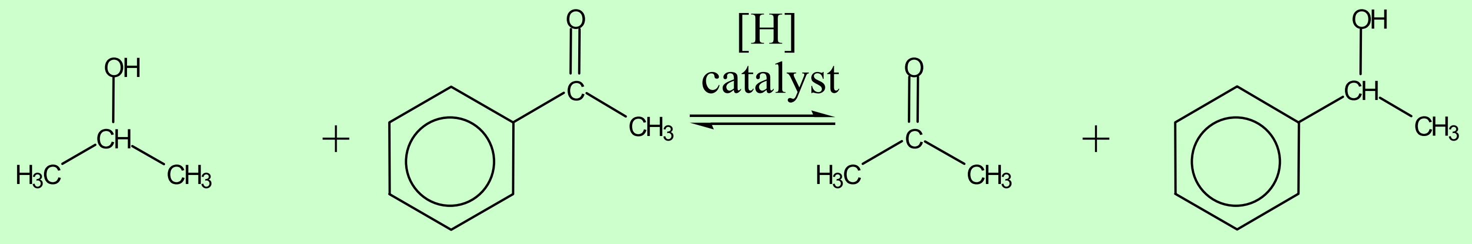
Intensified Acetone Stripping and Asymmetric Transfer Hydrogenation in a Mesh Contactor

A. Gavriilidis
Dept of Chemical Engineering
University College London

Outline

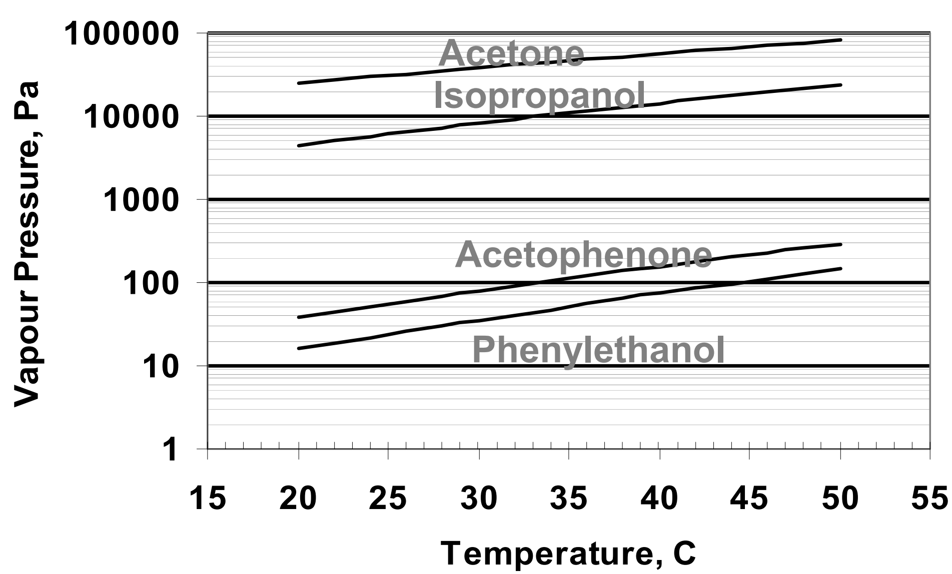
- **Asymmetric transfer hydrogenation**
- **Mesh reactor**
- **Acetone stripping in mesh reactor**
- **Asymmetric transfer hydrogenation in mesh reactor**
- **Mathematical model for stripping and reaction**
- **Conclusions**

Asymmetric Transfer Hydrogenation



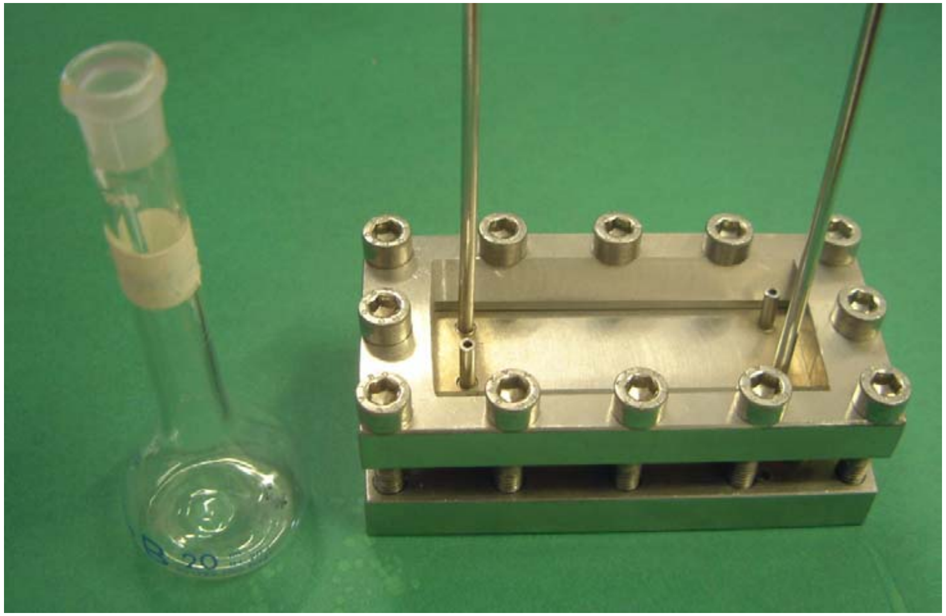
Isopropanol **Acetophenone** **Acetone** **1-Phenylethanol (R, S)**

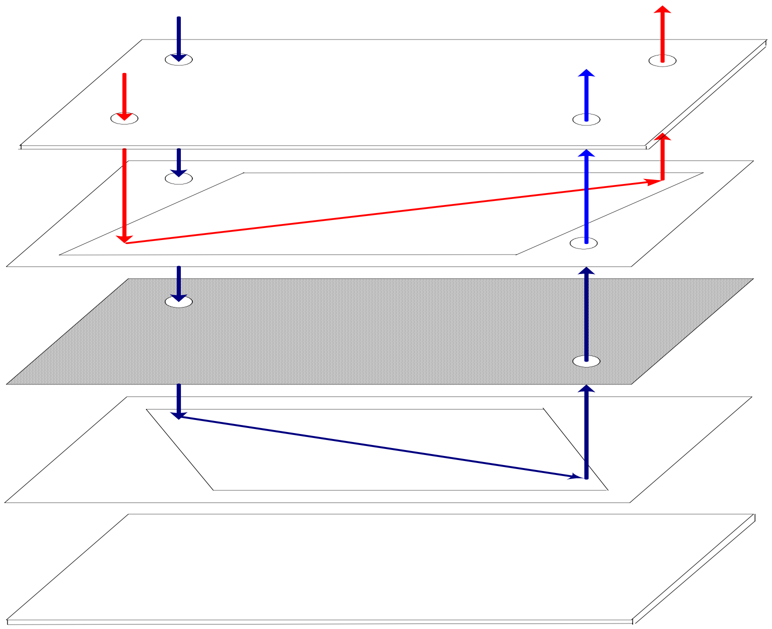
- **Reversible reaction**
- **Backwards reaction limits the reaction conversion and enantioselectivity**
- **Acetone is the most volatile compound**

