AM Technology
Engineering Chemistry

Agitated Cell and Tube Reactors

PIN NL, May 11th 2011

13:45 to 14:20

Robert Ashe AM Technology

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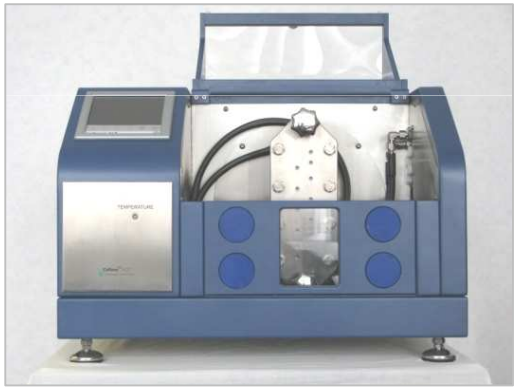
AM Technology

- Based in the UK
- Founded in 2000
- Manufacture chemical reactors
- Strong focus on innovation



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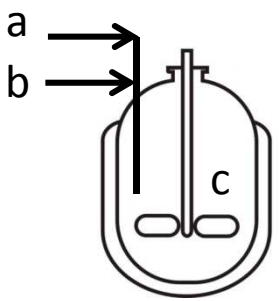
Agenda

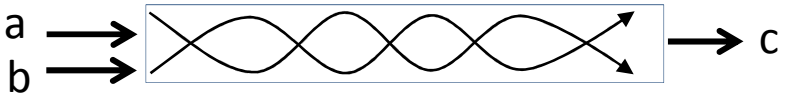
- Static mixing versus dynamic mixing
- Coflore Agitated Cell/Tube Reactors
- Results

Process intensification



Batch reactor

Continuous reactor (with orderly flow)



Orderly (plug) flow
Process material travels through and leaves the reactor in the same order that it enters

- Optimum reactor capacity
- Improved reaction time control
- Higher yield
- Higher purity

Flow reactors – controlling cost is a key objective

Minimise pressure drop
Reducing the pressure drop reduces pump costs, especially for corrosive fluids.

Minimise tube length
Short large diameter reactor tubes are cheaper per unit volume than long small diameter tubes. Large diameter tubes can also have significantly longer service lives than small diameter tubes

Two phase mixtures
Reliable handling of two phase mixtures (where applicable), is essential for minimising disruption and waste

Versatility
For cost effective operation in a multi step, multi product manufacturing environment, flow reactors need to be multi purpose

Statically mixed reactors

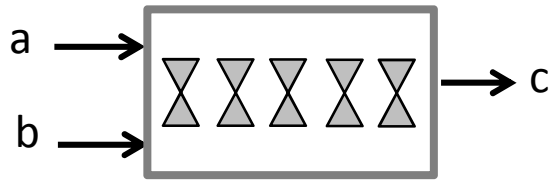
In small tubes (typically < ½ mm diameter), mixing and orderly flow are not dependent on fluid velocity. Small tubes however have high pressure drop, high cost per unit volume and poor handling capabilities for two phase mixtures

In large tubes, mixing and orderly flow rely on fluid changing direction as it flows through the reactor. This can be achieved with baffles, static mixers or turbulent flow. Axial velocity becomes a quality critical parameter.

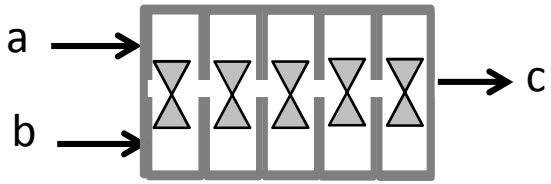
Mixing and orderly flow is dependent on fluid velocity through the channel

- Long tube lengths with long reaction times
- Limited flexibility and high minimum throughputs
- High pressure drop
- Poor handling of two phase mixtures
- Poor mixing with long tube lengths

