ENERGY – PART II

Contents of the lecture

High-Gravity (HiGee) fields in chemical processing

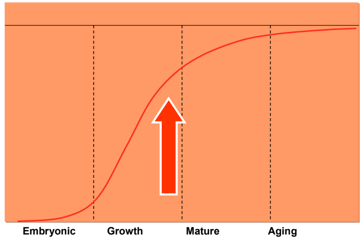
- Rotating Packed Bed (RPB)
- Spinning Disc Reactor (SDR)

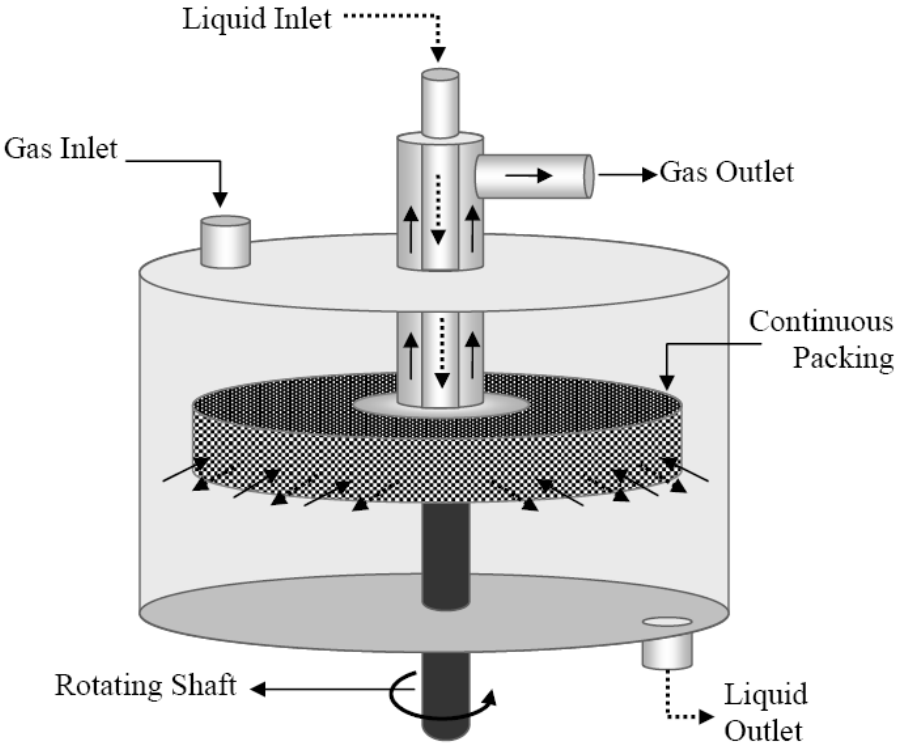
Processing in High-Gravity Fields - history

- dates to the beginning of the industry (pumping, compression, and solid/liquid separations)
- mass and heat transport operations such as liquid/liquid extraction is a much more recent development
- 1945 - commercial application of a centrifugal liquid extractor to recovery of penicillin, based on earlier patents by Podbielniak

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Basic process equipment – Rotating Packed Bed

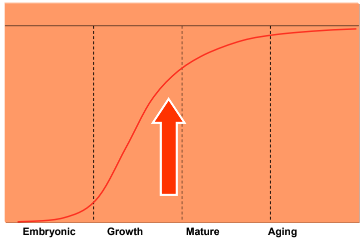


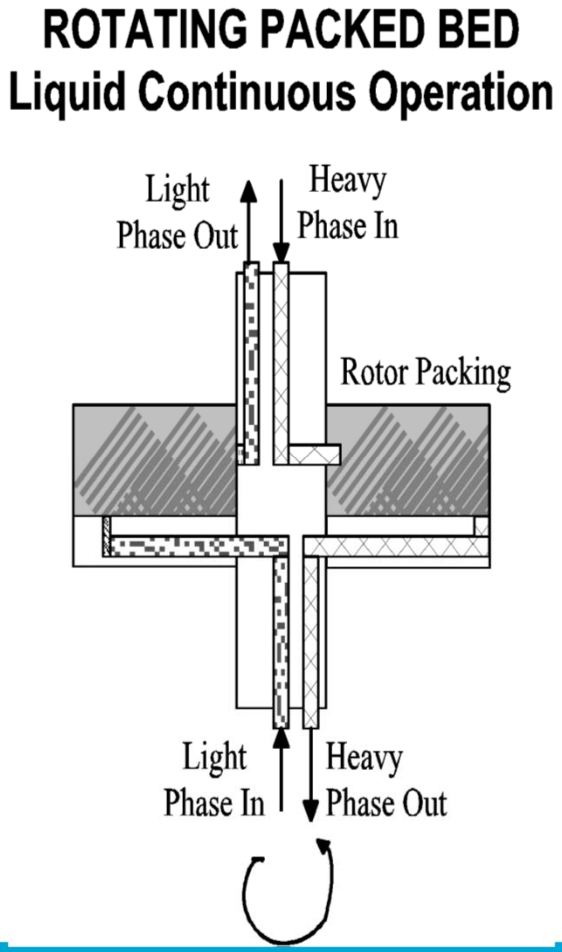


- Liquid enters at the eye of the rotor, being distributed on the rotor packing at the inside diameter.
- The centrifugal force of the spinning rotor accelerates the liquid radially outward.
- Gas enters the stationary housing and passes through the rotor from outside to inside.
- The gas exits at the eye of the rotor, while liquid drains from the housing.

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Basic process equipment – Rotating Packed Bed

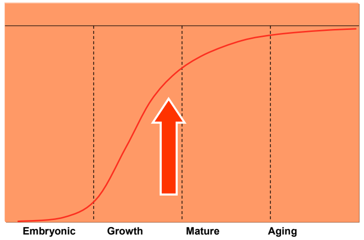


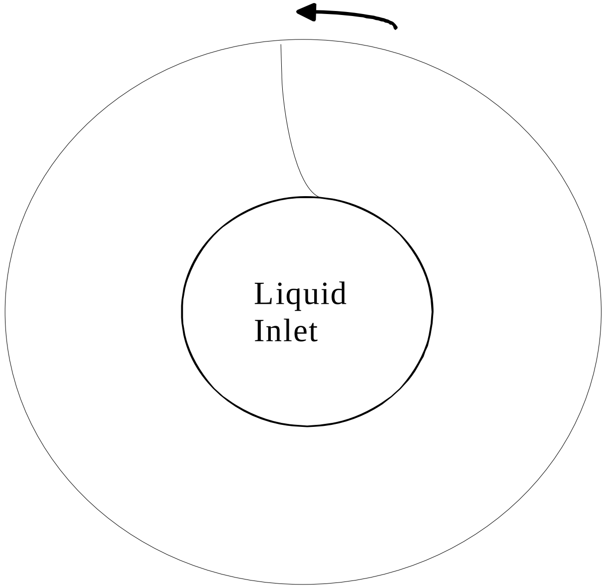


- For **liquid/liquid extraction**: the light phase enters through the drive shaft and channels radially in the rotor end plate to the outer periphery of the rotor for distribution into the heavy phase

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Hydrodynamics



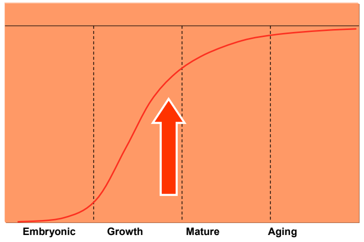


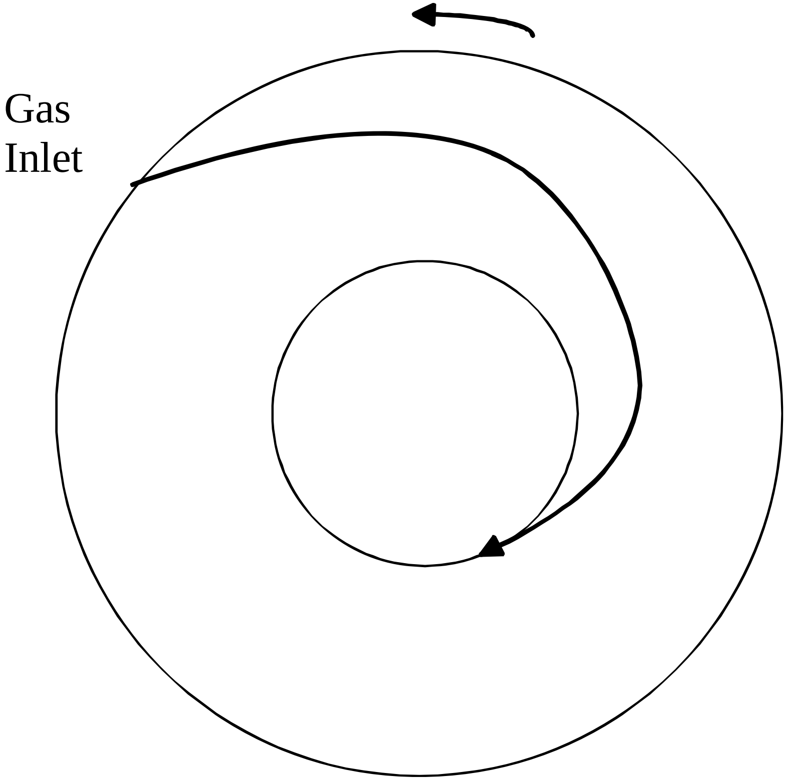
- Flow basically in the radial direction with very little tangential or axial spreading.
- A slight curvature in the radial flow results from the direction of rotation.

  The degree of curvature and spreading is primarily a function of rotor speed and liquid viscosity and less a function of packing type and liquid flow rate.

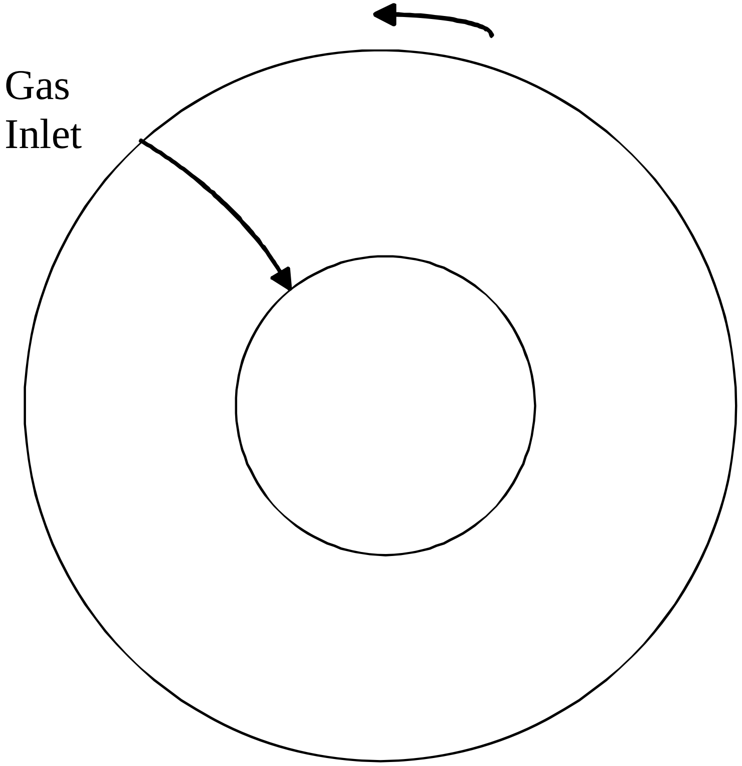
(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Hydrodynamics





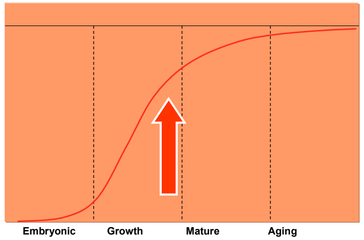
Relative path of gas in a **rotor of low resistance (e.g. parallel flat plates).**



Relative path of gas flow in **rotor of high resistance (low porosity, high surface area).**

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

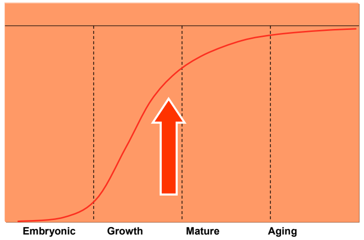
Hydrodynamics

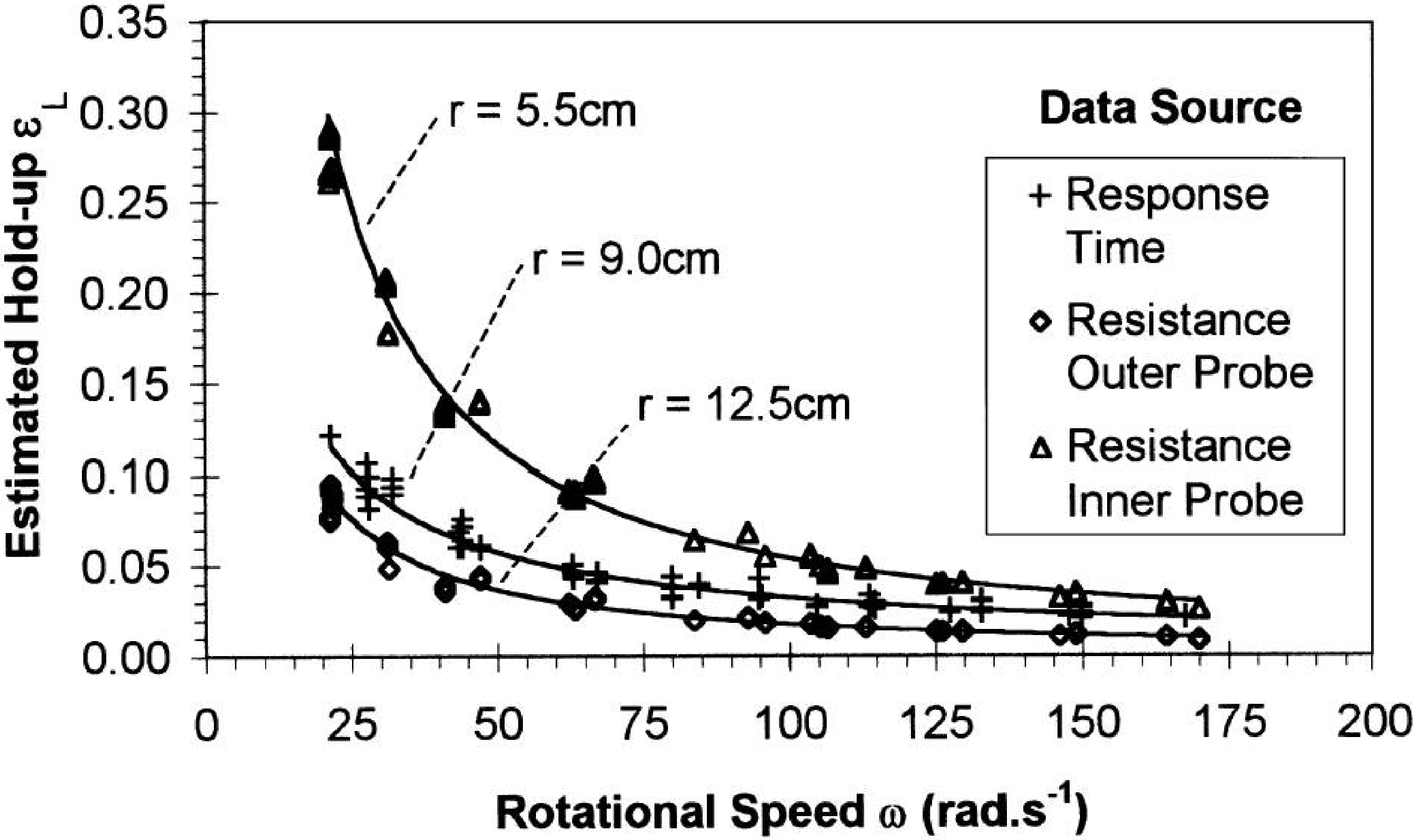


- Film thickness usually varies between 10 and 80 microns (water).
- Film flow is laminar.
- In addition to rotor speed, liquid flow rate and fluid properties impact the film thickness.

