined and added to the model in order to construct detailed process models
- Process models were then used to simulate large scale behaviour in plug-flow (PFR), continuous stirred-tank (CSTR), and batch reactors.

Results

- Very fast initial reaction phase (Qr rose rapidly on addition of chloride, fell rapidly after first addition)
- Slower second stage (Qr does not fall between successive additions indicating chloride accumulation)
- Long (>10h) stir-out period was required to achieve complete conversion
- Stir-out period was found to be affected by pH and agitation
  - Reaction stalled if pH was kept constant at 9.5
  - Reaction stalled if stirring rate dropped below 450 r.p.m.
- Agitation was an important variable due to the phenomenon of two discrete mixing zones, attributed to NaCl precipitation and accumulation.
- Kinetic model constructed from Qr data predicted temperature rise of 25 to 172°C in < 1 second!

(continued overleaf)

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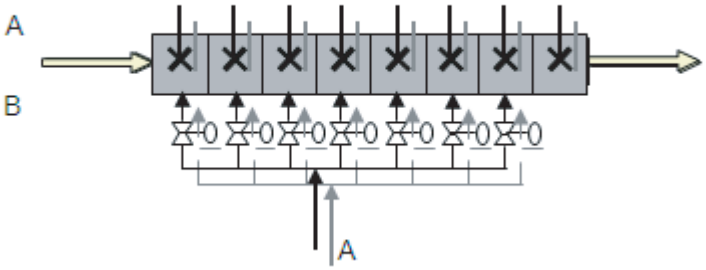
Process Modelling Conclusions

- A PFR would require addition of reagents along length of reactor to regulate exotherm and control pH.
- This was deemed too complex to develop and difficult to operate at production scale for this process



- The long stir-out period would be unsuitable for CSTR operation. A 'train' of reactors would be needed to provide the residence time required to achieve high conversion (each with its own temperature and pH control).
- This was also felt to be overly complex to develop

Outputs

- Process models suggested the process was unsuitable for continuous operation modes
- Using date already obtained, a batch model was constructed to simulate scale-up in 2,000 gallon Pfaudler vessel
- Chloride accumulation predicted in the batch model matched that observed in calorimeter runs, thus validating the results from simulation experiments
- Thermal safety data (heats of reaction) for the process was also provided