Dynamically mixed reactors



Dynamic mixing generates good mixing independently of net fluid velocity



Stage separation (or long tubes) prevents back mixing independently of net fluid velocity

Good mixing and orderly flow is achieved independently of tube diameter, tube length or fluid velocity through the reactor

- Good mixing and orderly flow independently of fluid velocity
- Short tube lengths
- High flexibility
- Low pressure drop
- Good handling for two phase mixtures (due to large diameter and good mixing)

Dynamically mixed flow reactors deliver substantial cost advantages through shorter tubes, lower pressure drop and flexibility

Flow conditions for turbulent flow

Can be run under laminar flow conditions →

Reactor type	Tube diameter	Tube length per litre	Minimum flow for turbulent flow*	Pressure drop , 30 second reaction at minimum flow
	mm	m	litres/hour	bar
Micro	0.5	5,090	3.3	1880
Static mixed tube	1	1,270	6.6	104
Static mixed tube	5	51	33	0.15
Static mixed tube	10	13	66	0.01
Static mixed tube	40	<1	263	0.0004
Dynamic mixed tube	**40**	**<1**	**0**	**<0.0001**

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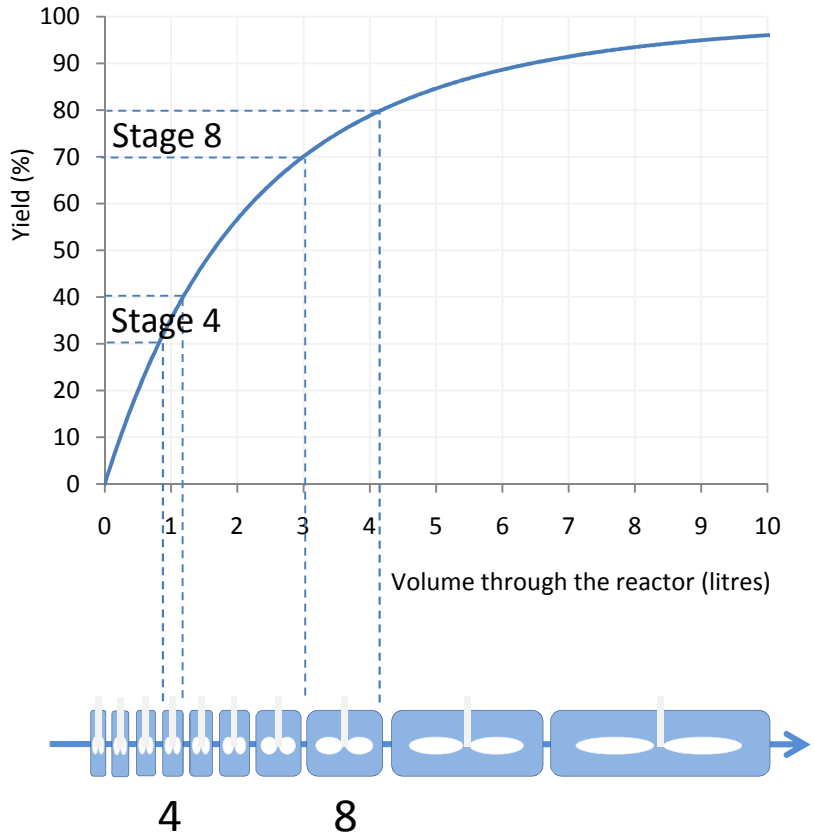
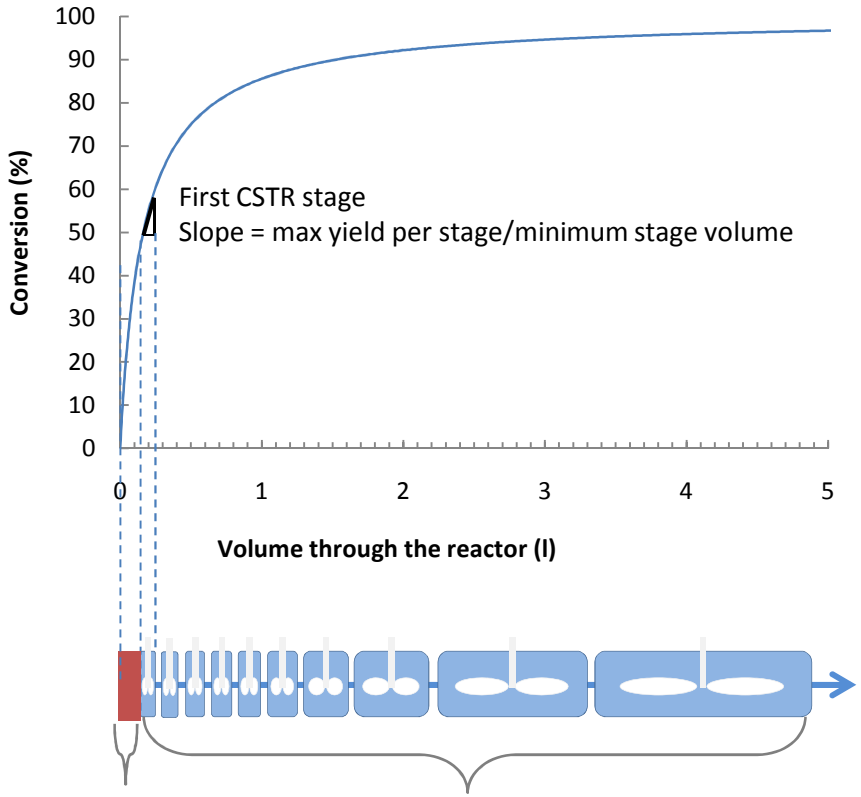
Scale up design is simpler in multi stage dynamically mixed flow reactors but attention must be given to kinetics. The reactor is treated as a series of equal conversion stages