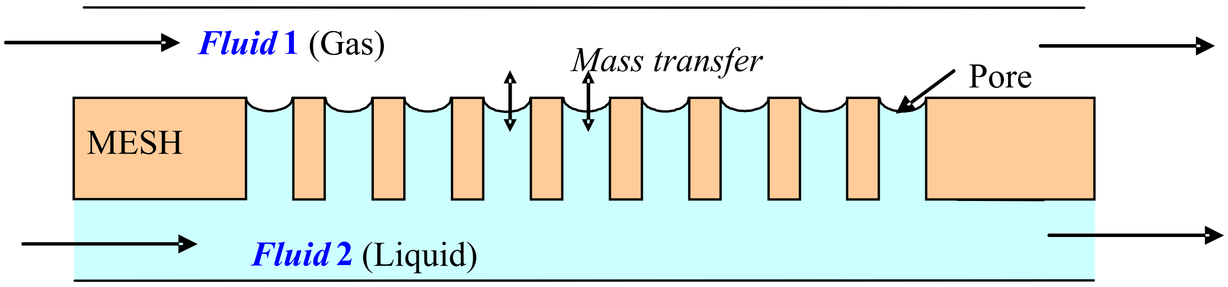


Acetone removal

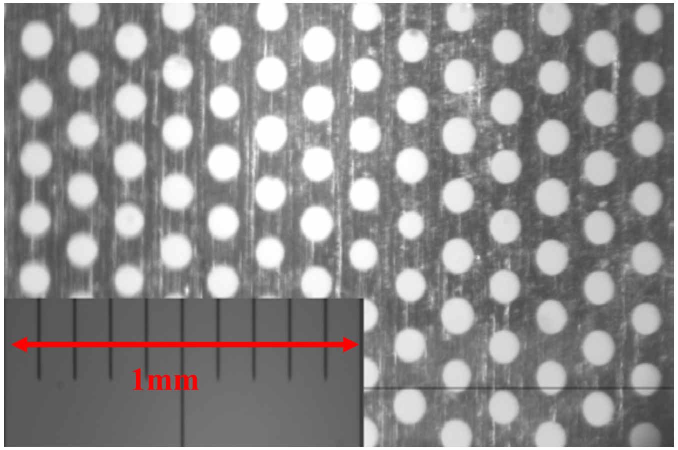
Mesh Reactor







Mesh



Mesh	Average pore size (μm)[a]	Maximum pore size (μm)[b]	Thickness (μm)[a]	Open area	Material
Internetmesh	76	100	50	23%[a]	Stainless steel 304

(a: From manufacturer, b: Determined from optical pictures)

Breakthrough Studies

$$\Delta P = \frac{2\gamma \cos\theta}{r}$$

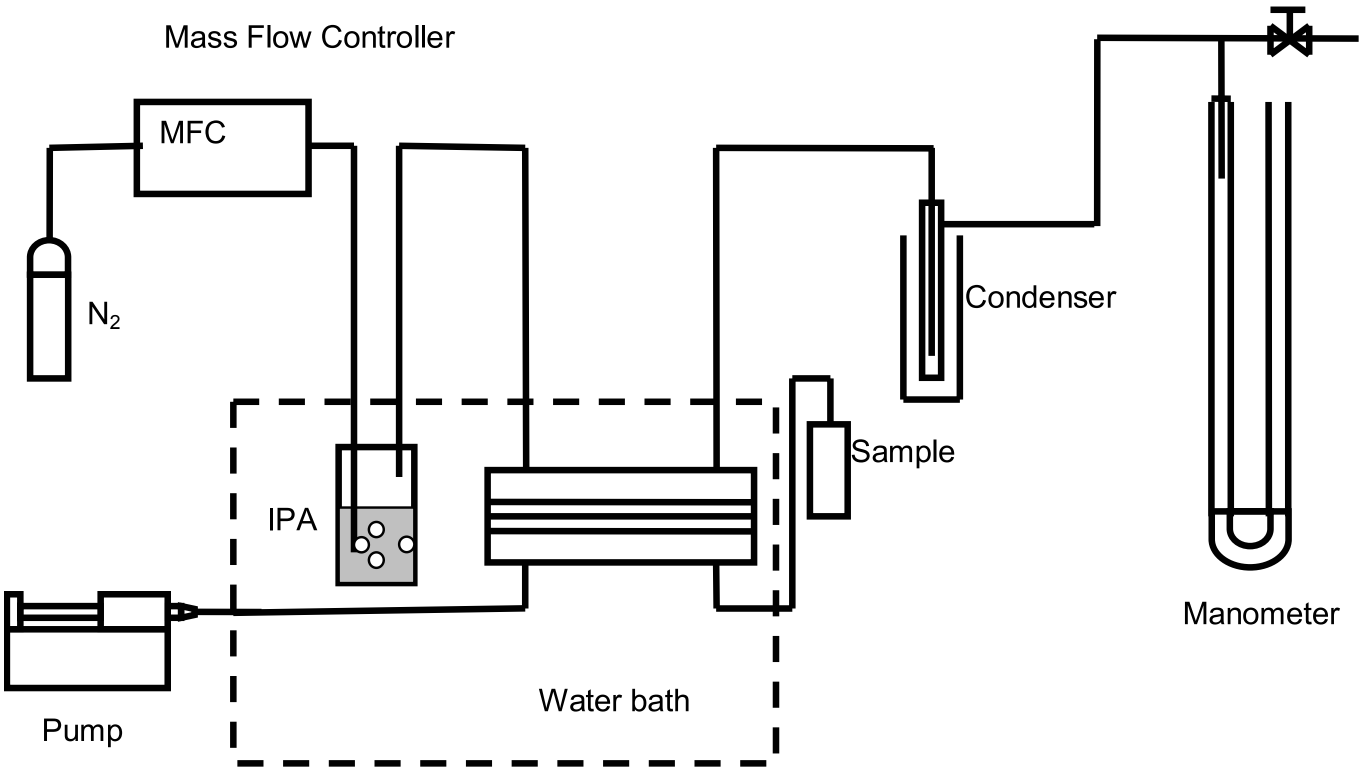
$$\Delta P_{B,IPA} = P_L - P_G = -\frac{2\gamma\cos(0+90)}{r} = 0$$

$$\Delta P_{B,N_2} = P_G - P_L = \frac{2\gamma\cos(0)}{r} = \frac{2\gamma}{r}$$

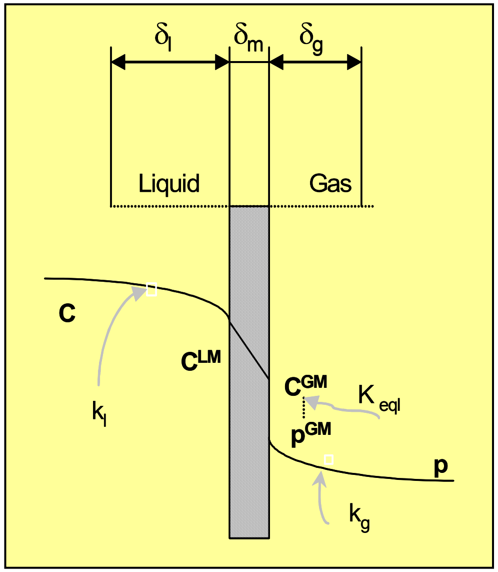
Mesh	Breakthrough pressure (G → L) P_G-P_L (mm H_2O)		Breakthrough pressure (L → G) P_L-P_G (mm H_2O)	
	Experiment	Model*	Experiment	Model*
Internetmesh	70~80	84	-5	0