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

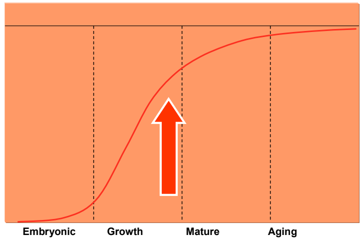
Hold-up





(source: J.R. Burns et al., Chem. Eng. Sci., 55 (2000), 2401-2415)

Flooding



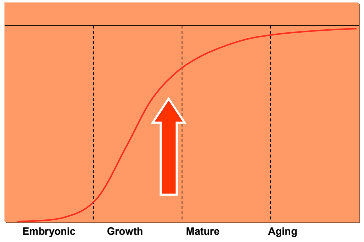
- Sherwood flooding correlation for packed towers is expressed as a plot of

$$\frac{U_G^2 a_p}{g\varepsilon^3}\left(\frac{\rho_G}{\rho_L}\right)\mu_L^{0.2} \qquad \text{versus} \qquad \frac{L}{G}\left(\frac{\rho_G}{\rho_L}\right)^{0.5}$$

- flooding correlation for packed towers applied equally well to RPBs when the gravity term (g) was replaced by centrifugal acceleration ($r\omega^2$).

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

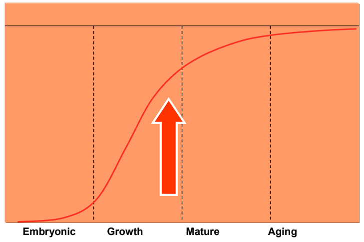
Residence time



- Varies as a function of packing depth, packing type, rotor speed, and liquid properties.
- Measured liquid residence time ranges from about 0.2 seconds to about 1.8 seconds.
- Time decreases with the rotor speed and with liquid flow rate.

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

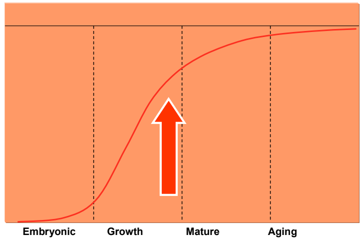
Mass transfer



- intensive mass transfer resulting in height of transfer unit (HTU) values of 1.5-4 cm.
- atomization of the liquid creates high surface area liquid drops in addition to the film wetting of the packing; this results in significant mass transfer apart from the packing surface.
- as a result low surface area packings produce equivalent volumetric mass transfer coefficients as compared to high surface area packings; the result is lower cost packing, reduced pressure drop, and higher throughput.

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

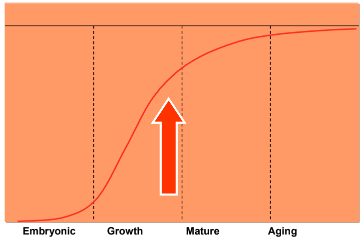
Mass transfer

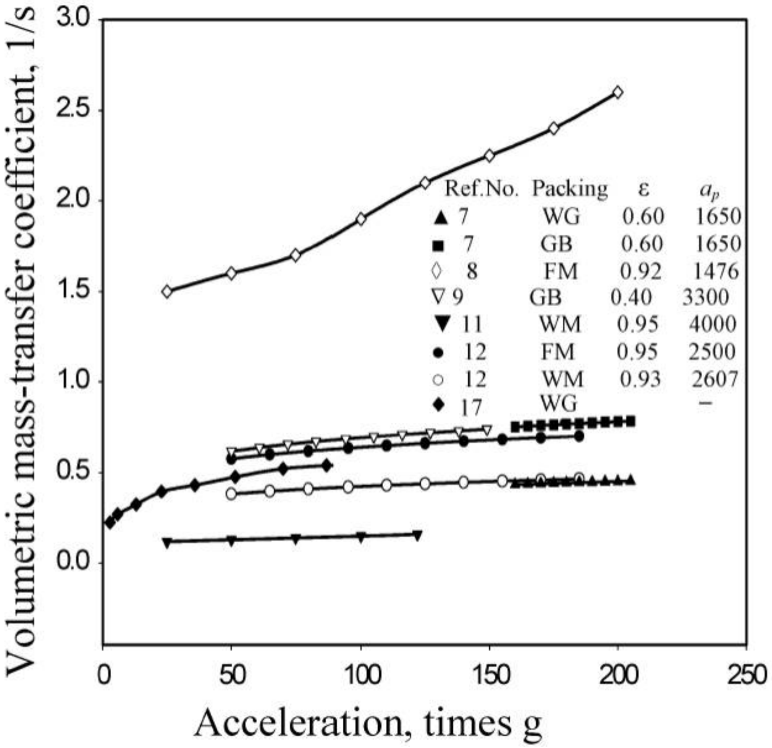


- operational parameters of importance: rotor speed, liquid rates, and gas/liquid ratios. Mass transfer increases proportionately to rotor speed, decreases with increasing liquid flow, and increases with gas/liquid ratio .
- gas-side mass transfer coefficients of 40-50 s^{-1} are achieved .
- liquid-solid mass transfer – limited number of studies available. Water flow over naphthalene pellets provided 4-6 times higher volumetric mass transfer coefficients compared to gravity flow at similar superficial liquid velocities

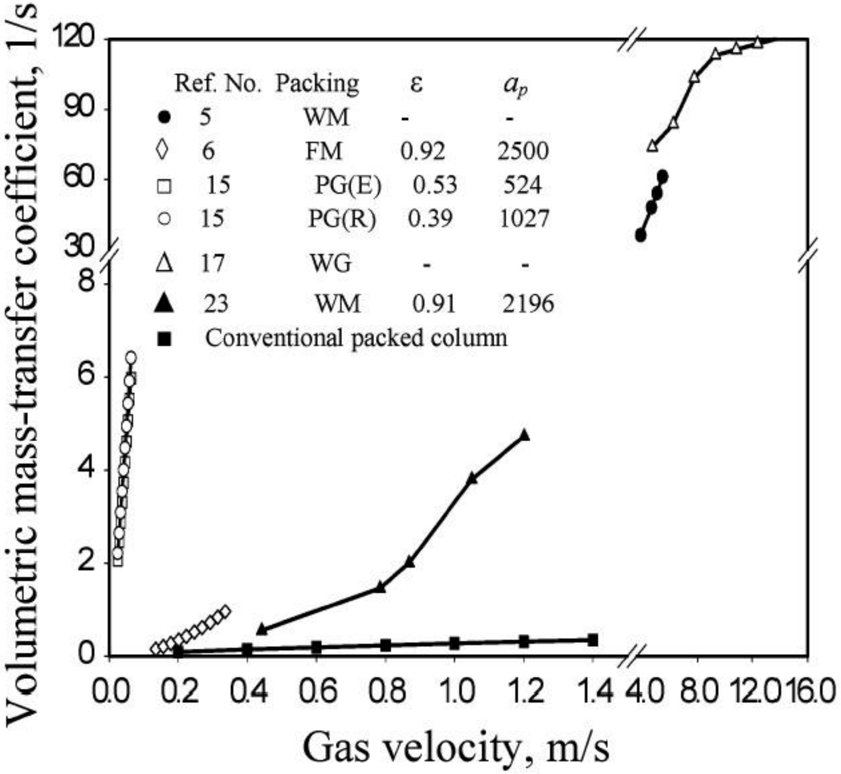
(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Mass transfer





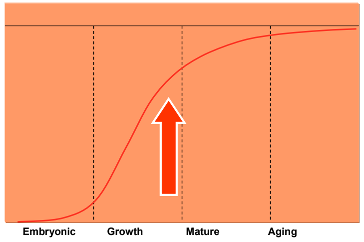
Liquid-side



Gas-side

(source: D. P. Rao et al., Ind. Eng. Chem. Res., 43 (2004) 1150-1162)

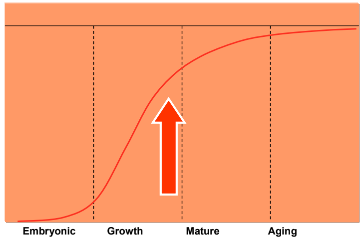
Pressure Drop

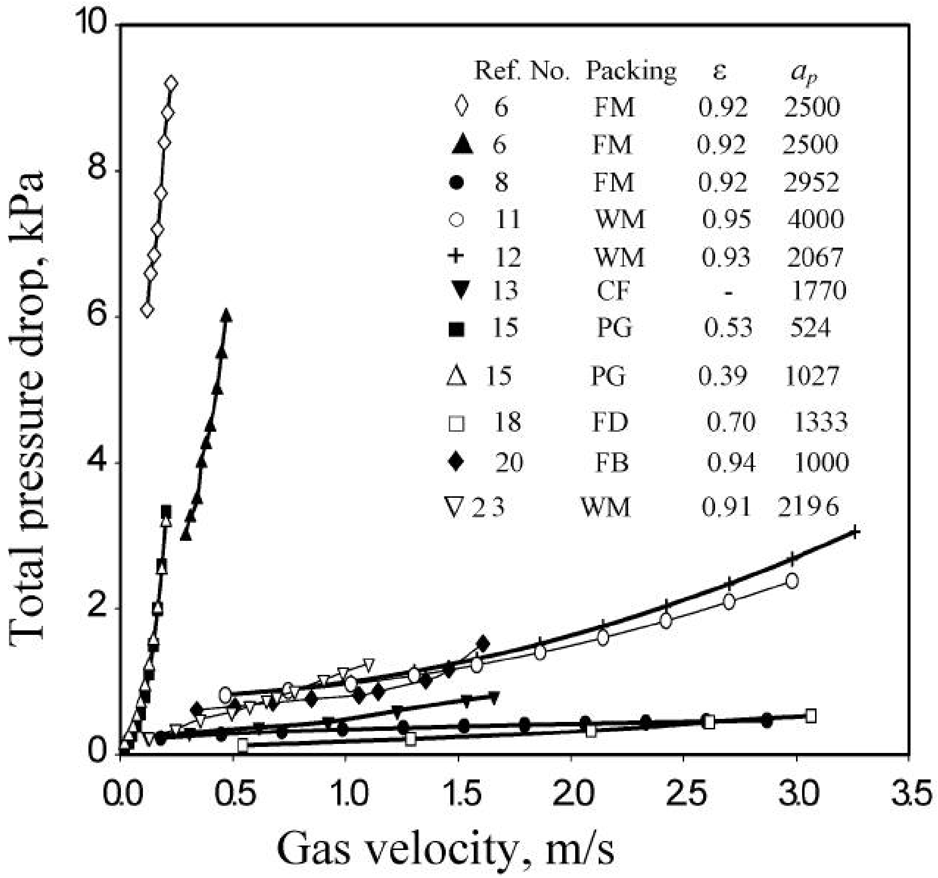


- important consideration when comparing the performance of the RPB with other mass transfer devices such as a packed tower
- pressure drop is proportional to the square of rotor speed

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

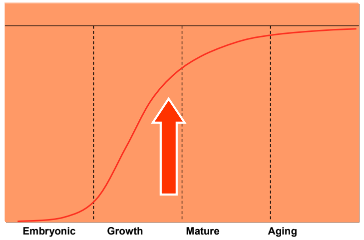
Pressure Drop





(source: D. P. Rao et al., Ind. Eng. Chem. Res., 43 (2004) 1150-1162)

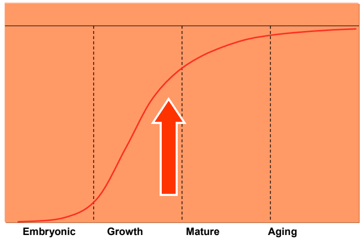
Heat transfer



- **problem of heat transfer** within a porous packed bed
- **heat input** can be achieved by use of **eddy currents, microwaves, or sonic energy,** thus operations such as evaporation, stripping, and endothermic reactions are in principle possible
- **heat removal** is more problematic
- exothermic reactions must be **conducted adiabatically** within the rotor
- **alternating packing and heat transfer plates** could be an option
- **evaporative cooling** is possible if the evaporation is compatible with the chemical process.

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

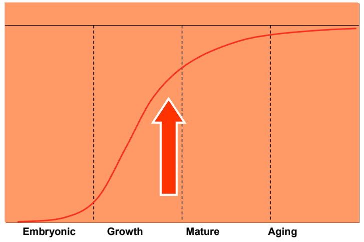
Mechanical design



- rotor type
- shaft orientation
- seals
- power train
- liquid distribution
- rotor packing
- single/multiple rotors

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

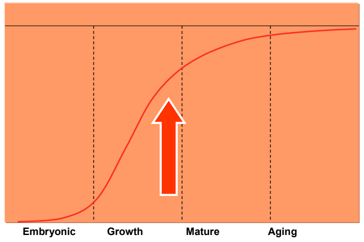
Rotor type



- **cantilever design** - rotor at the end of the shaft
- **centerhung design** - rotor in the middle of the shaft with bearings on either side of the rotor
- determining factor for selection is the **ratio of axial height (AH) to outside rotor diameter (OD)**
- limit **cantilever selection: AH/OD < 0.5-0.85**
- **centerhung design is more stable**, but has a higher cost of manufacture due to the split case housing, is more difficult to maintain, and requires two shaft seals instead of one

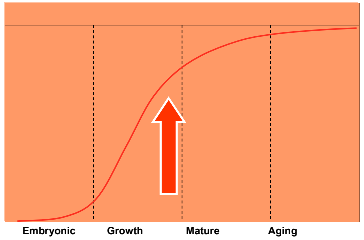
(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

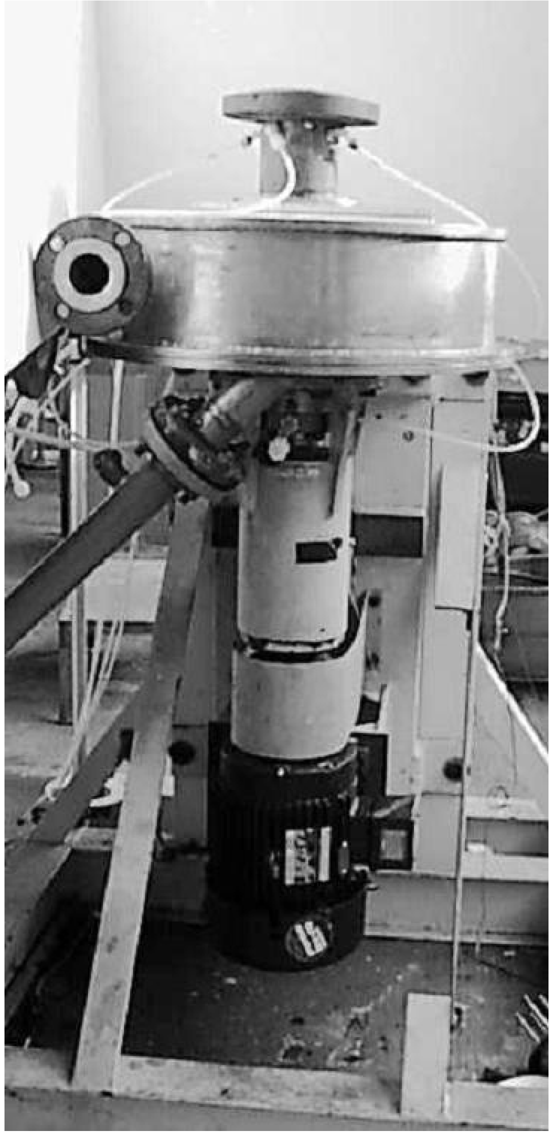
Power train



- vertical shaft cantilever - **belt drive** to reduce cost of manufacture of the support structure and to facilitate maintenance
- horizontal shaft - additional option of **direct coupling**.
- **variable speed** can be accomplished through a **gearbox** or preferably through **variable frequency control** on the motor

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

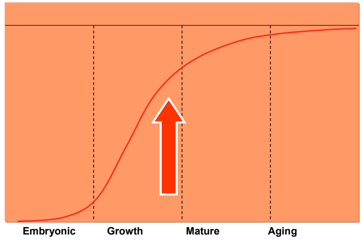




Pilot scale RPB illustrating the vertical shaft, cantilever design with direct motor drive

(photo courtesy of The Dow Chemical Company).