Where the reaction rate or heat evolution exceeds the minimum size constraints of a stirred stage, micro reactors can be combined with the dynamically mixed system



Micro reactor
50% of reaction 3% of reactor volume

Stirred tanks in series
50% of reaction 97% of reactor

= >95% reduction in tube length and pressure drop)

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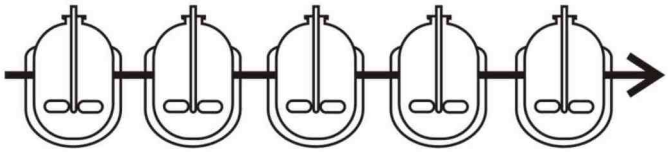
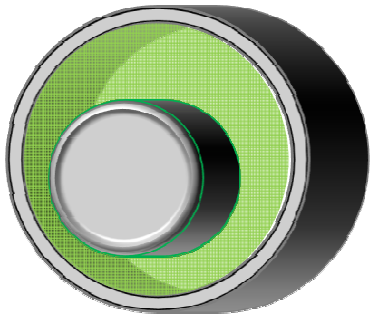
Conventional rotating stirrers are difficult to use in flow reactors



- Sealing problems (cost, leaks, buffer fluid leak, pressure limits)
- Shaft stability problems (with long axial shafts)
- Centrifugal separation problems (two phase mixtures)
- Baffle design problems (baffles difficult to fit)