* using maximum pore size

Set-up for Acetone Stripping



Analytical Modeling



$$\frac{C_{out}}{C_{in}} = \frac{1}{1+\Omega}\left\{1+\Omega\cdot\exp\left[-\beta\cdot\left(1+\frac{1}{\Omega}\right)\cdot H\right]\right\}$$

$$\Omega = \frac{F_g}{F_l}\cdot\frac{P}{R_g T}\cdot\frac{1}{K_{eq}} \qquad \beta = \frac{K_T\cdot\varepsilon\cdot\tau_l}{\delta_l\cdot L}$$

Ω is related to

- ratio of gas flowrate over liquid flowrate
- volatile component solubility

β is proportional to

- overall mass transfer
- liquid residence time
- open area of mesh

Correlation for k_l and k_g

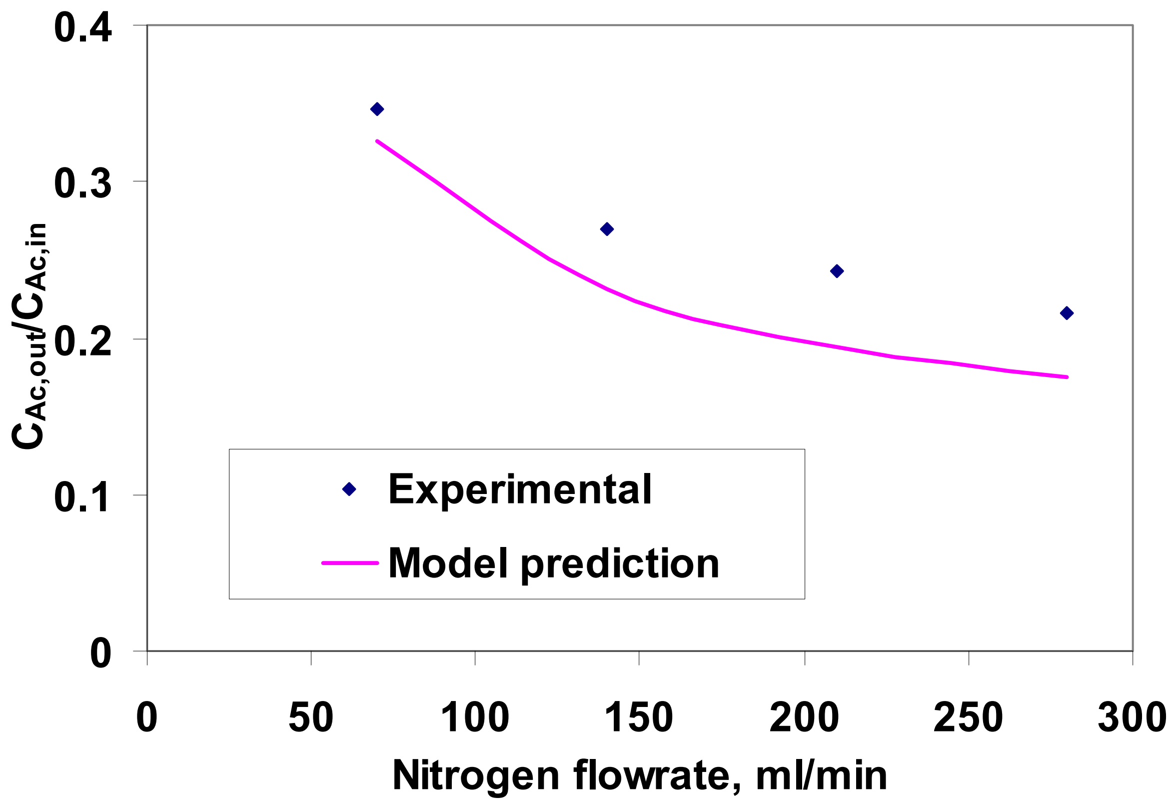
$$Gz = \frac{Re\cdot Sc\cdot 2\delta}{L} < 20$$

$$Sh = \frac{k\cdot 2\delta}{D} = 8.23$$

$$k_m = \frac{D_{Acetone-isopropanol}\cdot\varepsilon}{\delta_m\cdot\tau}$$

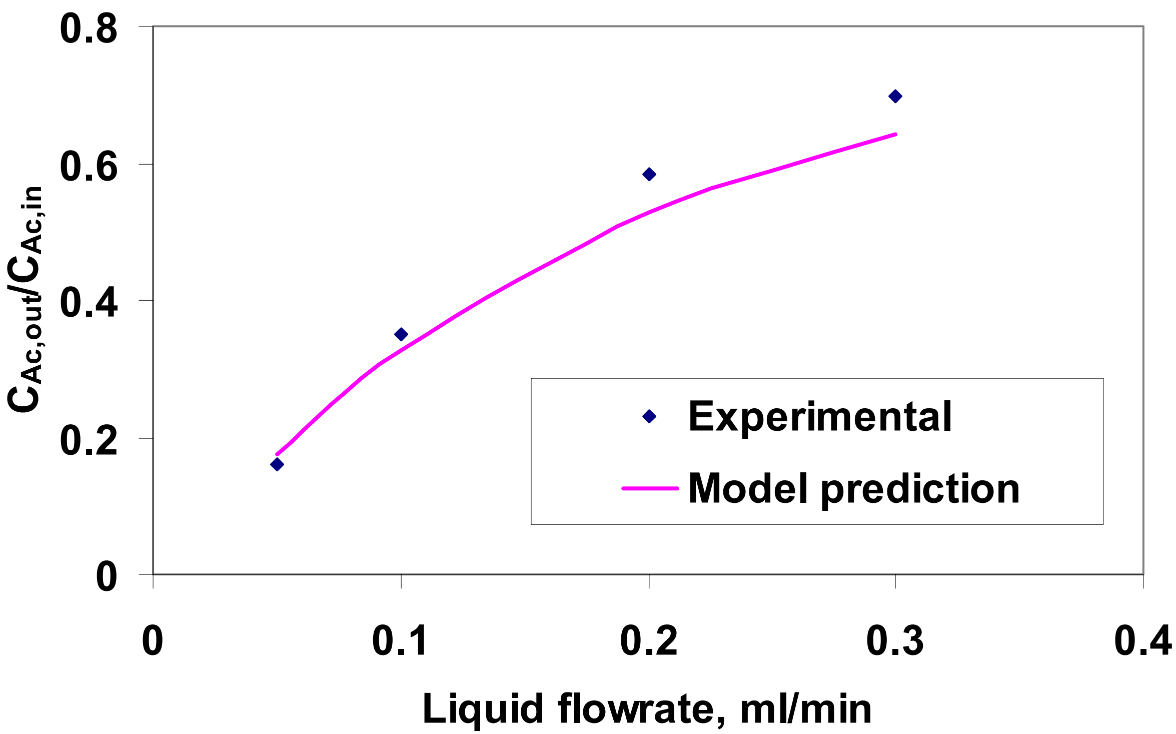
$$\frac{1}{K_T} = \frac{1}{k_l} + \frac{1}{k_m} + \frac{K_{eq}}{k_g}$$