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

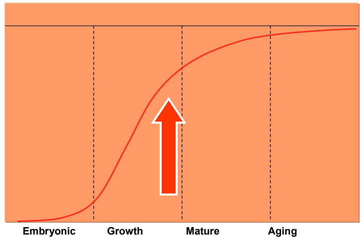


Pilot scale RPB illustrating the horizontal shaft, cantilever design with direct motor drive

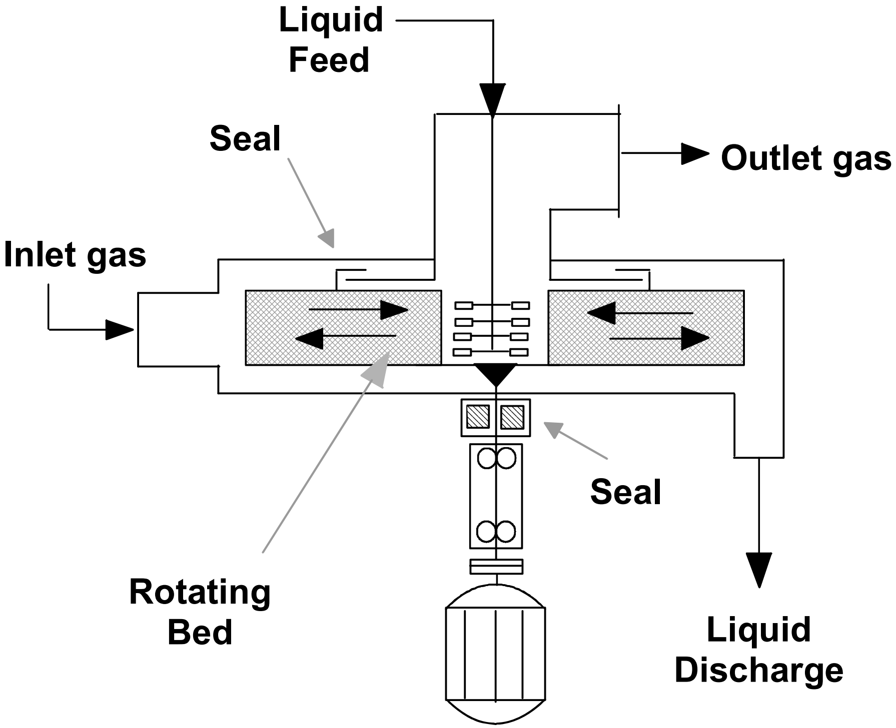
(photo courtesy of Higravitec Center of Beijing University of Chemical Technology).

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Seals



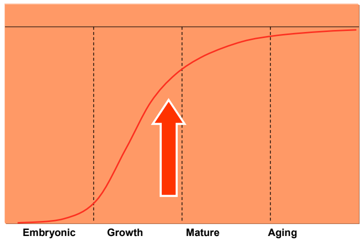
ROTATING PACKED BED
Gas Continuous Operation



- seals on the shaft: mechanical seals, lip seals, packing glands
- seals of the rotor (to prevent gas bypassing): labyrinth seals and liquid ring seals

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

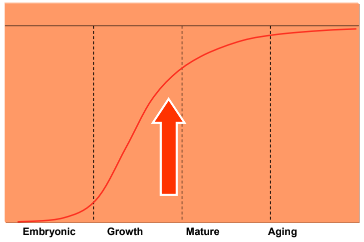
Liquid distribution



- proper liquid distribution is critical to performance, but is also important to prevent rotor imbalance.
- rotor imbalance from liquid maldistribution is especially a problem with high viscosity fluids.

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

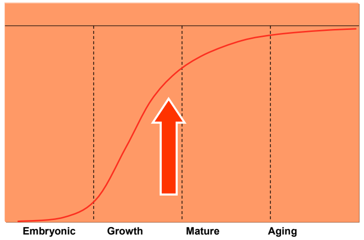
Rotor packing



- woven wire screen
- pellets randomly packed
- foam metal
- structured packing
- packing must have **physical properties sufficient to withstand the hydraulic forces** created by the accelerating liquid
- packing must be **dimensionally stable** during operation to avoid rotor imbalance issues.
- some packing materials may require **supports** to keep them in place

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

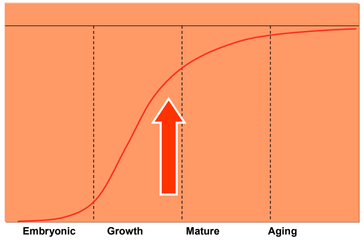
Multiple rotors



- to accommodate the need of additional transfer units in countercurrent gas/liquid contact
- to allow for heat transfer in addition to the mass transfer
- more complex in mechanical design and construction

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

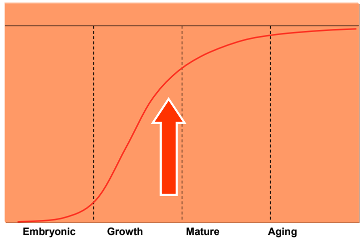
Applications



- absorption
- stripping
- reaction
- crystallization
- distillation

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

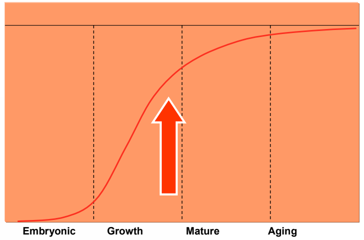
Absorption

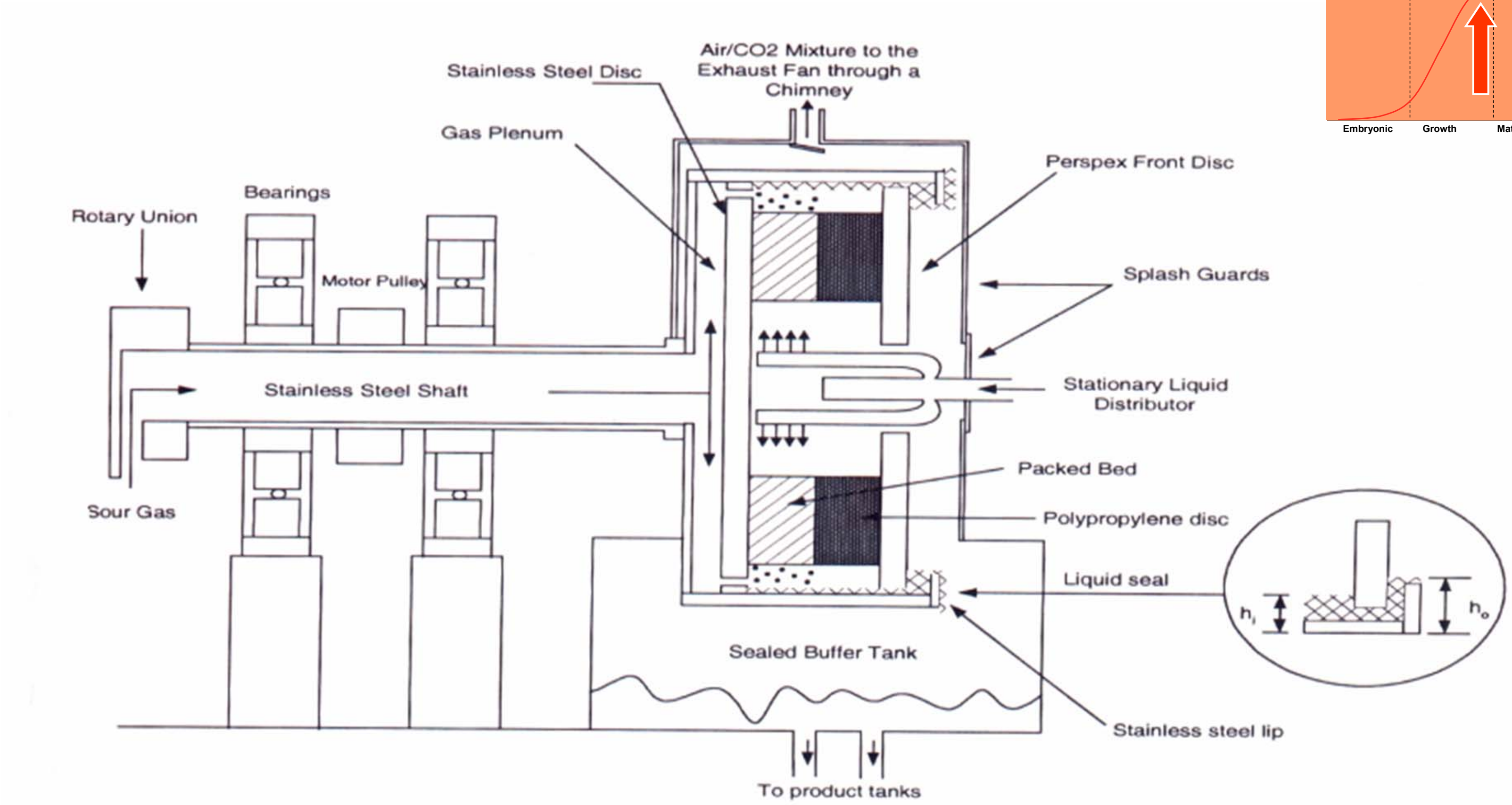


- CO_2 Sequestration in monoethanolamine solutions using Higee Technology
- The absorbent used - monoethanolamine/water solution (30 – 100% w/w MEA)

C. Ramshaw, Process Intensification: What? Why? How?

Absorption





Cross sectional view of the HiGee rig

Bed OD 40 cm

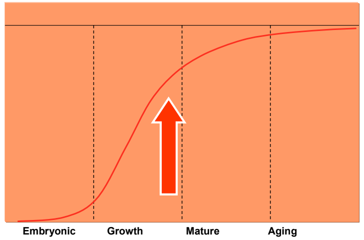
Thickness 2.5 cm

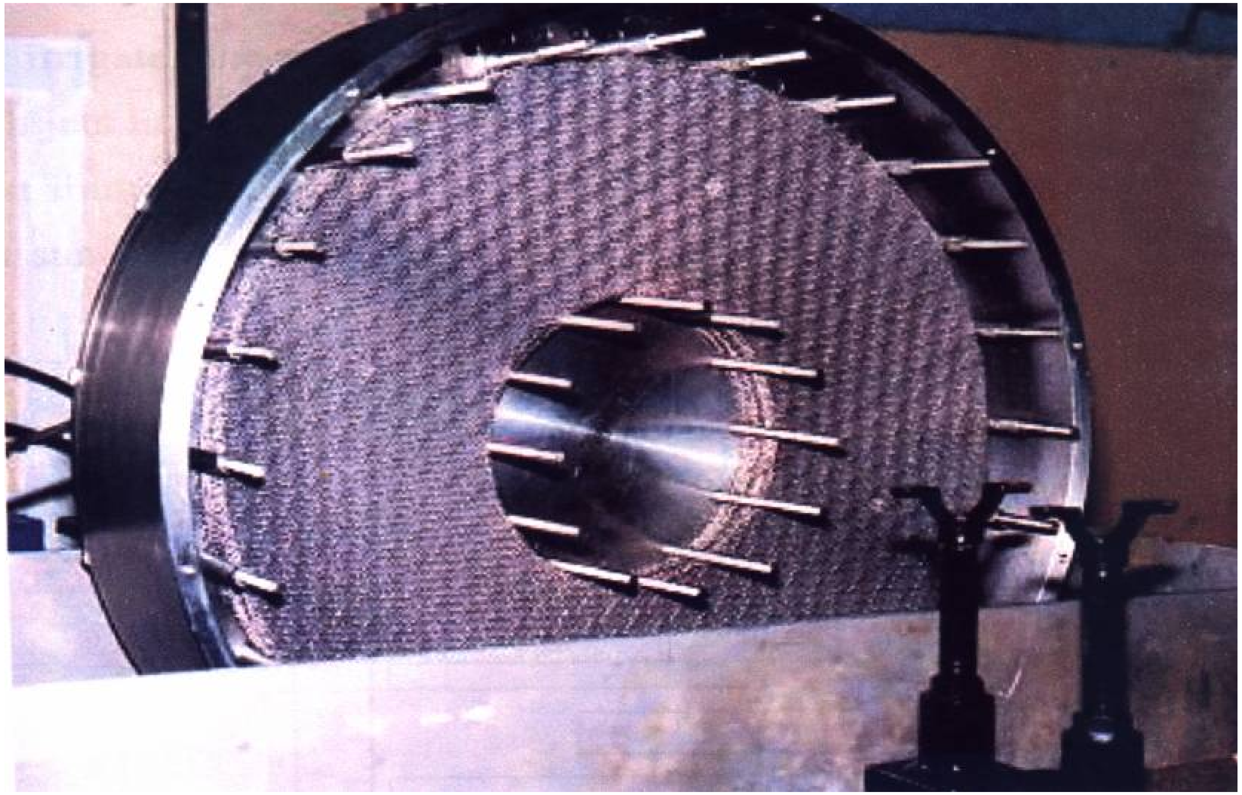
Bed ID 16 cm

RPM up to 1000

C. Ramshaw, Process Intensification: What? Why? How?

Absorption

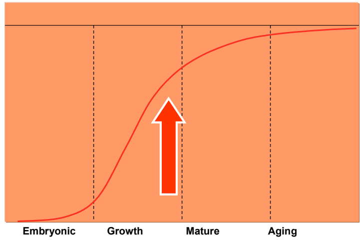


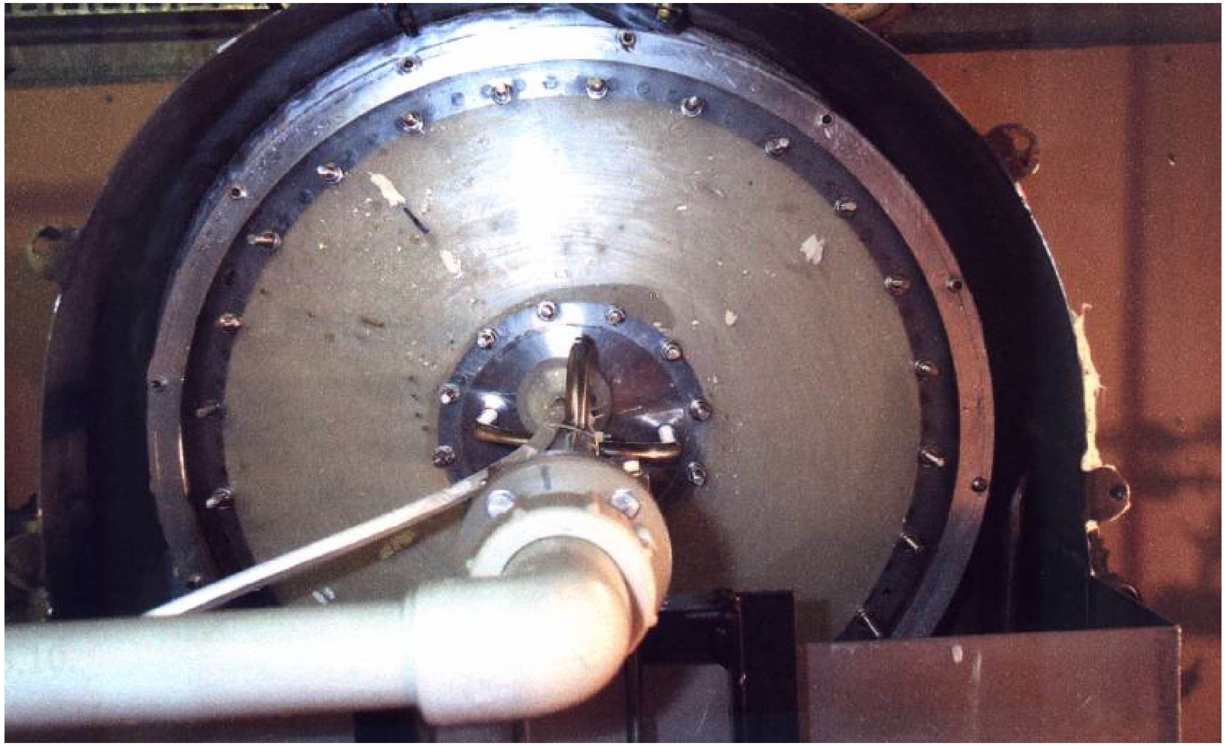


Mesh installed in the rotor

C. Ramshaw, Process Intensification: What? Why? How?

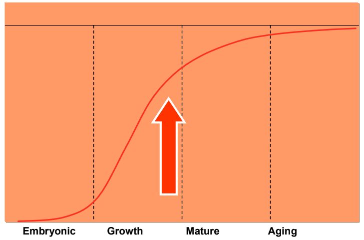
Absorption





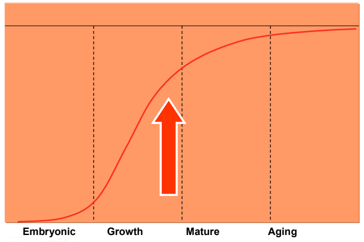
Position of the 4-arm distributor in the “eye” of the rotor