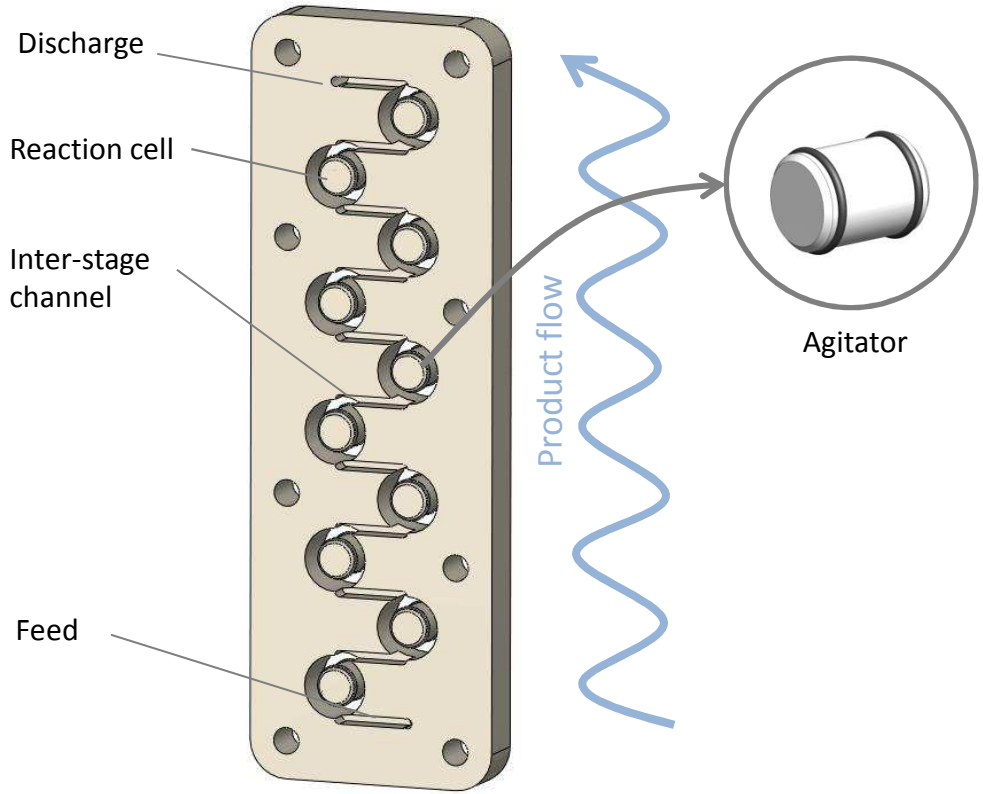
Coflore reactors use transverse shaking to generate mixing



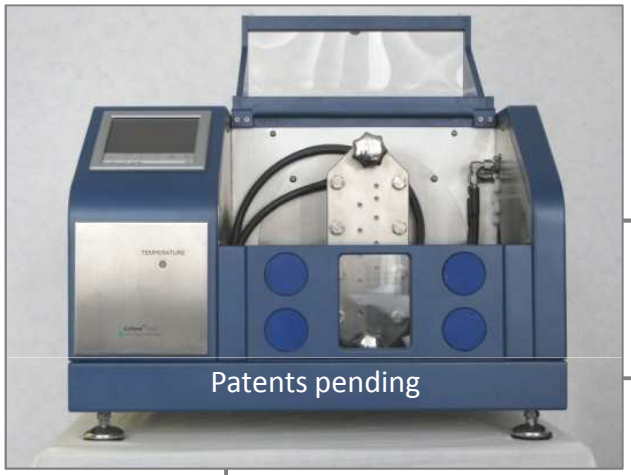
Coflore - an alternative method of mixing

- High mixing efficiency (radial)
- No baffles (self baffling)
- No seals or magnetic couplings
- No centrifugal effects
- No shaft stability problems

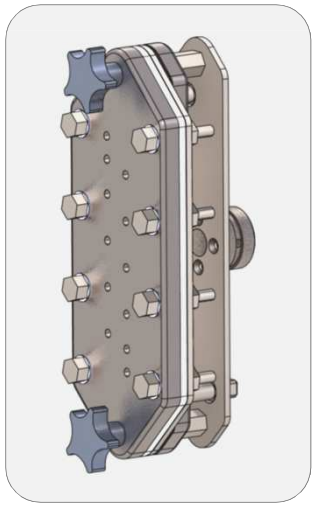
In small systems the reaction stages are cut within a monolithic block