Effect of Nitrogen Flowrate



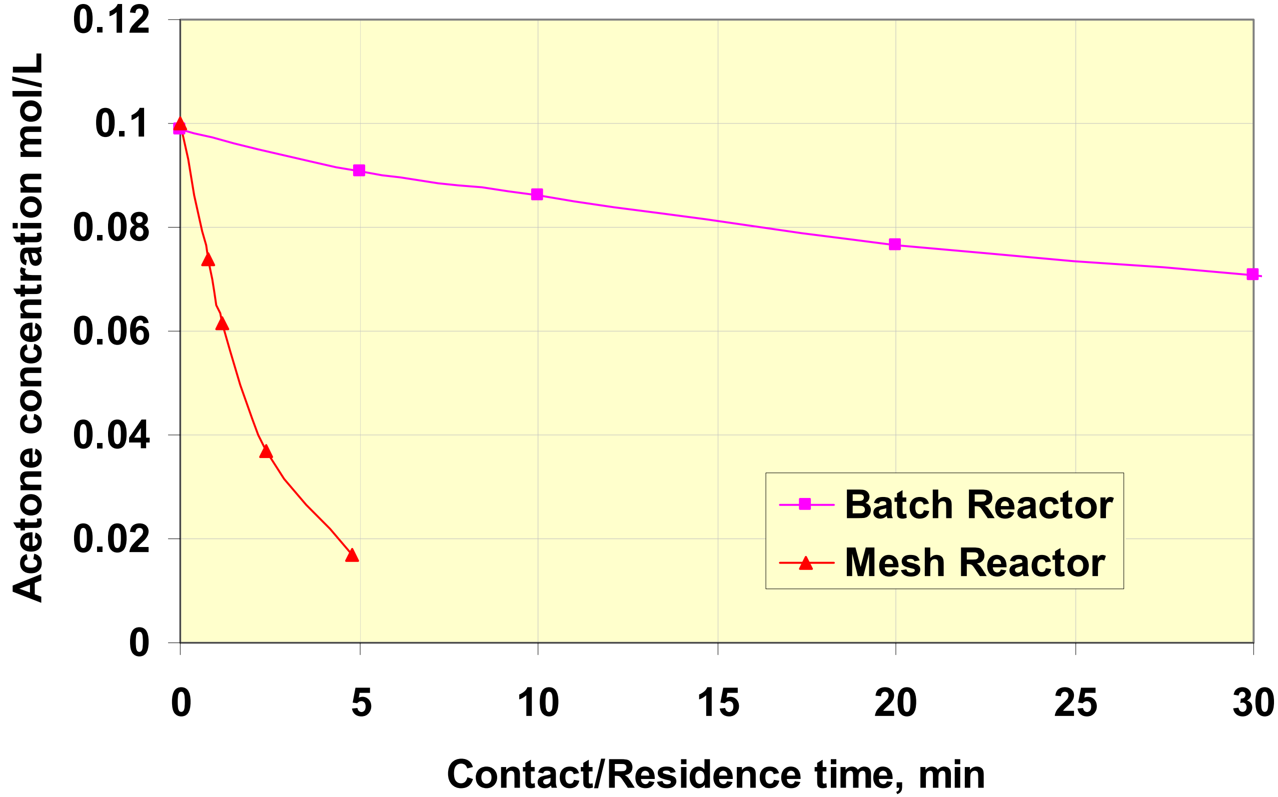
Internetmesh; dry N_2; T=30^{o}C; solvent: isopropanol; liquid inlet flowrate 0.1ml/min, ΔP: pressure difference (P_{gas}-P_{liquid})=30 mm H_2O; Acetone concentration in the liquid inlet=0.1M

Effect of Liquid Flowrate



Internetmesh; dry N_2; T=30°C; solvent: isopropanol; Nitrogen flowrate 70ml/min, ΔP: pressure difference (P_{gas}-P_{liquid})=30 mm H_2O; liquid inlet acetone concentration =0.1M

Acetone Stripping in Batch Reactor and Mesh Contactor



Mesh reactor: Dry N_2 flowrate: 70ml/min; ΔP=50mmH_2O; Temperature=30^{o}C;

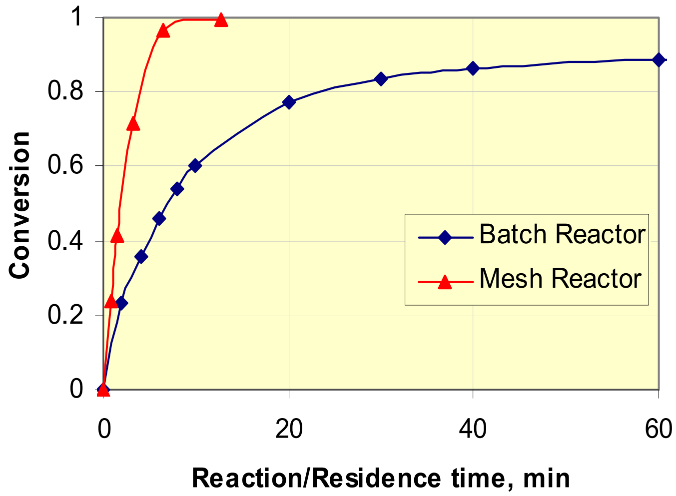
Batch reactor: Dry N_2 flowrate: 800ml/min; Liquid volume:250ml; temperature=30^{o}C

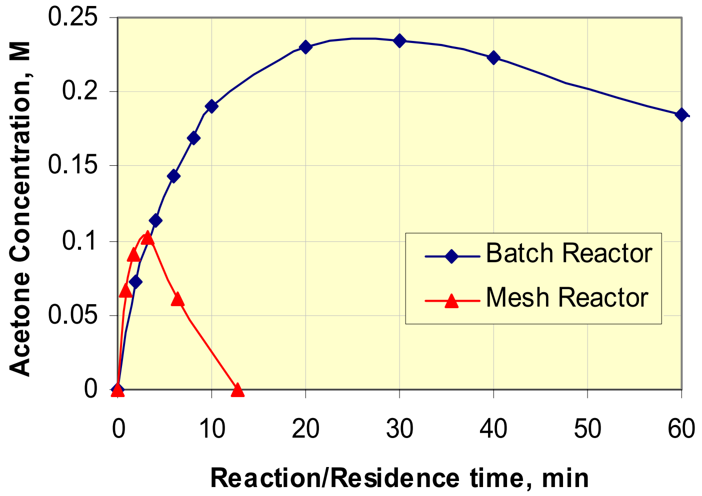
Acetone Stripping with Nitrogen Bubbled in IPA

