DECHEMA FORSCHUNGSINSTITUT

Chemical Technology

Determination of mixing efficiency of micro structured mixers by isothermal heat balance calorimetry

A. Pashkova, L. Greiner
e-mail: pashkova@dechema.de
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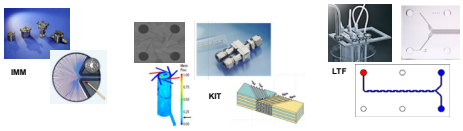
Motivation

Mixing can be vastly enhanced by use of micro structured elements. For liquid liquid applications a number of manufacturers of micro mixing devices exist, each offering hardware based on different mixing principles.



Important
assessment of mixing efficiency and comparison of devices

Drawbacks of existing methods for micro mixer characterisation:

Reactive methods
- experimentally complex
- high uncertainties about the reaction
- extrapolated reference point
- poor comparability

Optical /spectrometric methods
- need for optical transparency

Computational methods
- parameters for calculation of characteristic numbers (Re, Xs, Pe) often not available

Assumtion
Ideal mixing leads to perfect selectivity

⇨ Problem
ideal case is experimentally not accessible

Project idea

Use the excess enthalpy h_E (enthalpy of mixing) as a reference point for determination of mixing efficiency of micro mixing devices

h_E
- measure for ideal mixing of two fluids on molecular scale
- summarised in databases
- accessible by isothermal calorimetry

$h_{E,eff}$
- obtained from continuous heat balance calorimetry for the case when mixing takes place inside a micro mixing device

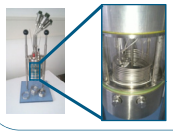
Dimensionless goodness of mixing coefficient $G = h_{E,eff} / h_E$

Advantages of the calorimetric method
- very well defined reference point
- high reproducibility and comparability
- possible wide variation of fluid viscosity
- no need for optical transparent devices
- no need for knowledge of mixer channel geometry

Experimental set-up

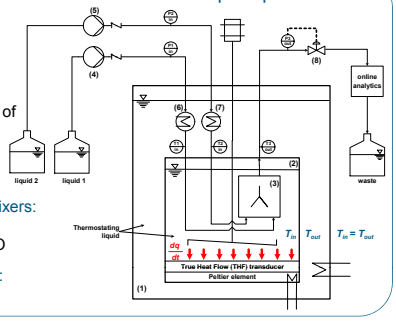
ChemiSens CPA202 reaction calorimeter - "true heat flow" principle

Benefits:
- factory calibrated
- stable baseline
- tolerant to changes in:
  - reactor fill volume
  - heat transfer properties of insides
  - reactant viscosity
  - stirring intensity

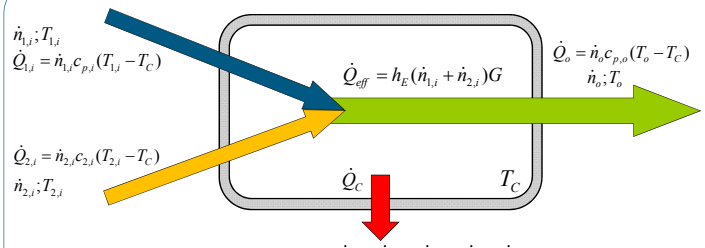


Integrated micro mixers:
- SIMMV2 / IMM
- T – piece /VALCO

Model substances:
ethanol / acetone



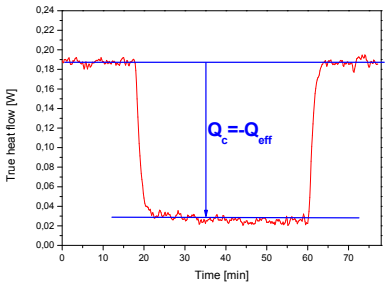
Heat balance of the continuous system



Isolated system $0 = \dot{Q}_C + \dot{Q}_{1,i} + \dot{Q}_{2,i} + \dot{Q}_o + \dot{Q}_{eff}$

Experimental prerequisite / isothermal conditions $T_C = T_i = T_o \longrightarrow Q_C = -\dot{Q}_{eff} = -h_E(\dot{n}_1 + \dot{n}_2)G$

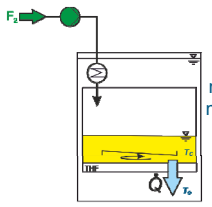
$$G = -\frac{\dot{Q}_C}{h_E(\dot{n}_1 + \dot{n}_2)}$$



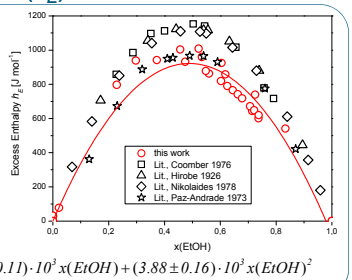
$\dot{n}$ – mole flows [mol min-1]
$\dot{Q}$ – heat flows [W]
T – temperature [K]
c_p – molar heat capacity [J K-1 mol-1]

eff – effective
i – inlet
c – calorimeter
o – outlet

Determination of the reference point (h_E)



Calorimetric measurements of mixing enthalpy in batch mode

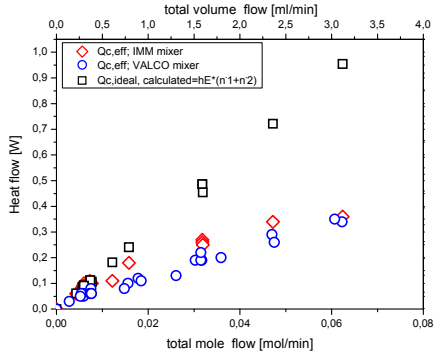


Second order polynomial estimated for reference values

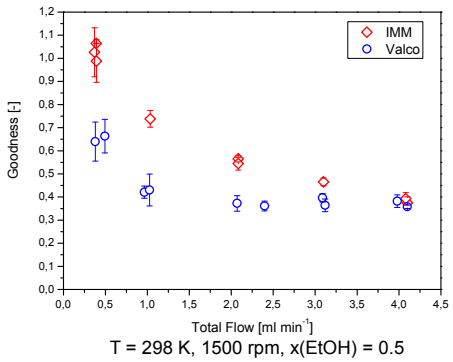
$$h_E = (3.78 \pm 0.11) \cdot 10^3 x(EtOH) + (3.88 \pm 0.16) \cdot 10^3 x(EtOH)^2$$

Mixer characterisation

Effect of volumetric flow rate / residence time

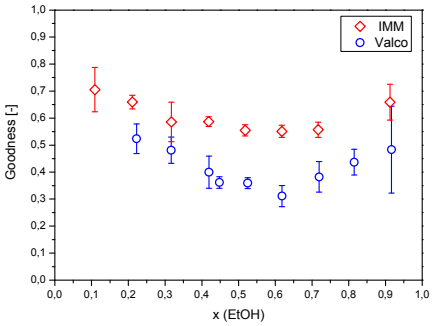


T = 298 K, 1500 rpm, x(EtOH) = 0.5



T = 298 K, 1500 rpm, x(EtOH) = 0.5

Effect of stoichiometry



T = 298 K, 1500 rpm, Ftotal = 2 ml min^{-1}

Summary

- the derived G-value is suitable for experimental determination of mixing efficiency
- G lifts a number of constraints imposed by other techniques
- preliminary results: A. Pashkova,L. Greiner *Chem. Eng. Sci.* 76 (2012) 45-48

Outlook

- improvement of sensitivity and signal to noise ratio
- improvement of the experimental set-up: pumps, T and P measurement
- comparison with reactive methods for mixer characterisation