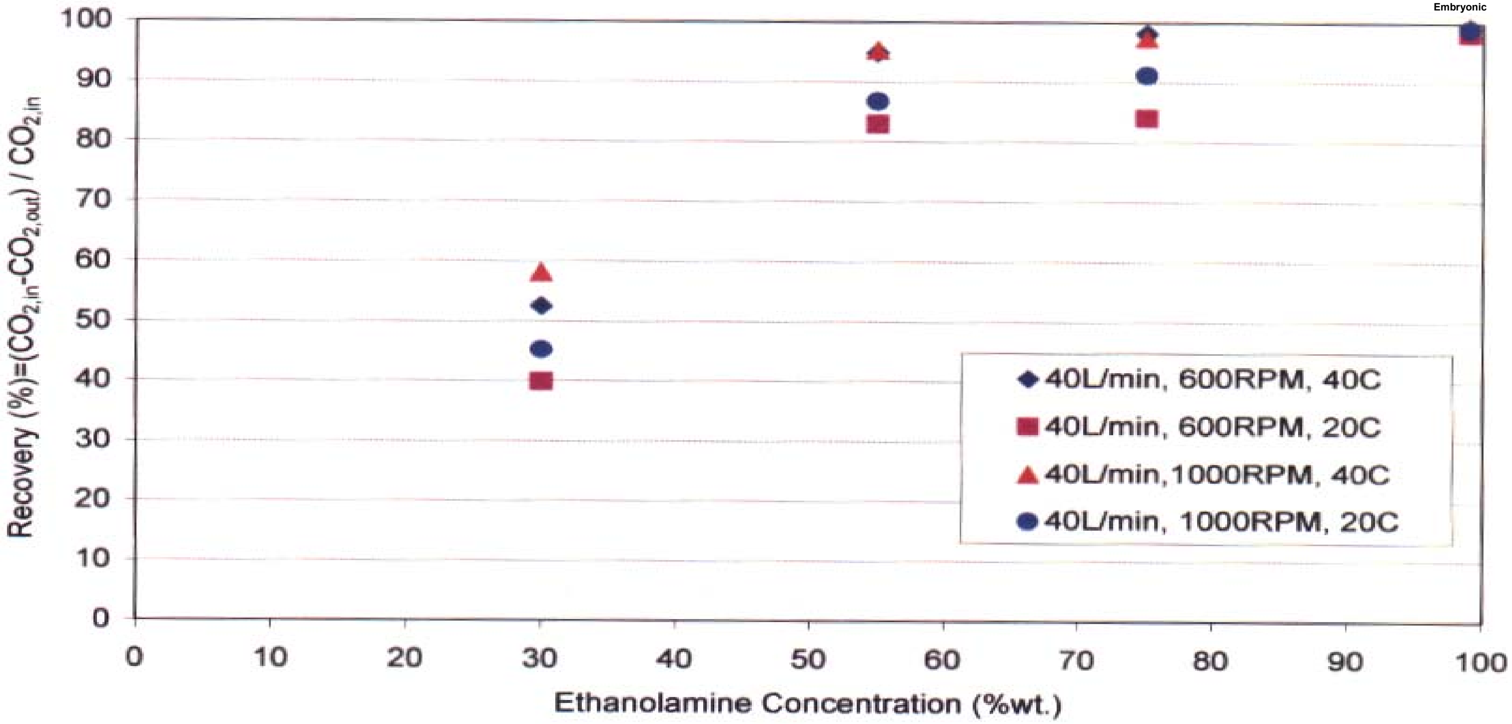
Absorption



A 4-arm liquid distributor

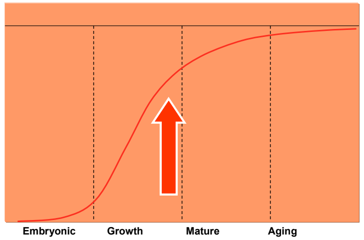
Absorption





Variation of % recovery with liquid concentration at constant temperature, rotor speed and liquid flowrate at 40 l/min

Absorption



Higee comparison with a static column

Duty Gas flow 72×10^6 ft^3/day (24 m^3/s)

Liquid flow 2000 gpm (121 kg/s)

Kohl & Nielsen Column Height 40 m (14 m packed)

Diameter 4.4 m

CO_2 in/CO_2 out ~ 60

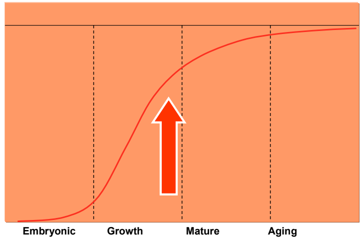
Higee Equivalent (2 baskets)

OD 1 m; ID 0.5 m; Axial length 1.15 m

RPM 1000; Packed depth 0.25 m

NTU ~ 5 CO2 in/CO2 out ~ 150

Stripping - commercial applications



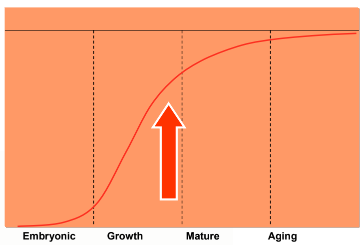
Commercial water deaeration RPB using the horizontal shaft, centerhung design and direct motor drive

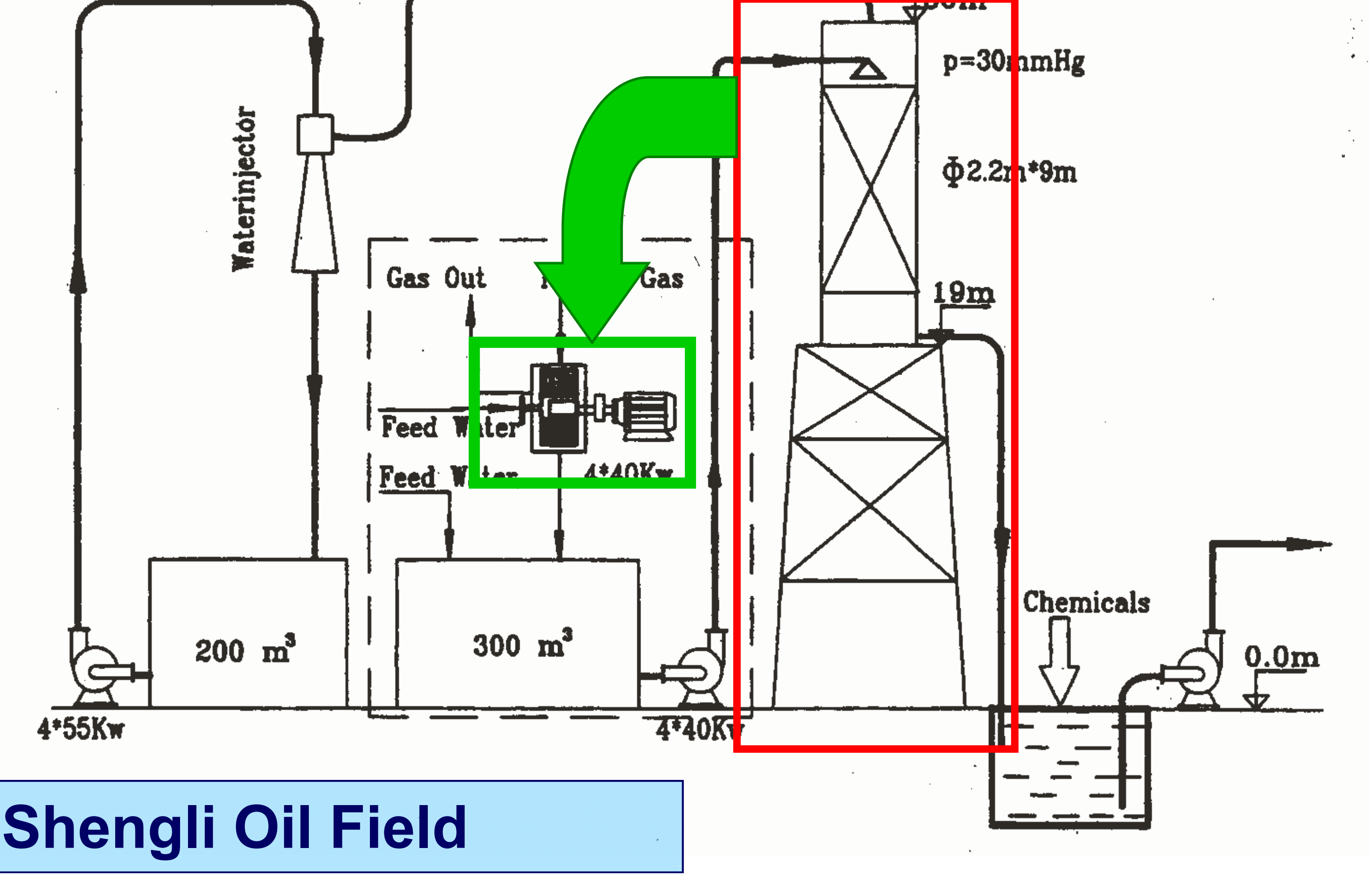
(photo courtesy of Higravitec Center of Beijing University of Chemical Technology)

- water deaeration in oil fields in China
- full-scale commercial RPB to process 300 T/h.
- unit has rotor dimensions of 600 mm ID, 1000 mm OD, and 700 mm AH; the wire screen packing has high porosity (92%) and low surface area (500 m2/m3); the rotor spins at a 750 rpm
- two 250 T/h units have been designed for installation on oil platforms

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Stripping - commercial applications

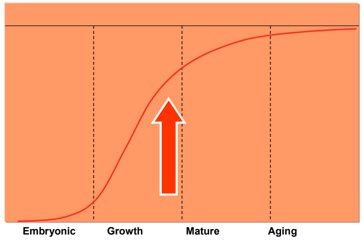




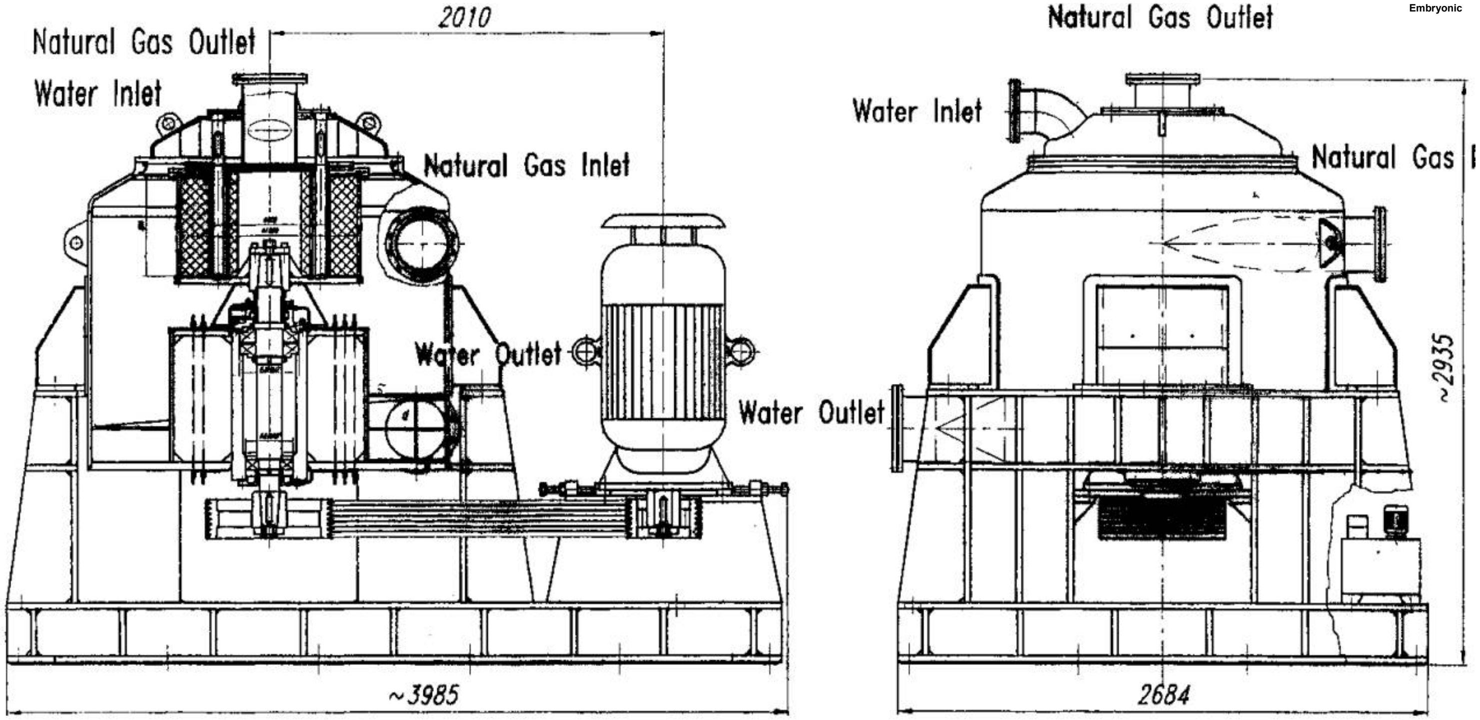
Shengli Oil Field

(source: Z. Zheng et al., 1998)

Stripping - commercial applications



250 T/h unit for installation off-shore

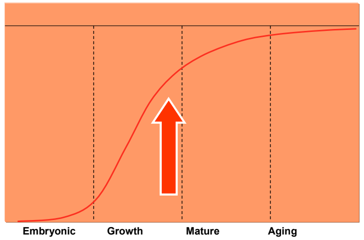


I.D. of RPB: 600mm
O.D. of RPB: 900mm
A.L. of RPB: 500mm
Rotating Speed: 860rpm.