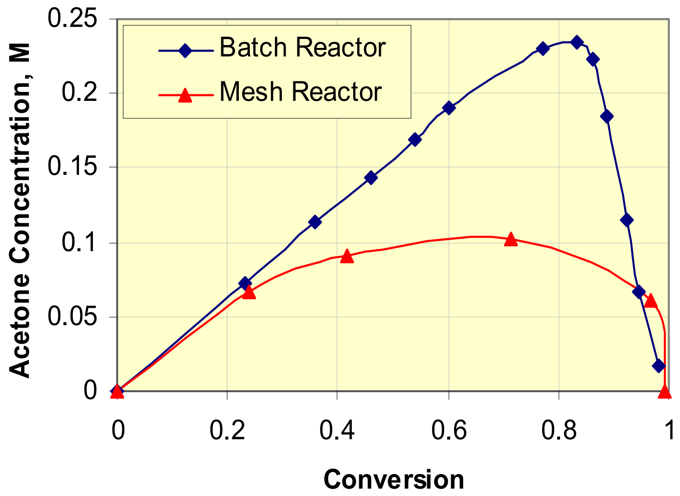
N_2	$F_{L\text{-}in}$	$C_{Ac,in}$	$C_{Ac,out}$	$F_{L\text{-}out}$	Acetone removed
	ml/min	M	M	ml/min	mol/min
Dry	0.1	0.107	0.037	0.079	0.0077
	0.1	0.108	0.038	0.078	0.0079
Bubbled in IPA	0.1	0.102	0.044	0.098	0.0059
	0.1	0.102	0.045	0.097	0.0058

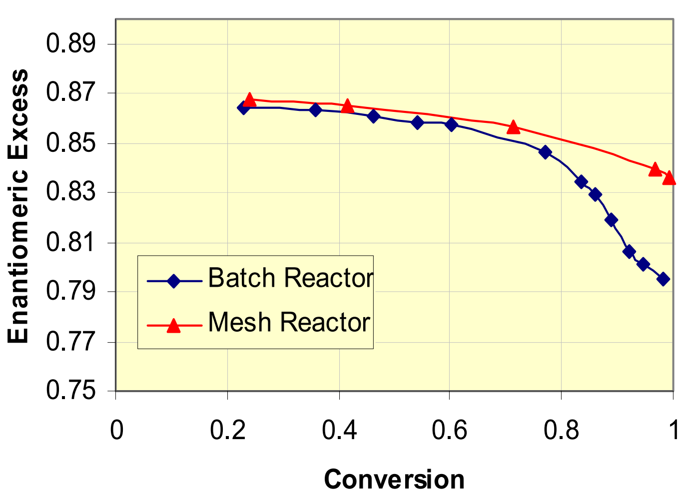
(T=30^{o}C; pressure difference (P_{gas}-P_{liquid})=30 mm H_2O; Nitrogen flowrate=70ml/min)

Asymmetric Transfer Hydrogenation in Batch and Mesh Reactor



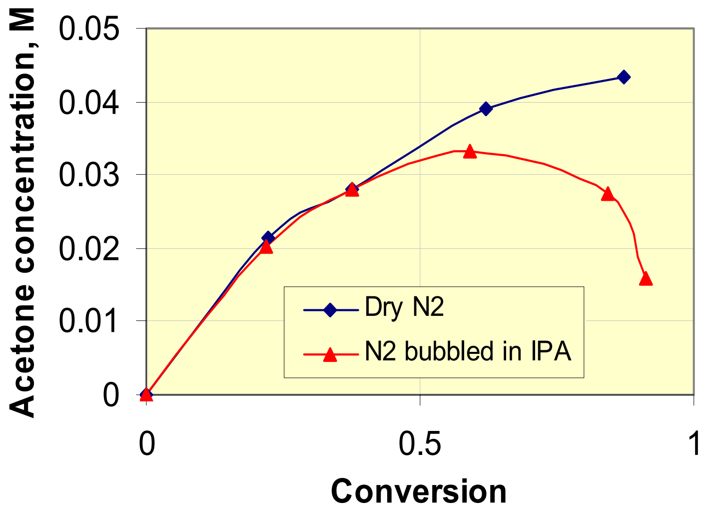


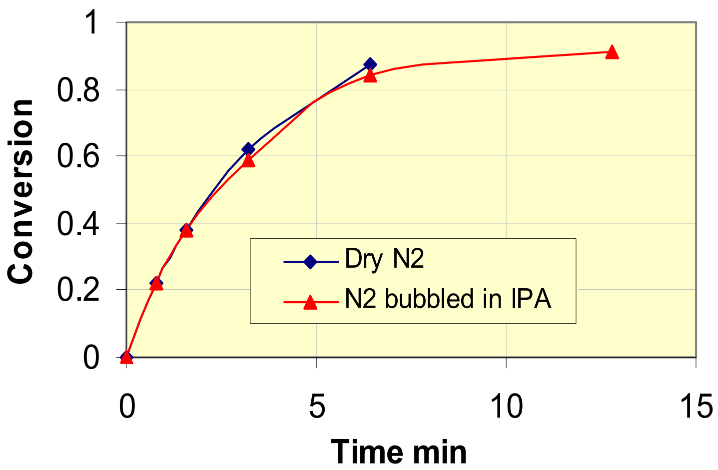


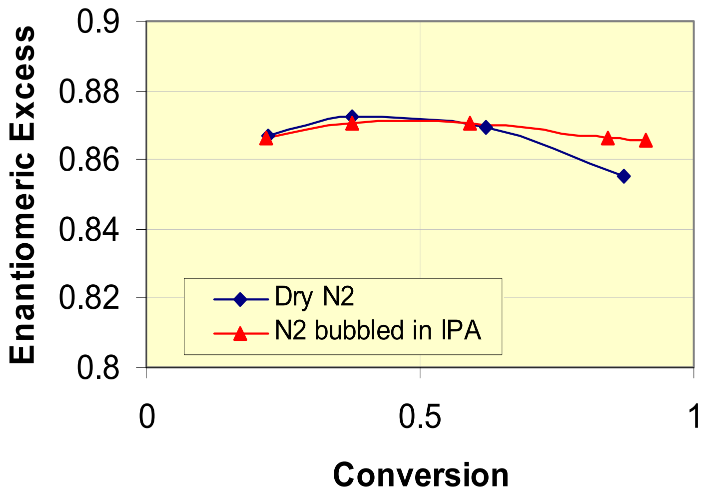


Temperature: 30oC, [Substrate]: 0.33M, [Substrate]/[Catalyst]=1000, In mesh reactor N_2=70ml/min bubbled in IPA. In batch reactor, N_2 flowrate=800ml/min, Reactor volume=500ml; Reaction solution volume=250ml.