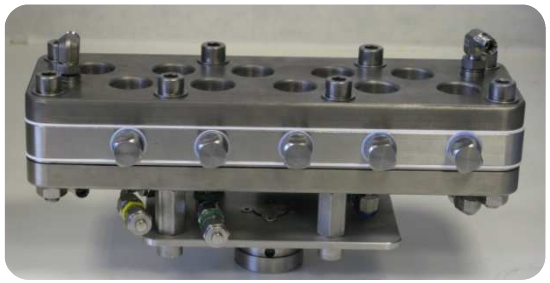
The bench top shaker platform can handle a range of reactor blocks



Counter current reactor block

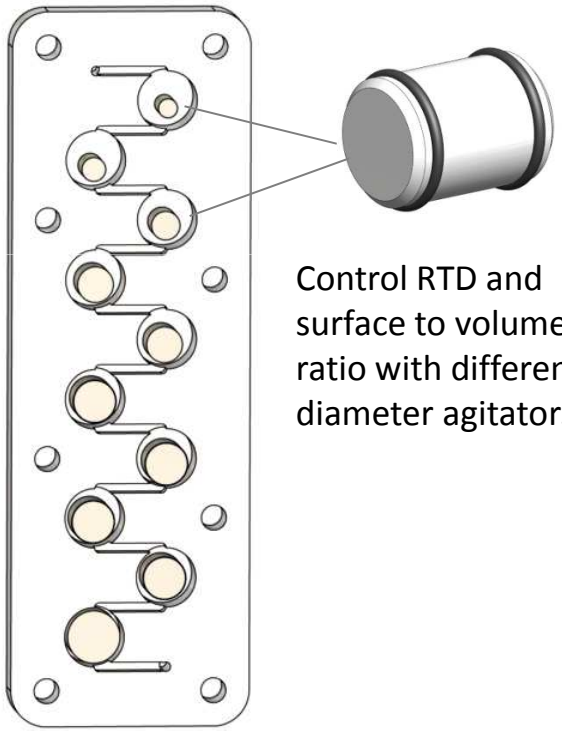


Standard 10 millilitre reactor block



Standard 100 millilitre reactor block

Interchangeable agitators are used for different applications



Control RTD and surface to volume ratio with different diameter agitators

Ceramic agitator

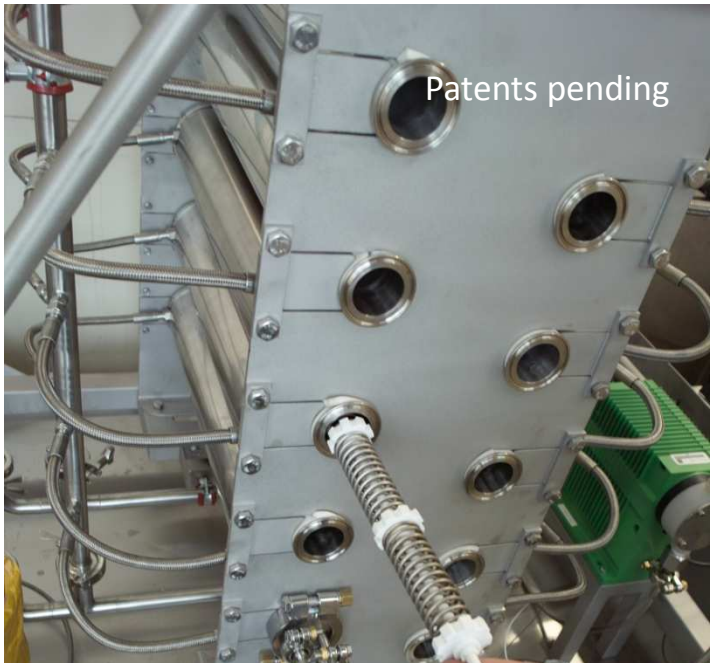
Spring agitator for two phase mixtures

Hastelloy agitator

Basket agitator for handling catalyst

Industrial scale Coflore reactors used tubes with free moving mixing elements

- Operating capacity - 1 to 10 litres
- 10 temperature control zones
- High design pressure/temperature
- Low pressure drop
- High mixing efficiency