Packing: Wire Reticulated, Ti.
Voidage: 92%
Specific Area: $500 m^2/m^3$

(source: Z. Zheng et al., 1998)

Stripping - commercial applications

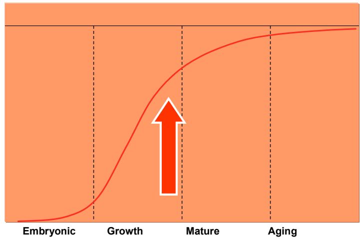


Two 250 T/h units have been designed for installation off-shore

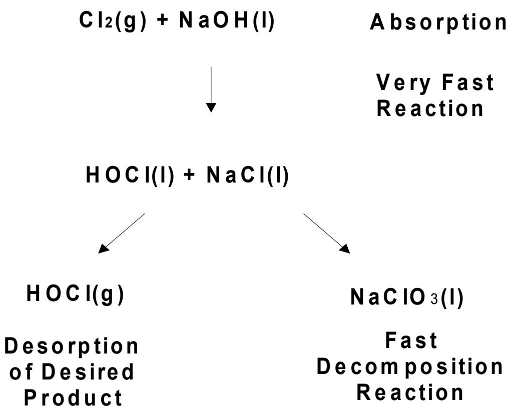
	Vacuum tower (one set 10,000 t/d)	**Higrav Deaerator (two sets 6,000 t/d)**
Platform area [m^2]	30	2x10
Height [m]	14	3
Weight (t)	60 (dry) 130 (operation) 180 (full of water)	2x10 2x10.5 2x11
Residual oxygen [ppm]	1 (summer) 2-3 (winter)	< 0.05 < 0.05
Investment	1	0.8
Power [kW]	155	2x160

(source: Z. Zheng et al., 1998)

Reactions - commercial applications



HOCl synthesis – conventional technology



$Cl_2(g) + NaOH(l)$ **Absorption**

Very Fast Reaction

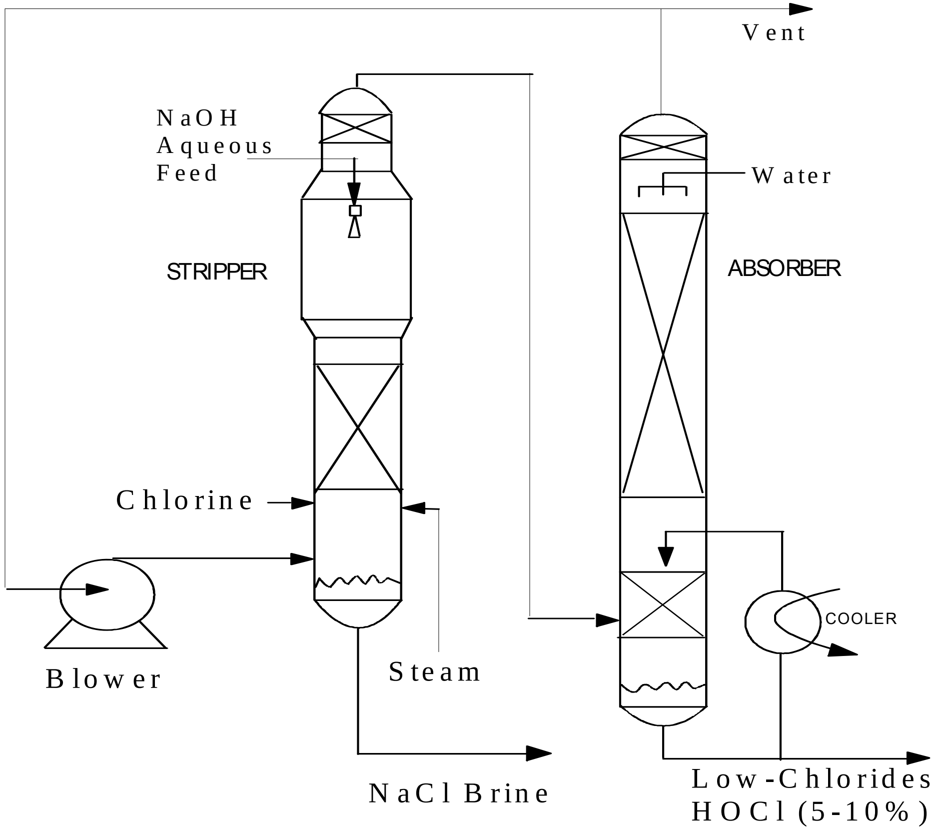
$HOCl(l) + NaCl(l)$

$HOCl(g)$

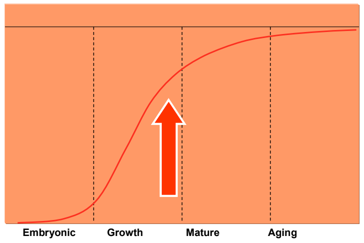
Desorption of Desired Product

$NaClO_3(l)$

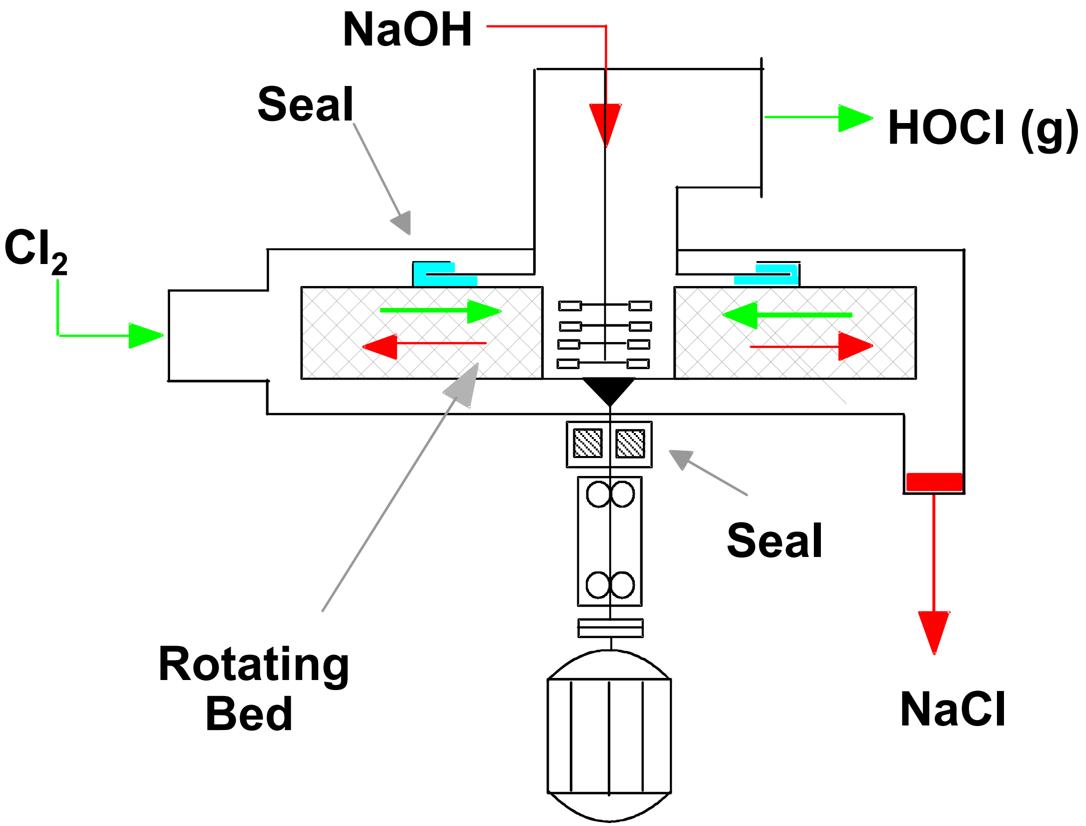
Fast Decomposition Reaction



Reactions - commercial applications

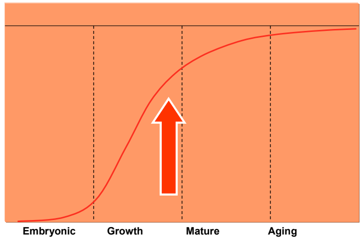


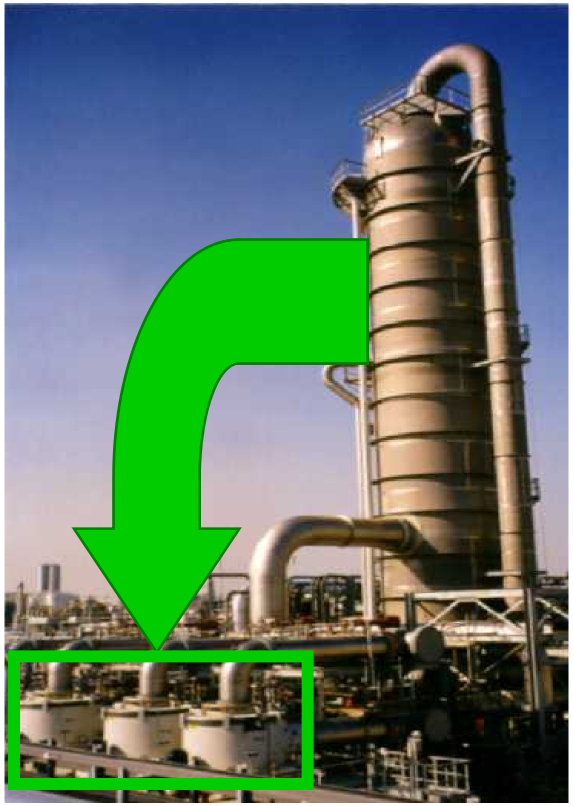
HOCl synthesis - High-Gravity Field technology



RPM < 400
90 g's @ ID

Reactions - commercial applications





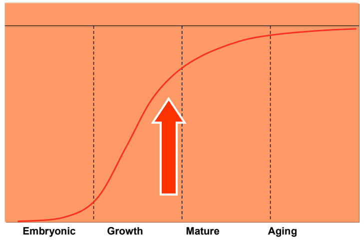
the three RPBs shown in the lower left of the picture process the same volume of gas and liquid as the tall absorber tower to the right!

HOCl synthesis - High-Gravity Field technology

- No mechanical issues
- Very easy to startup and shutdown
- Yield = 94-96% (80% conventional)
- RPBs < 40 x smaller
- 1/3 reduction in waste water & chlorinated byproducts

(D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Crystallization



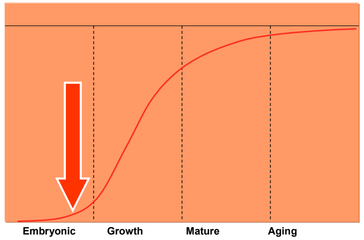
- reactive precipitation: $CO_2 + Ca(OH)_2$ slurry → $CaCO_3$
  - ✓ absorption of CO_2 is the rate limiting step;
  - ✓ narrow size distribution (15-30 nm);
  - ✓ reaction time reduced 4-10 fold compared to stirred tank reactors

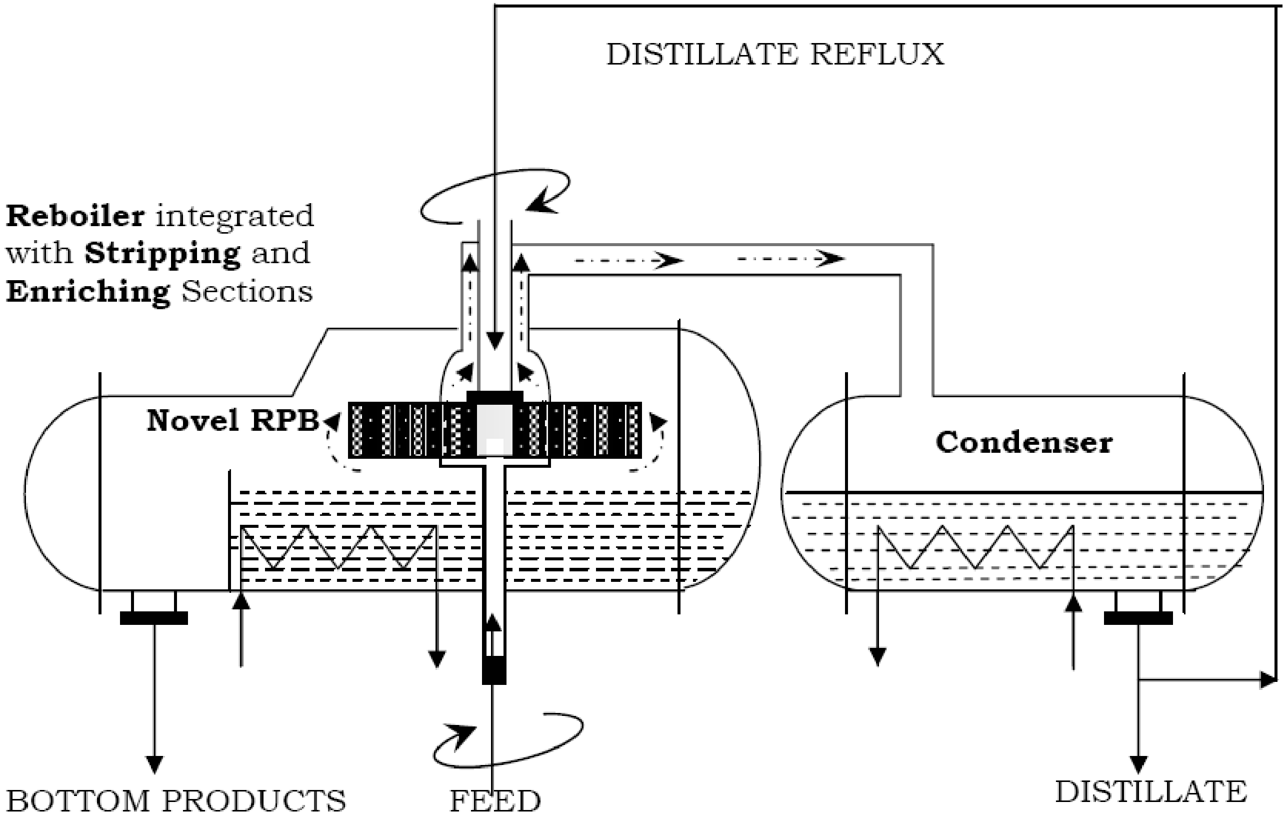
- reactive precipitation: $CO_2 + H_2O + NaAlO_2 \rightarrow Al(OH)_3$
  - ✓ $Al(OH)_3$ fibrils 1-10 nm in diameter and lengths of 50-300 nm;

- reactive precipitation: $NaCO_3 + Sr(NO_3)_2 \rightarrow SrCO_3$
  - ✓ $SrCO_3$ particles of 40 nm mean diameter and narrow size distribution

(source: D. Trent, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

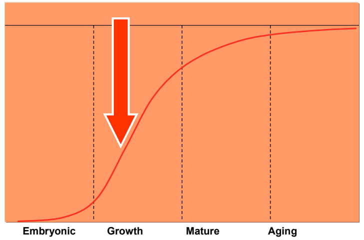
Distillation

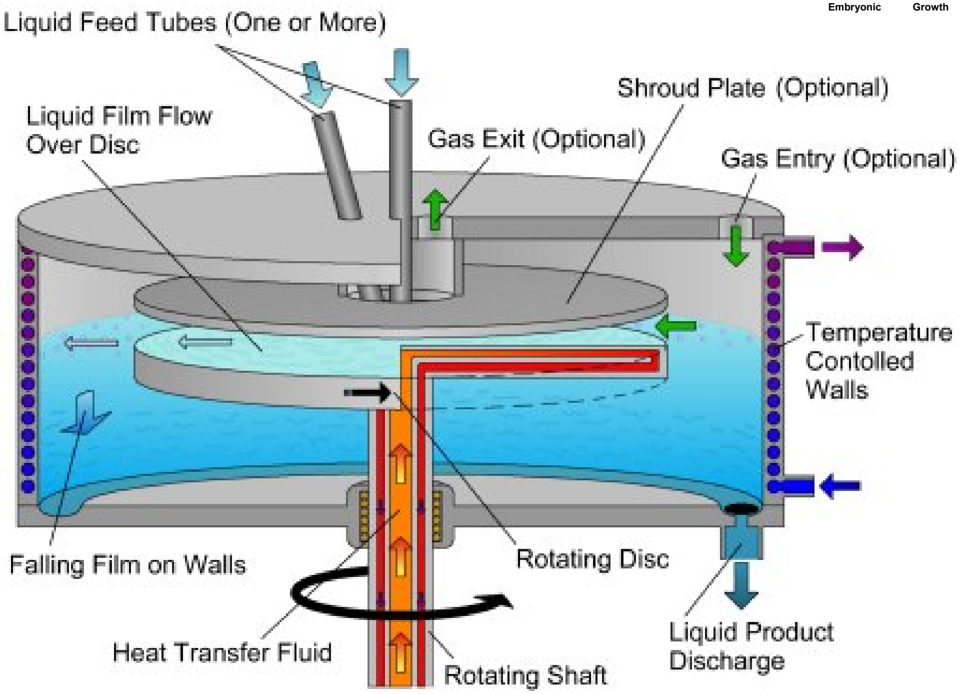


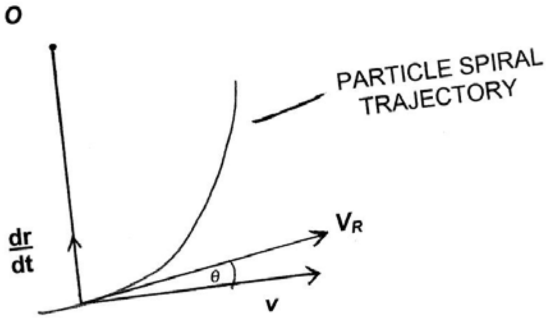


(Sivakumar et al.)