IPA Top Up During Asymmetric Transfer Hydrogenation







Reaction conditions:
Reaction temperature: 30oC, N2 flowrate: 70ml/min, [Substrate]: 0.14M; [Substrate]/[Catalyst]=1000)

Mathematical Model

Mole balances in the liquid phase

$$\frac{\partial}{\partial z}\left(u_L \cdot c_i^L\right) = D_i^L\left(\frac{\partial^2 c_i^L}{\partial z^2} + \frac{\partial^2 c_i^L}{\partial {x_L}^2}\right) + R_i$$

Decrease of liquid flowrate

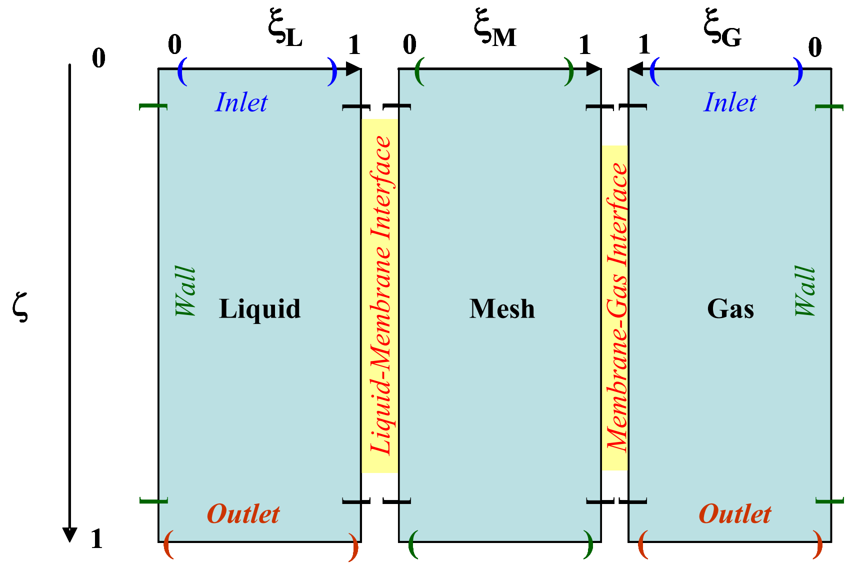
$$\delta_L \cdot \frac{du_L}{dz} = -\frac{P}{R_g T}\left(\sum_{i=1}^{2} D_i^G \left.\frac{\partial y_i}{\partial x_G}\right|_{x_G=\delta_G} \cdot \frac{M_i}{\rho_i}\right)$$

Mole balance in the mesh

$$D_i^L \frac{\partial^2 c_i^M}{\partial {x_M}^2} + R_i = 0$$

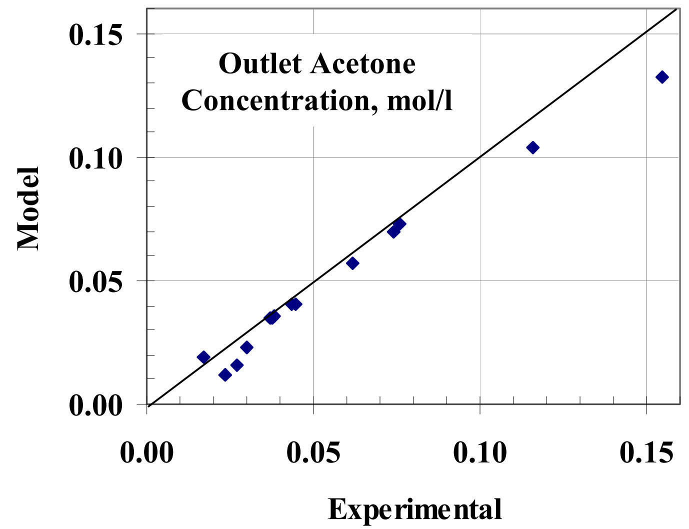
Mole balance in the gas phase

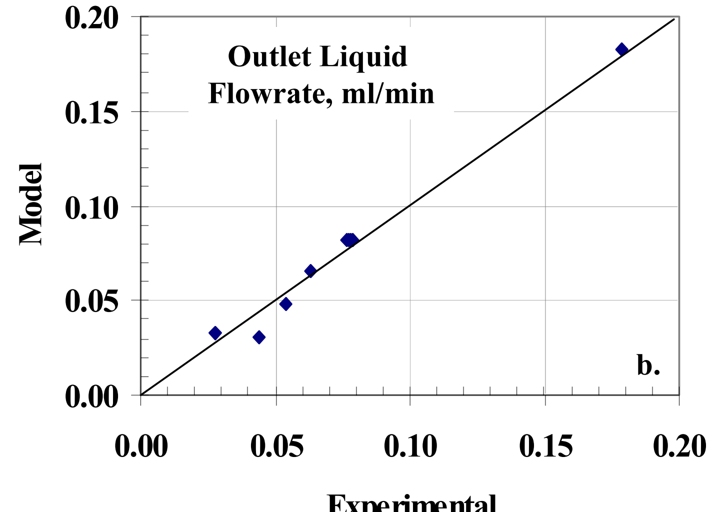
$$u_G \cdot \frac{\partial y_i}{\partial z} = D_i^G\left(\frac{\partial^2 y_i}{\partial z^2} + \frac{\partial^2 y_i}{\partial {x_G}^2}\right)$$



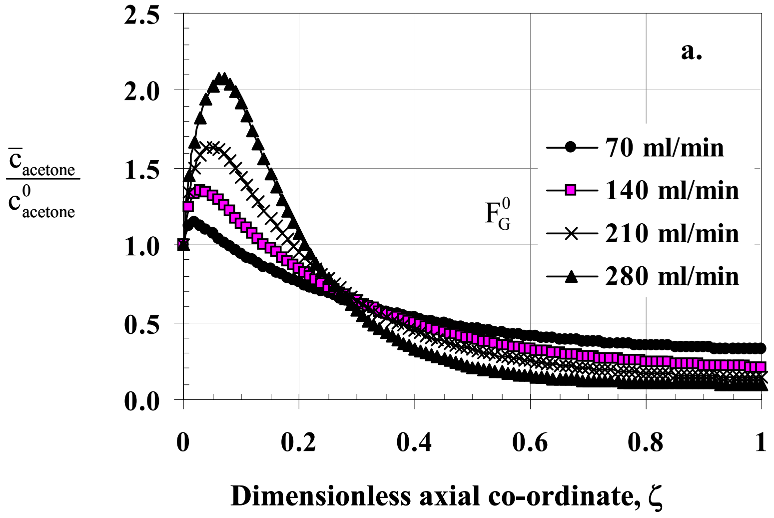
Model Validation

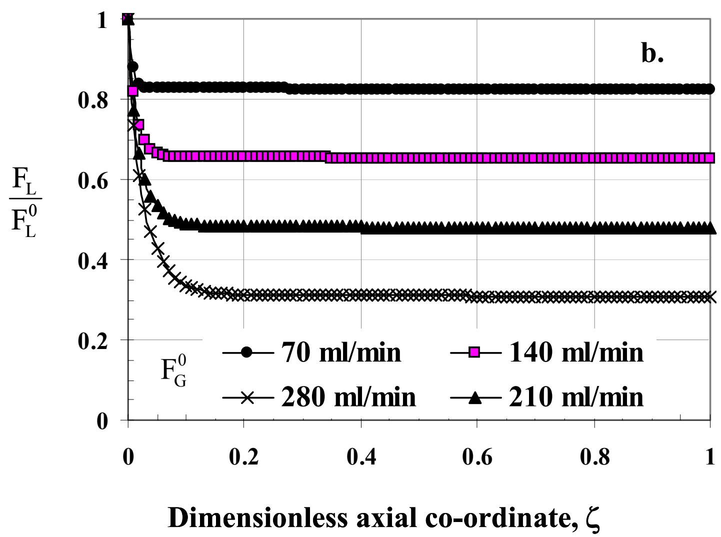
The model predicts well the behaviour of the contactor during acetone stripping from an acetone-isopropanol solution