Coflore performance

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Tests have consistently delivered good mixing, good handling of two phase mixtures and low pressure drop

Mixing
Homogenous fluid - comparable to >4m/s in static mixer
Immiscible fluids - comparable to 400 rpm in a 1 litre
Gas/liquid comparable to >600 rpm in a 1 litre batch vessel

Two phase mixtures
Slurries, immiscible liquids, gas/liquid mixtures

Pressure drop

Generally less than 0.01 bar

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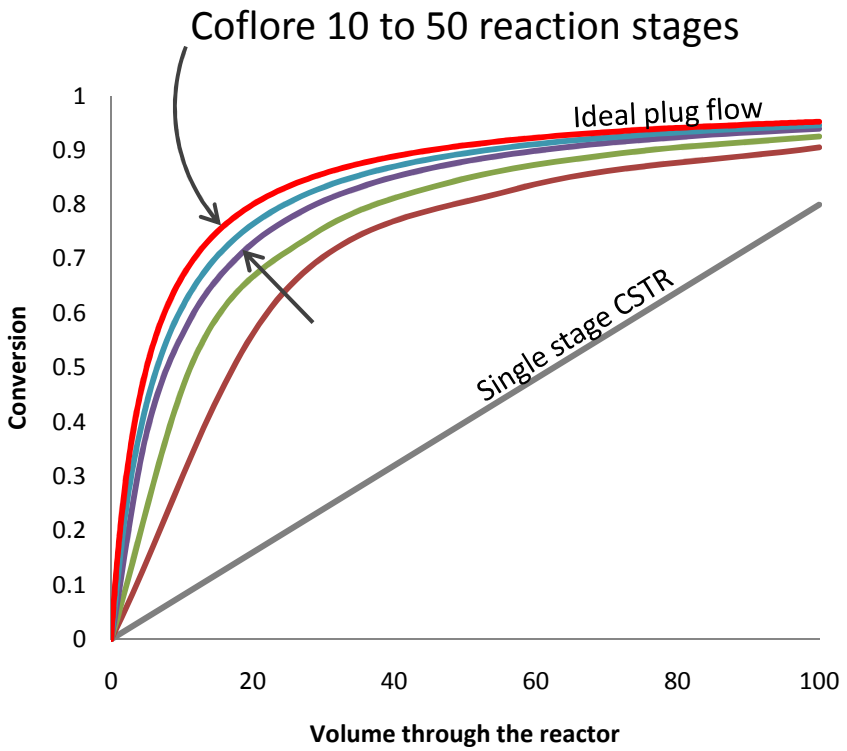
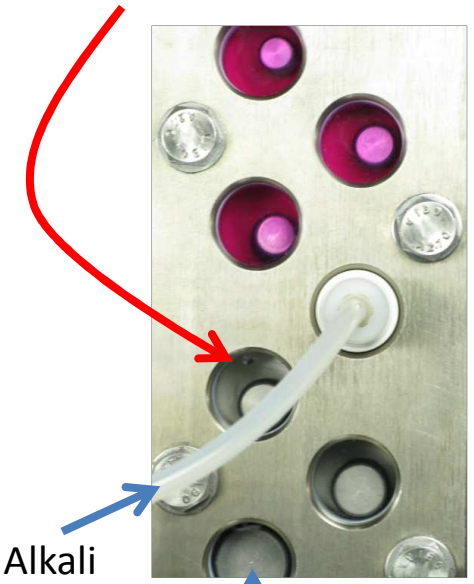
Orderly flow is maintained by using multiple stages + tube length