SDR – free motion of particle around axis







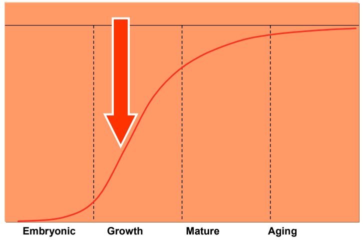
Angular momentum conservation:

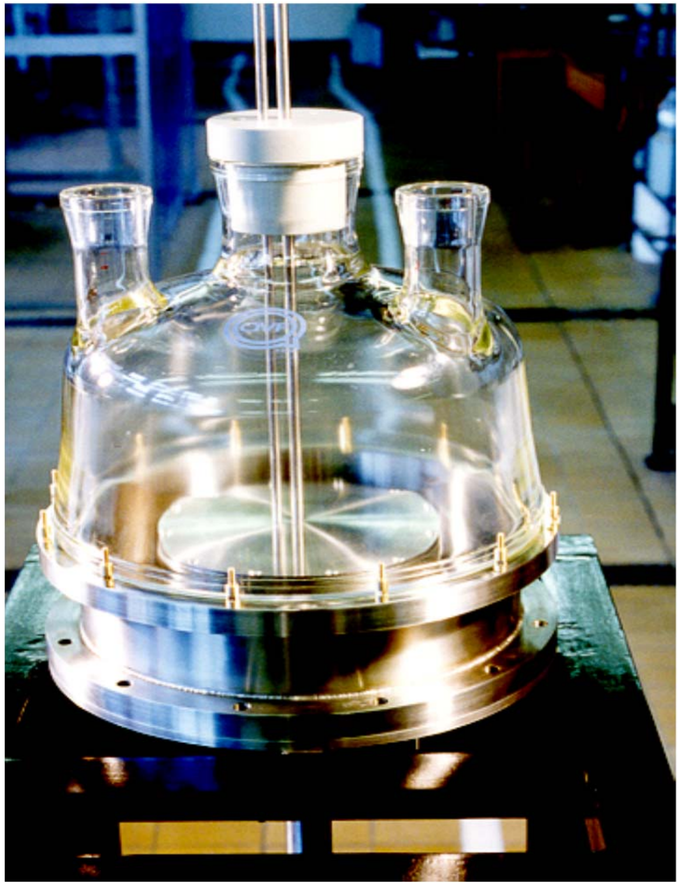
$$v_2 r_2 = v_1 r_1$$

velocity increases for an inward trajectory

(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Spinning Disc Reactor

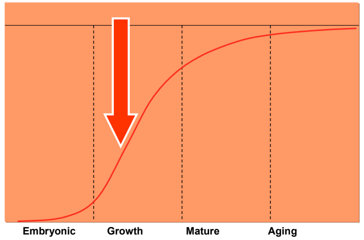


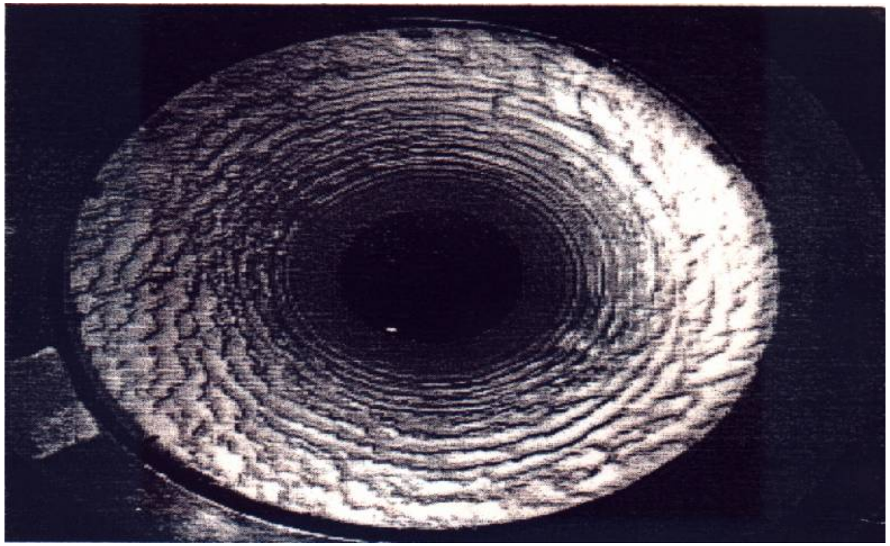


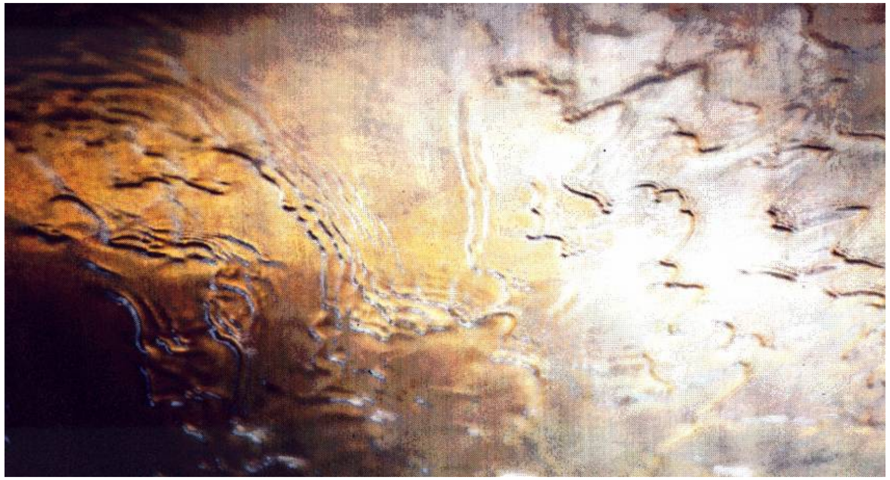
- particularly effective when high heat fluxes or viscous liquids are involved.
- the objective is to generate a highly sheared liquid film when a liquid is supplied to the unit at or near its centre.
- liquid film flow over a surface is **intrinsically unstable**
- the smooth inner film always broke down into an array of **spiral ripples**

(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Spinning Disk Reactor



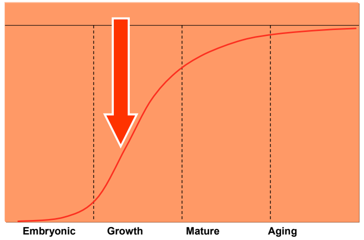


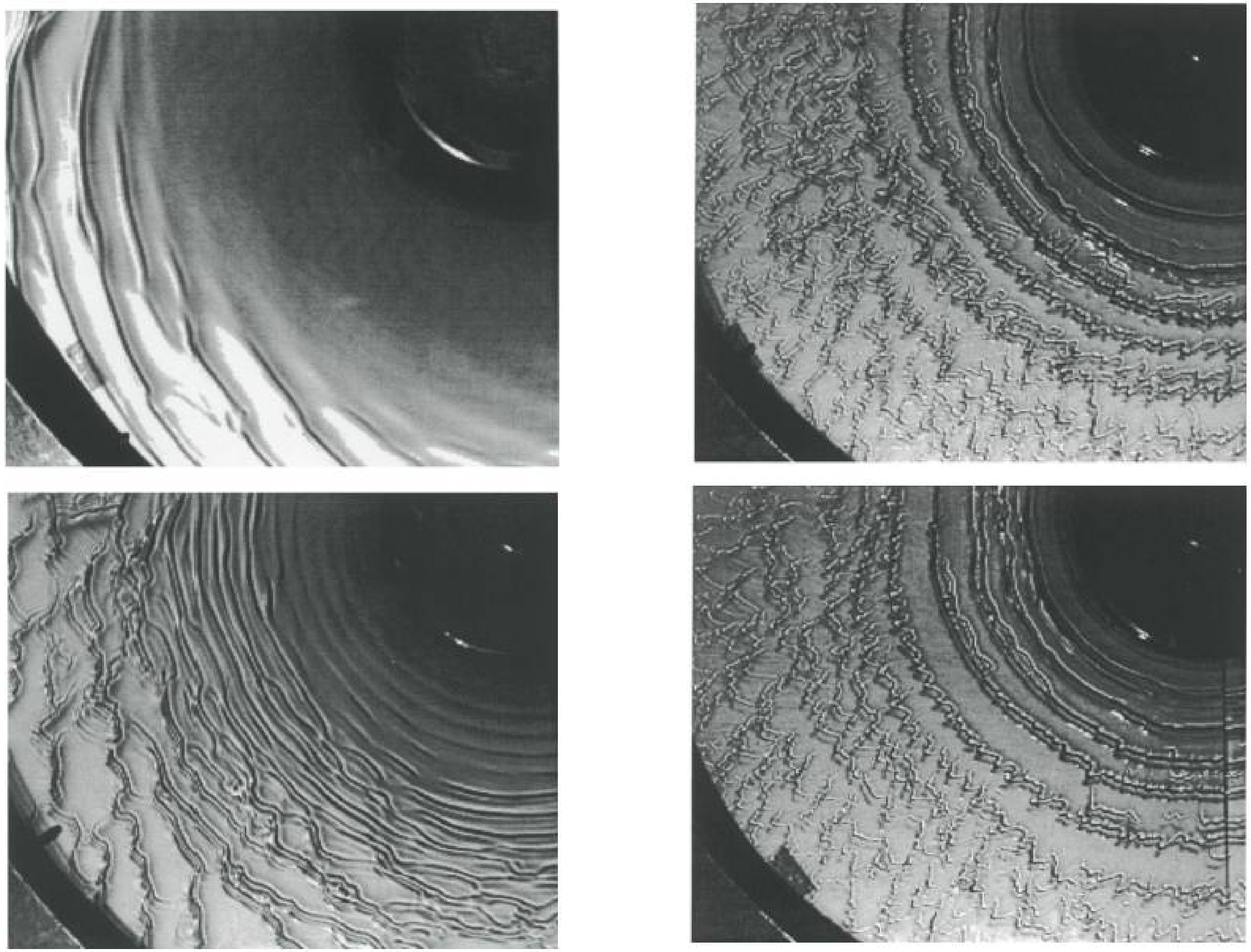


- improvement in mass/heat transfer performance generated by the waves is due to the additional shear which they induce
- even in the absence of ripples, highly sheared thin liquid films provide an ideal fluid dynamic environment, due to the short diffusion path length between the adjacent gas phase to the liquid film and then to the disc surface
- spinning disc is ideal for performing any intrinsically rapid transformation in a liquid, even if it is viscous

(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Spinning Disk Reactor

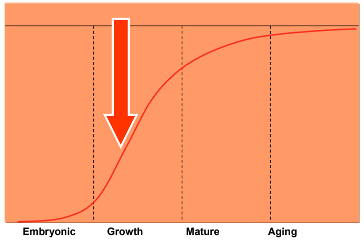


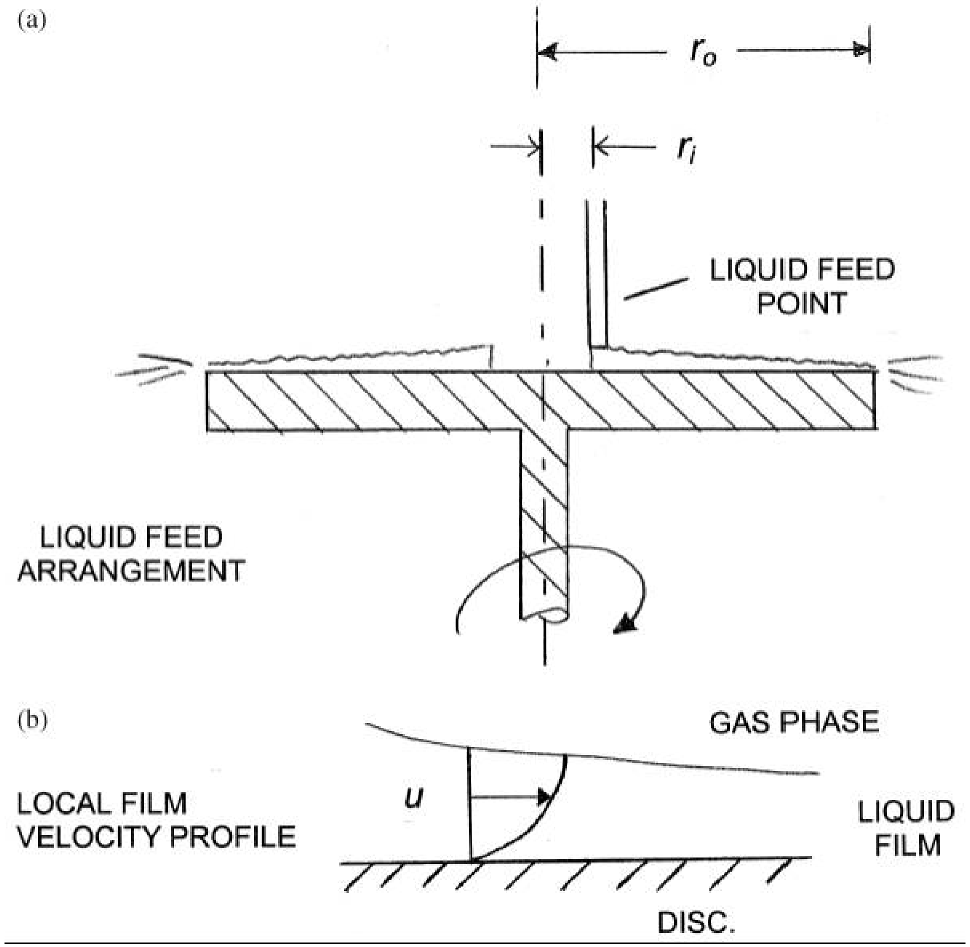


Liquid behaviour on spinning disk at flow of 19 cm^3/s and rotational speeds of 100, 200, 500 and 600 rpm

(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

Spinning Disk Reactor - hydrodynamics





Assumptions

- **Stable flow (ripple-free flow)**
- **No circumferential slip at the disc/liquid surface**
- **No shear at the gas/liquid interface**

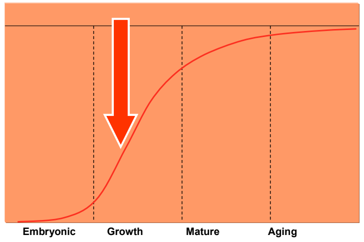
Average residence time between r_i and r_0

$$t=\int_{R_i}^{R_0}\frac{dr}{U_0}=\frac{3}{4}\left(\frac{12\pi^2\rho\mu}{M^2\omega^2}\right)^{1/3}\left(r_o^{4/3}-r_i^{4/3}\right)$$