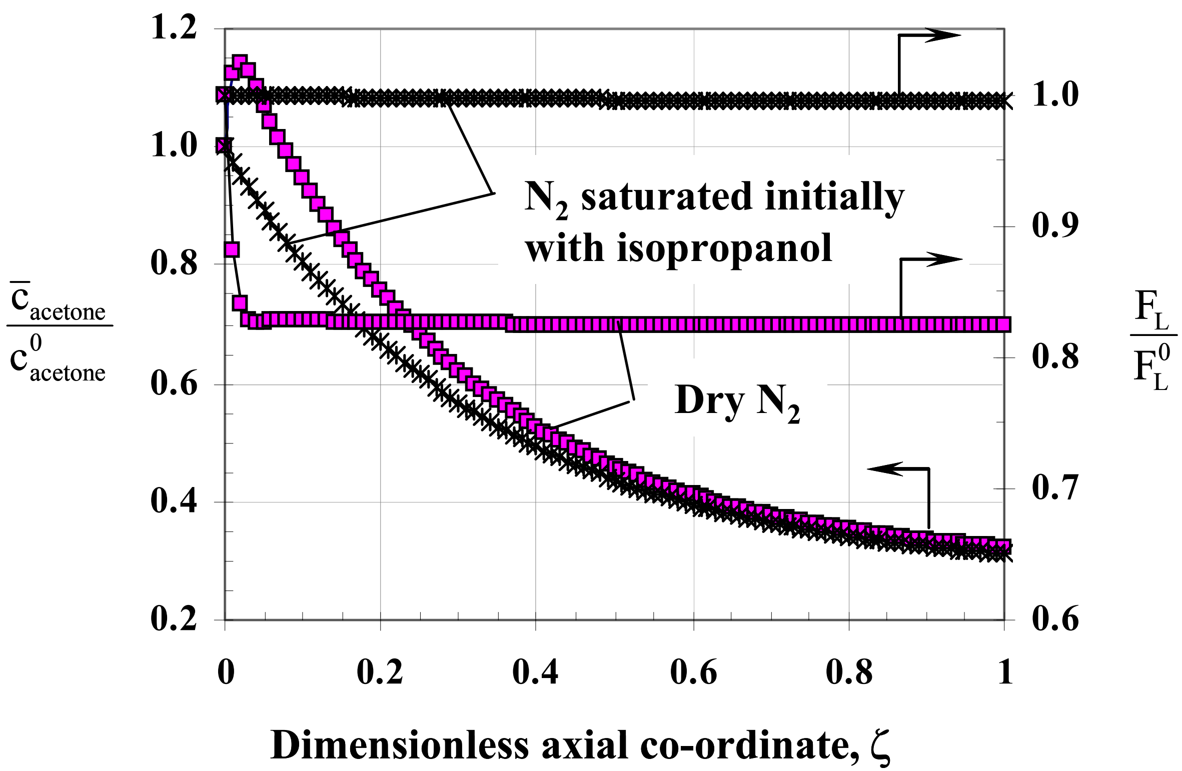
Effect of Increasing Stripping Gas Flowrate





- Increasing gas flowrate improves acetone removal
- Axial peak in acetone is due to isopropanol evaporation
- The stripping gas is saturated with isopropanol within the first 10% of the contactor length

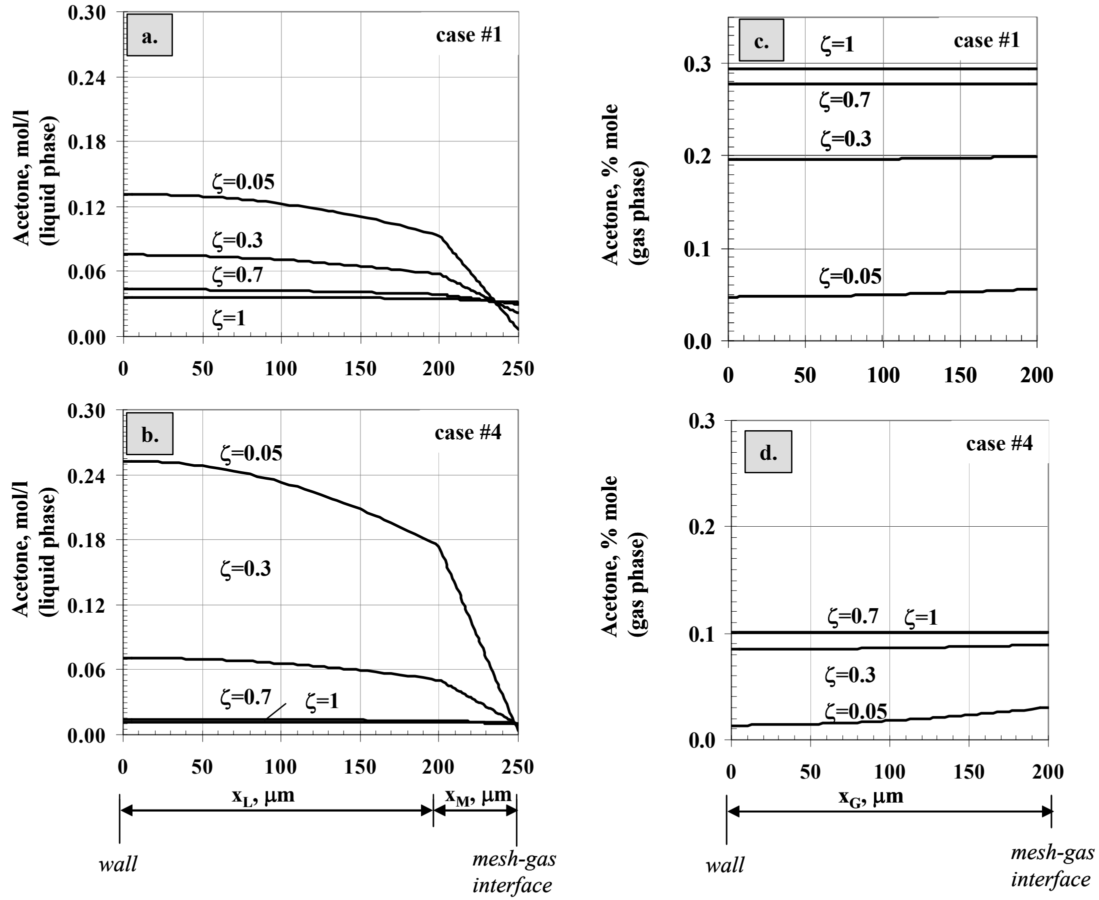
Effect of Stripping Gas Presaturation with Solvent