Zero back mixing with
>3 hour reaction time



Test results

Two stage reaction with gas and solids

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2 stage reaction for
nanoparticle synthesis

Step 2 : nanoparticle synthesis
Gas formation
Clumps of nanoparticles

Step 1 : gold solution + ligand

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Good scale up characteristics for mixing sensitive reactions

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Coflore

Homogenous reaction
High yield, low pressure drop
Quick to configure
0.1 – 100 litres per hour

$A + B \rightarrow P$
$B + B \rightarrow$ unwanted products
$B + P \rightarrow$ unwanted products



	Fed-batch	Fed-batch	ACR
T ,°C	20	20	20
Volume , ml	1-2	2000	60
Reaction time , minutes	5	10	5
Yield	90 %	64 %	94 %

5 litres an hour

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Lab scale - mixing sensitive reaction with gas, liquid and solids

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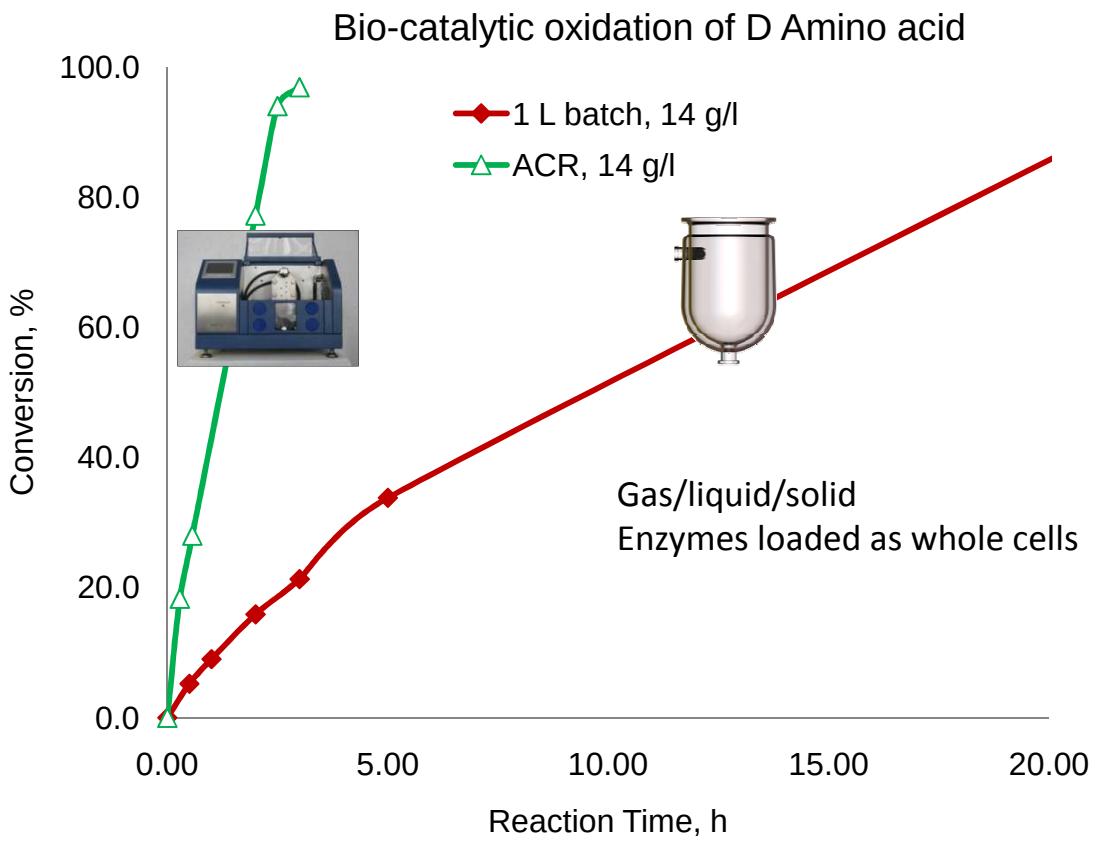
Oxidation of D Amino acid
Improved reaction rate, no blockage