Residence time: usually 1-5 sec

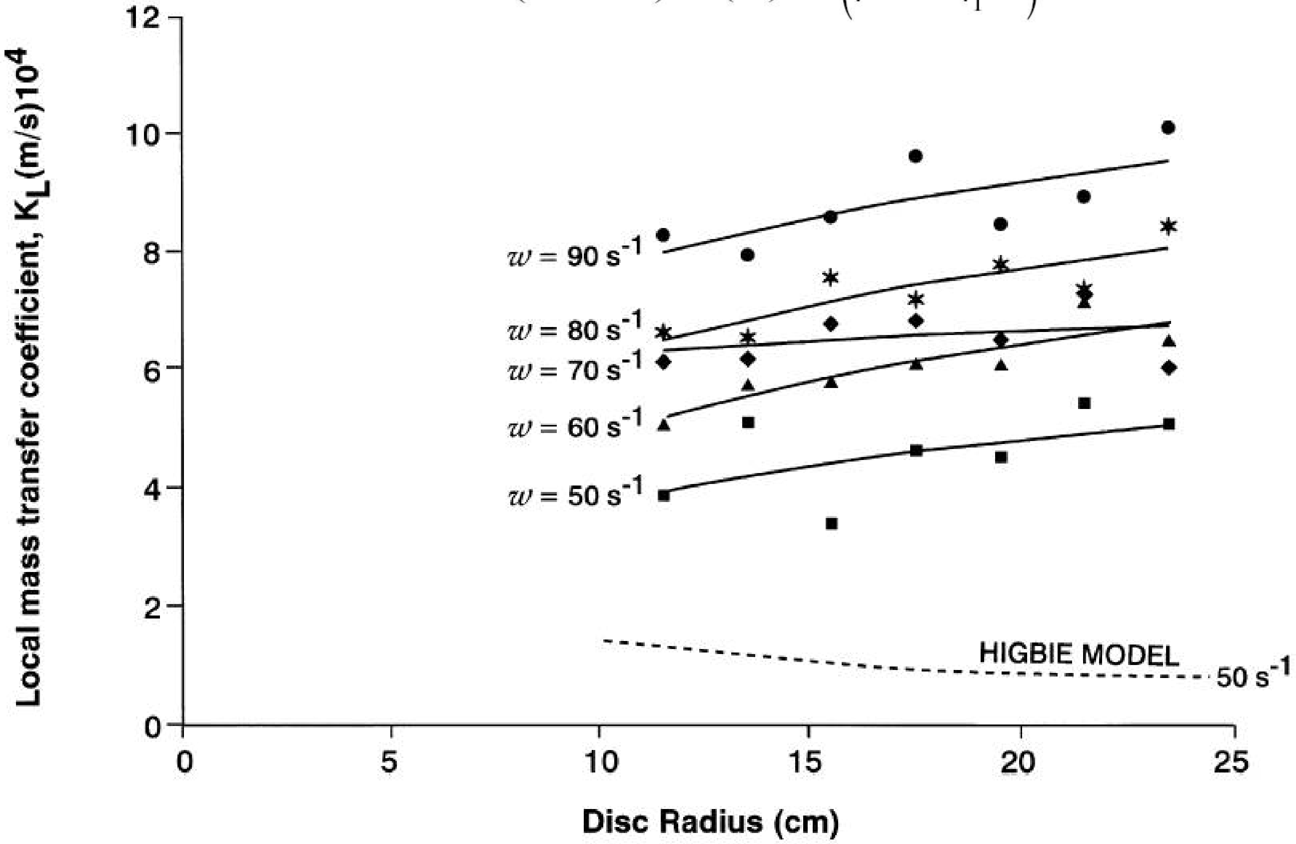
(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

SDR – mass transfer



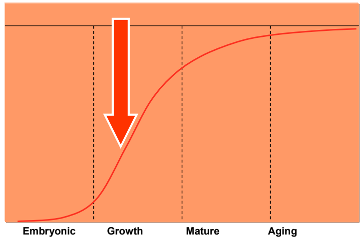
Conservative estimate of local k_L based on Higbie's theory:

$$k_L = \left(\frac{D}{\pi}\right)^{1/2} \left(\frac{2M^2\omega^2}{3\pi^2\rho\mu}\right)^{1/6} \left(\frac{r}{r_1}\right)^{2/3} \frac{1}{\left(r^{4/3} - r_1^{4/3}\right)^{1/2}}$$



(source: A.Anoue and C. Ramshaw, 1999, Int. J. Heat & Mass Transfer, 42 (1999) 2543-2536)

SDR – heat transfer

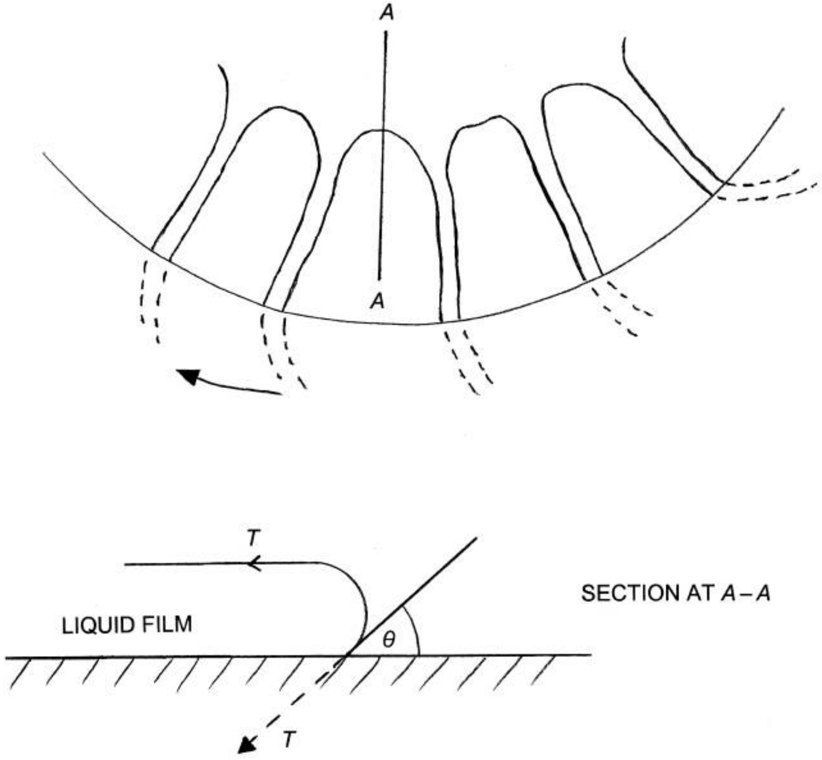


Effective film heat transfer coefficient:

$$h = \frac{Q}{T_w - T_s} = \frac{2k}{s}$$

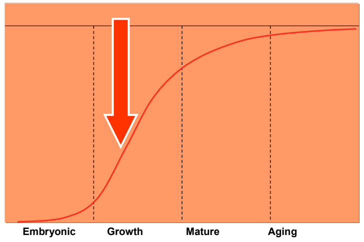
for 0.5 m disc with 28 micron film, heat transfer at the periphery: 43 kW/m^{2}K !

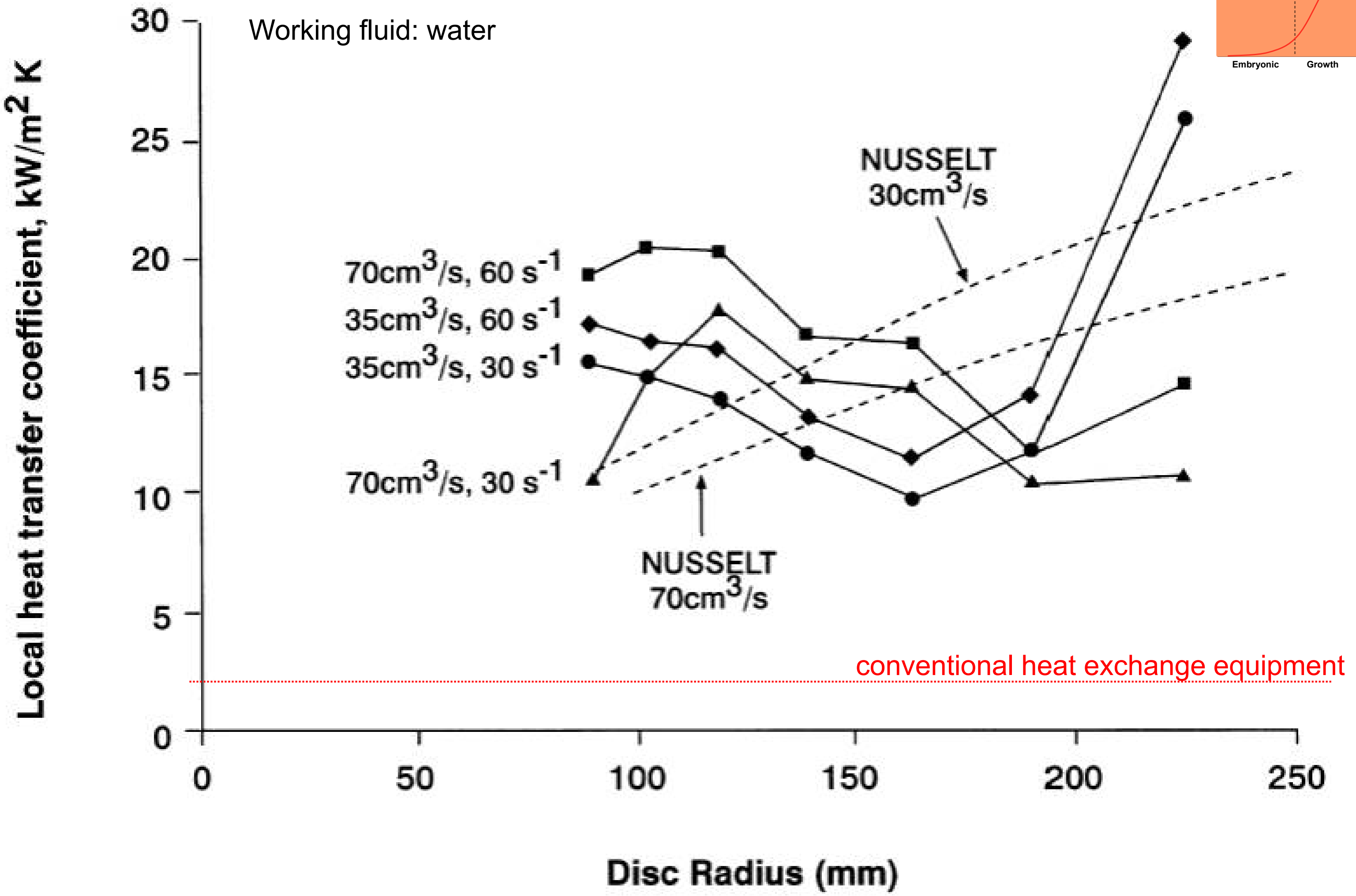
Condition: film must be continuous, no rivulets can be formed



(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

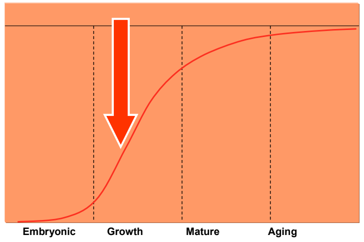
SDR – heat transfer



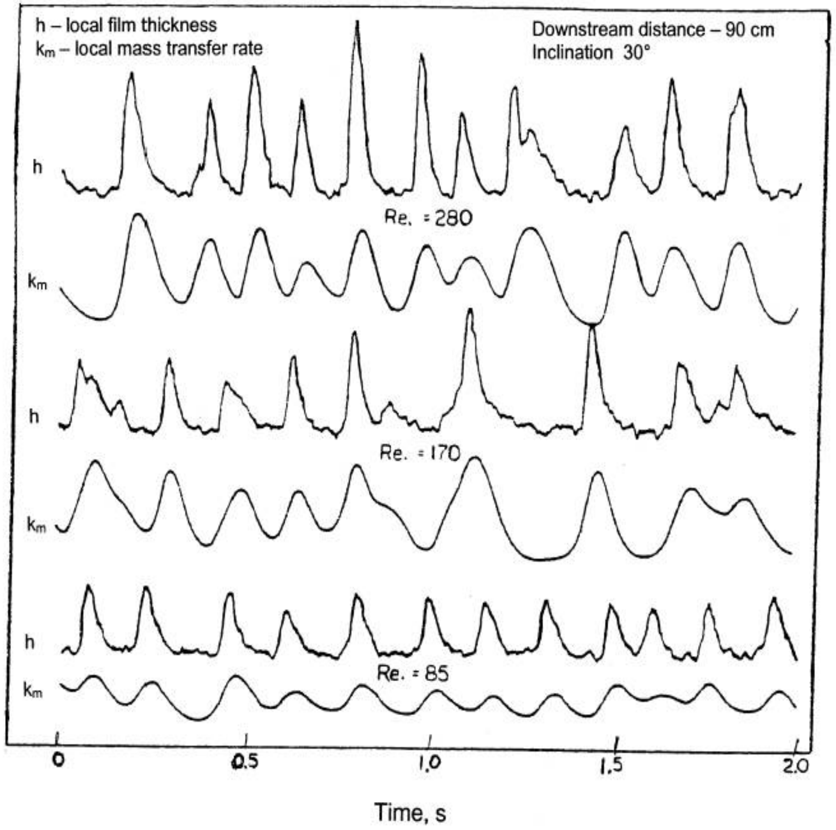


(source: A.Anoue and C. Ramshaw, 1999, Int. J. Heat & Mass Transfer, 42 (1999) 2543-2536)

SDR – film flow stability

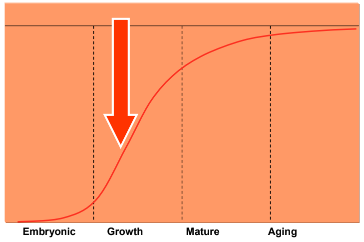


- instantaneous local film thickness of a liquid flowing down a stationary inclined plane measured using a capacitance technique.
- simultaneously the local mass transfer coefficient measured between the disc and the liquid, using the limiting electrolytic current method
- passage of a ripple was associated with a significant enhancement of the mass transfer coefficient, as a consequence of the flow field associated with ripple propagation
- mass transfer performance can be enhanced further by engineering the disc surface profile.



(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

SDR – reaction applications

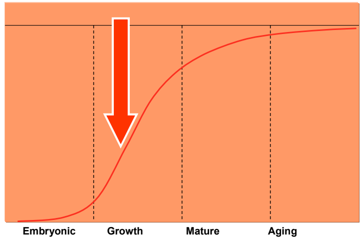


Processing of viscous liquids:

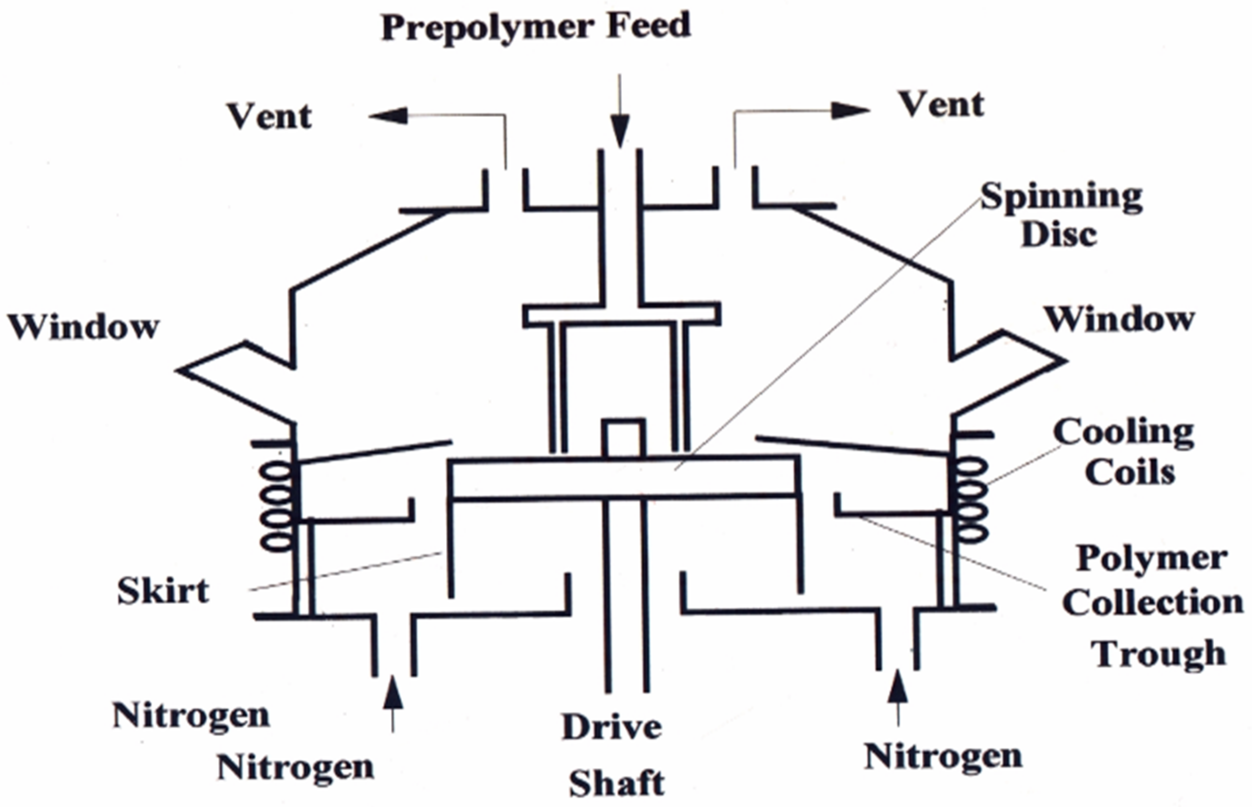
- **Condensation reactions**
- **Radical reactions**
- **Devolatilisation**