- **Presaturation removes the acetone peak**
- **Liquid flowrate remains practically constant**

Transverse Concentration Profiles

	Inlet Liquid Flowrate, ml/min	Inlet Gas Flowrate , ml/min
# 1	0.1	70
# 4		280

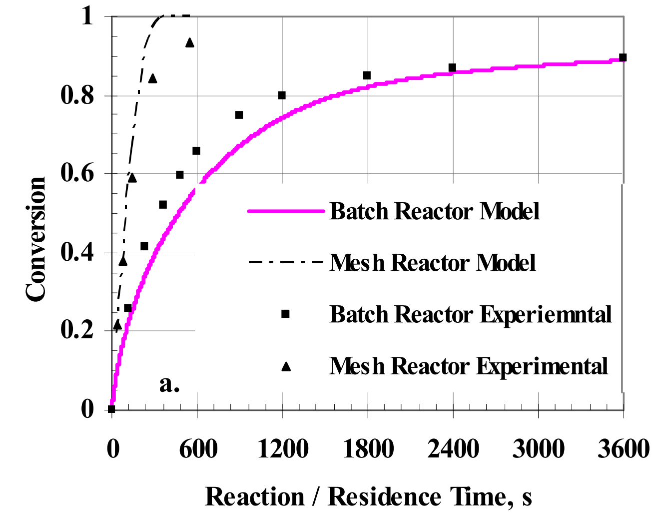


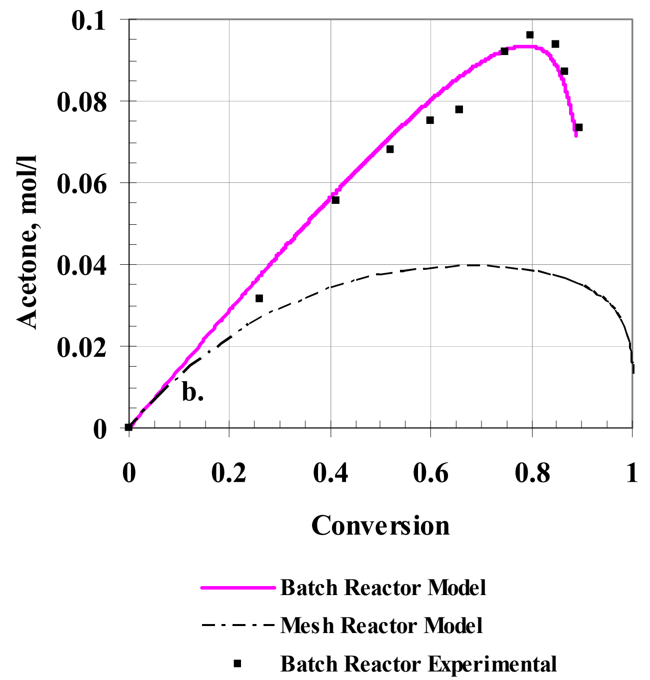
Dominant mass transfer resistance in the mesh

Comparison with Experiments

- Good agreement between model and experimental results for conversion

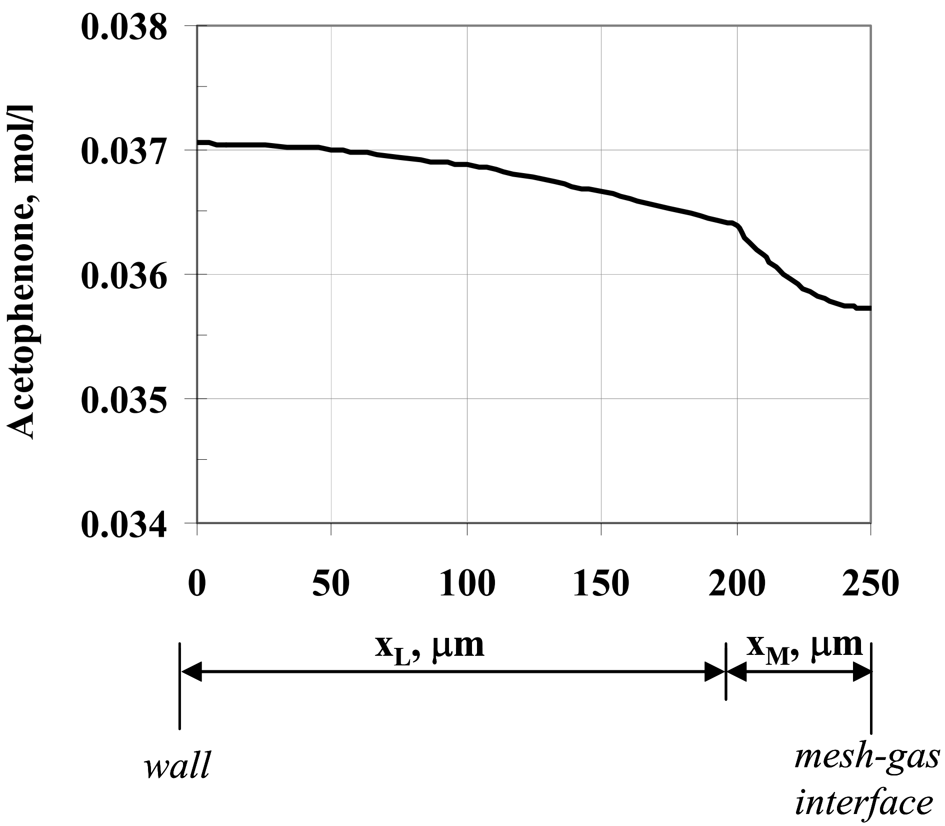
- Discrepancies possibly due to velocity variations along the height and the width of the channel





Reactant radial concentration gradients

Radial concentration gradients obtained for the reactant are small



Concluding Remarks

1. Mesh reactor is very efficient for stripping acetone from isopropanol
2. Higher gas/liquid flowrate ratio gives better acetone stripping performance
3. Bubbling nitrogen into isopropanol can prevent solvent loss
4. Mesh reactor gives better performance for acetone stripping and asymmetric transfer hydrogenation than traditional batch reactor
5. Higher enantioselectivity was obtained with isopropanol top up during the reaction than using dry nitrogen

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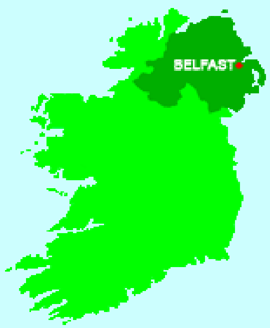

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CenTACat
School of Chemistry &
Chemical Engineering,
David Keir Building,
Stranmillis Road,
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3. *Sustainability through Greener Processing*
   (**Professor Volker Hessel, Eindhoven University of Technology / IMM Mainz**)

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(**Professor Graham Hutchings**)

Catalysis in a sustainable fine chemical industry, to include chemicals from renewables
(**Professor David Jackson**)

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