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Industrial scale - mixing sensitive reaction with gas, liquid and solids

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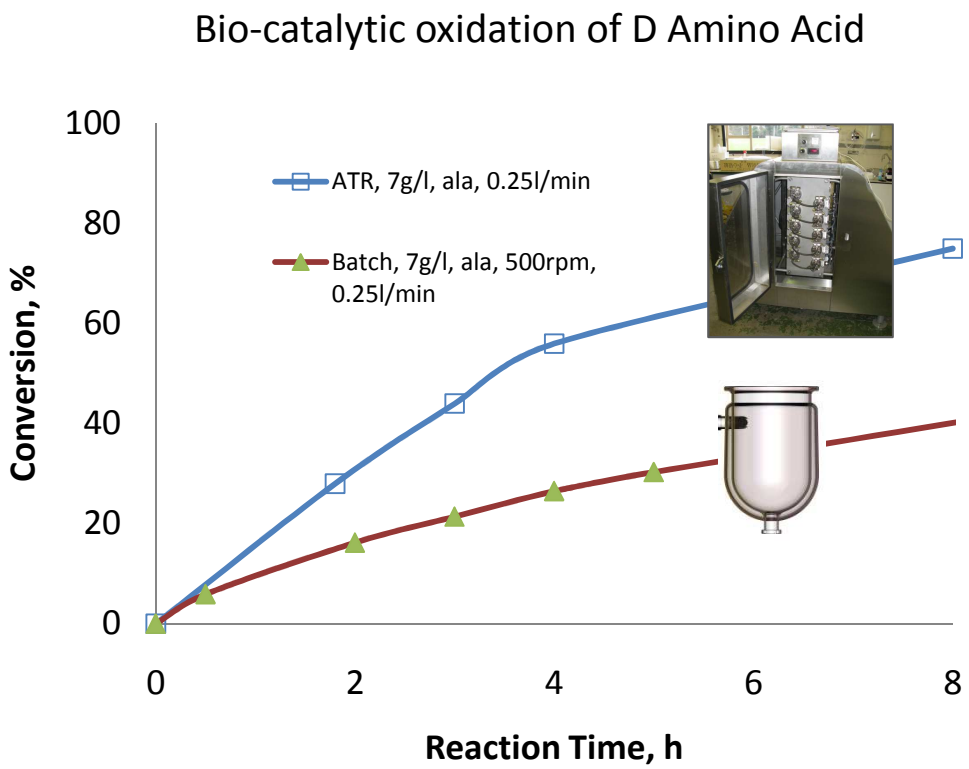
First results at the 10 litre scale with slow agitation speed.
Reactor length 4 metres



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Coflore reactors have handled a wide range of reactions

- Suzuki reaction
- Hoffmann reaction
- Grignard reaction
- Biocatalysis
- Nitration
- Hydrogenation
- Bourne reaction
- Oxidation of D Amino acid with live cells and oxygen
- Emulsion polymerisation
- N-iodomorpholinium hydroiodide salt
- Nano particle synthesis
- Acid treatment of wood pulp (10% by weight, ≈ 30% by volume)
- Counter current extraction

Conclusion

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1. Dynamic mixing combined with stage separation deliver:

   - Flexibility
   - High capacity per unit length in short tubes
   - Excellent mixing and good plug flow
   - Low pressure drop
   - Good handling of two phase mixtures
   - Inherently simpler to scale up

2. Used in combination with a micro reactor, CSTR's can deliver optimum performance and cost for a wide range of reaction types

3. Historically, dynamically mixed flow reactors have been difficult to use due to the cost and technical challenges of employing multiple dynamic mixers in small cells and long tubes. The Coflore reactor addresses these problems and can be used for a broad range of applications.

Thank you

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