(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

SDR – reaction applications

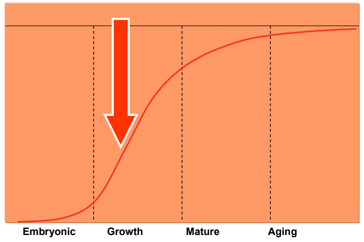


Styrene polymerization

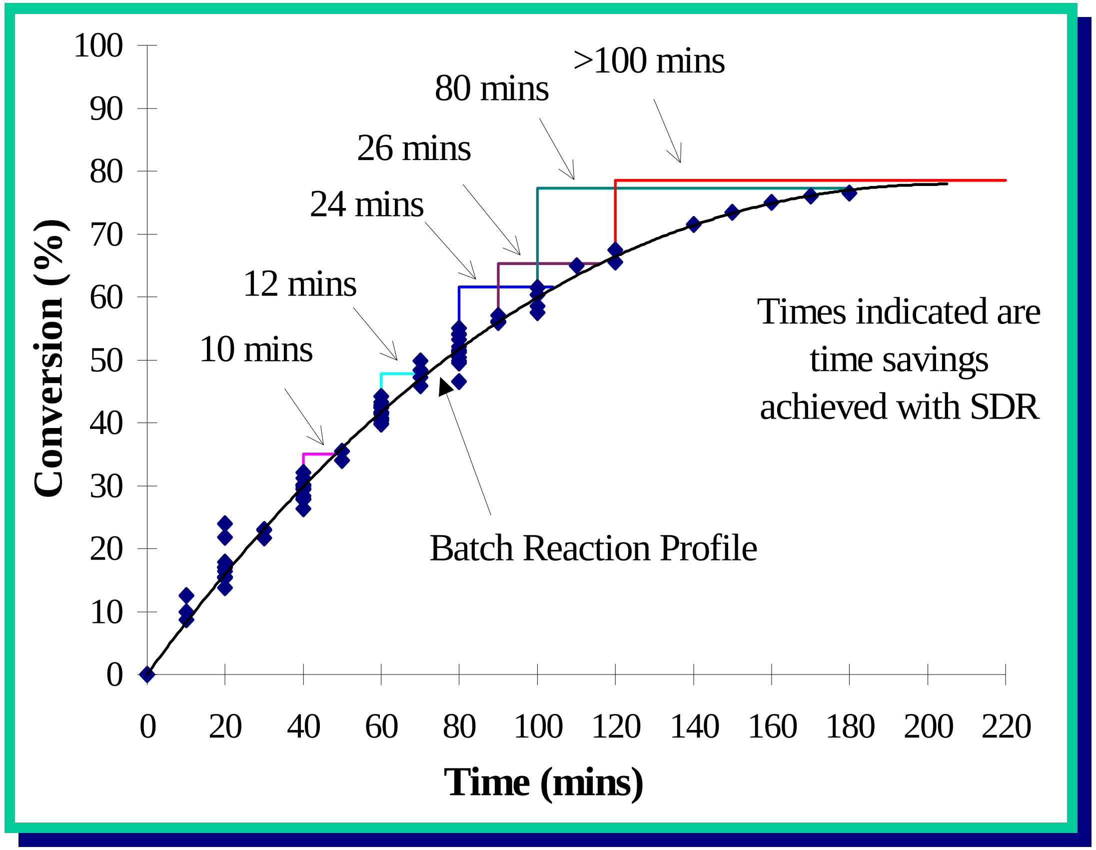


(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

SDR – reaction applications



Styrene polymerization

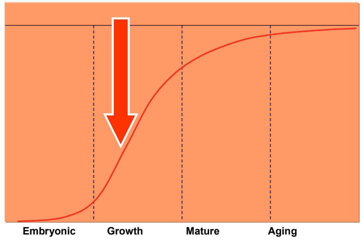


Times indicated are time savings achieved with SDR

Time saving in SDR operated at 850 rpm

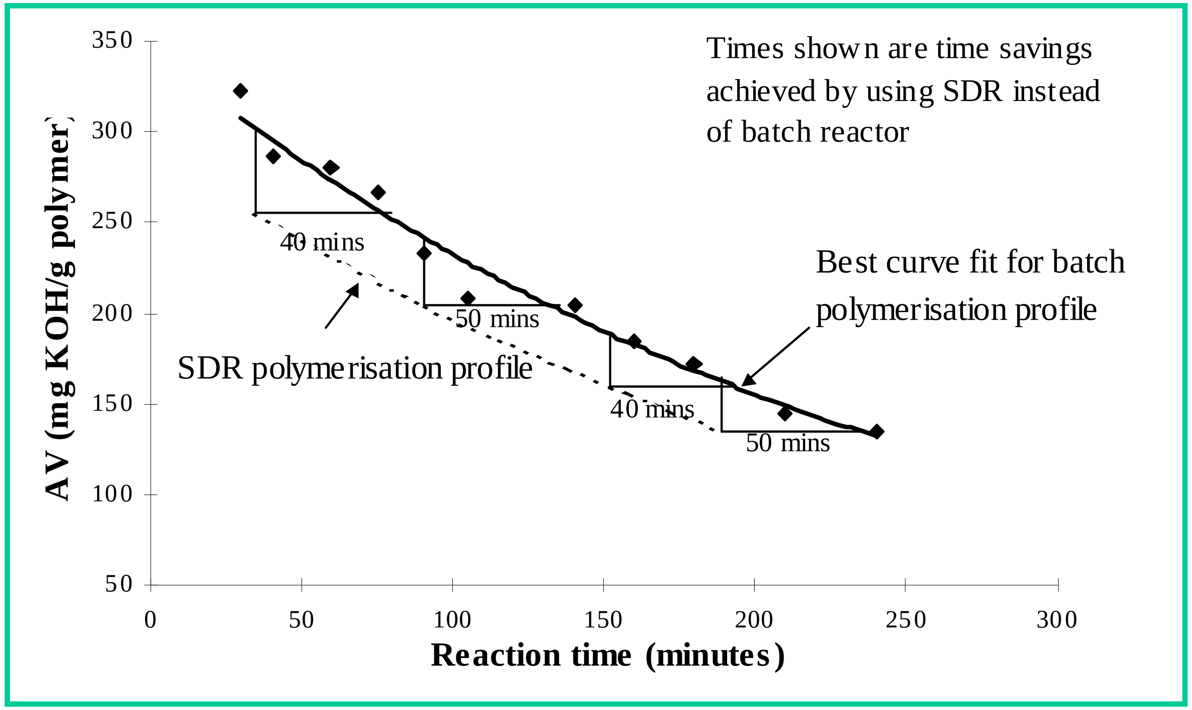
(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

SDR – reaction applications



Polycondensation

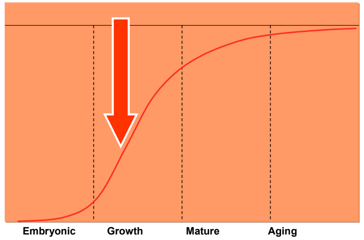
- polyesterification reaction between maleic anhydride and ethylene glycol
- water produced must be eliminated from the increasingly viscous polymer melt (to shift equilibrium)
- grooved brass 36 cm disc described above at a temperature of 200°C and a disc speed of 1000 RPM
- as the acid number decreases the conversion to polymer increases



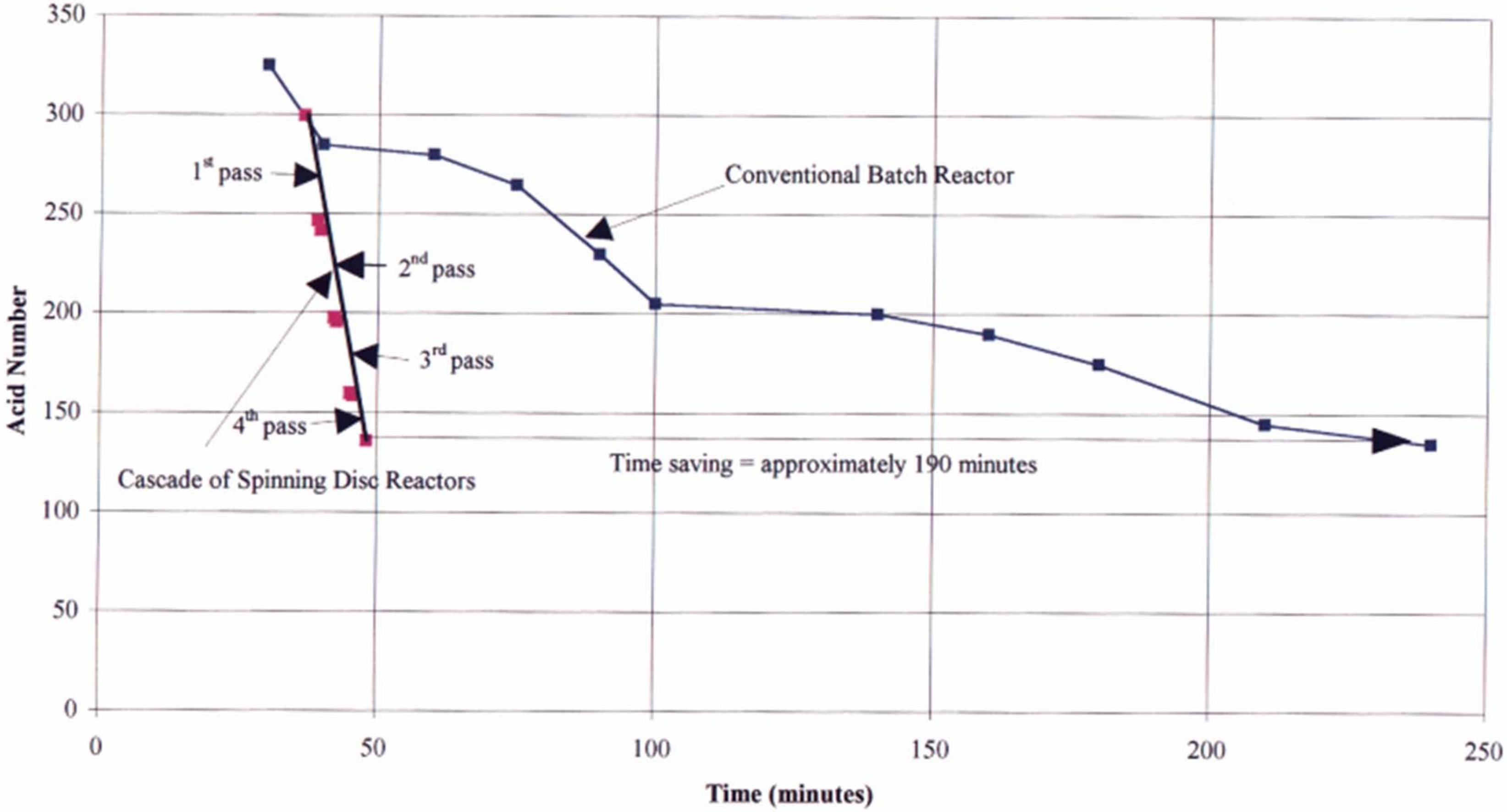
Times shown are time savings achieved by using SDR instead of batch reactor

(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

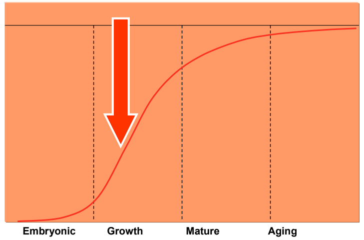
SDR – reaction applications



Performance of the SDR for the polymerisation of the unsaturated polyester



SDR – reaction applications



SmithKline Beecham process:

Fine chemical processes

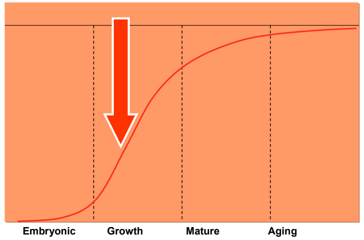
phase-transfer-catalyzed Darzen's reaction to produce a drug intermediate

Effects **(with respect to batch process):**

- **99.9% reduction of reaction time;**
- **99% reduction of inventory;**
- **93% reduction of impurity level;**

(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

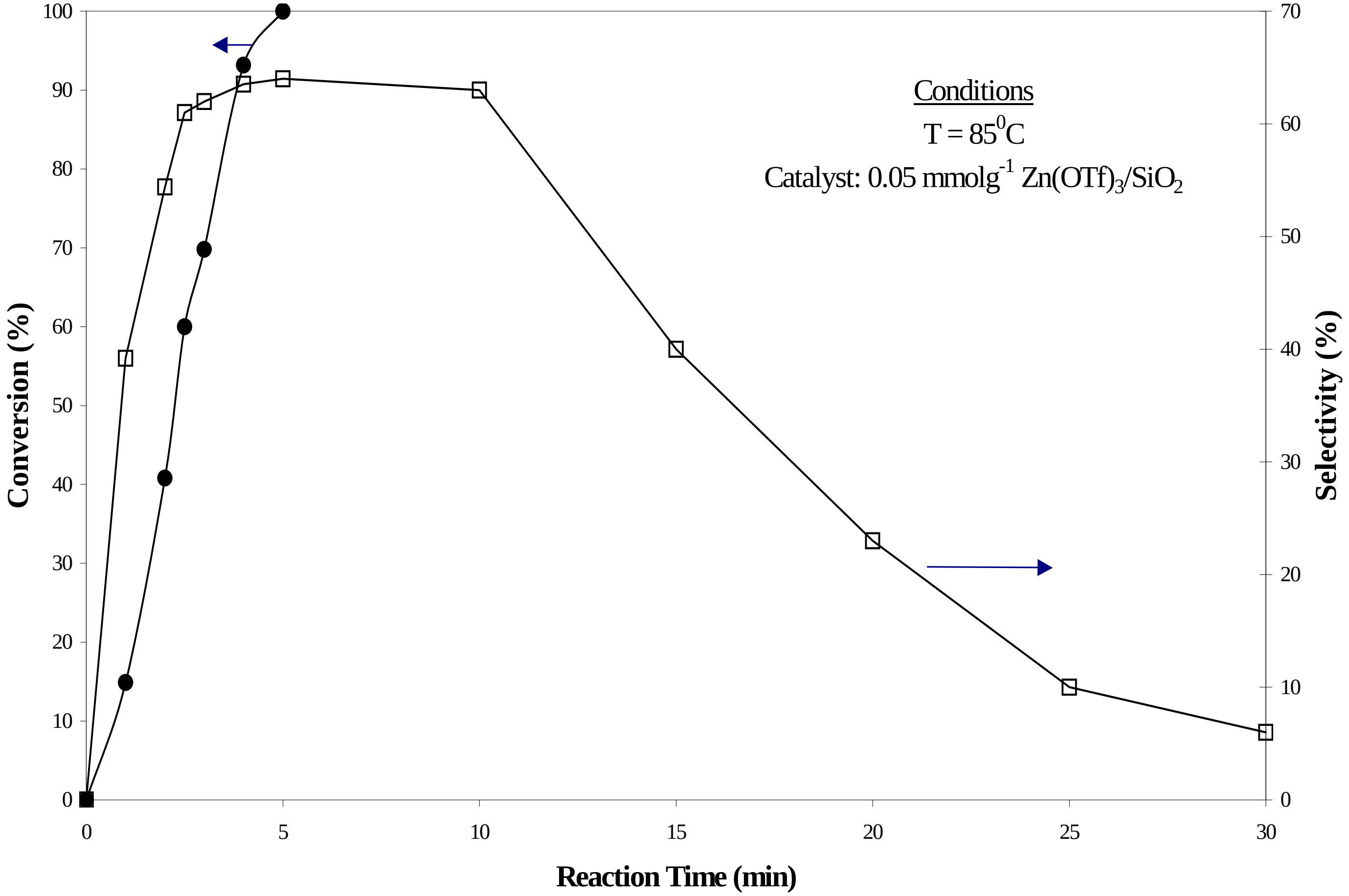
SDR – reaction applications



Fine chemical processes

rearrangement of α-pinene oxide to campholenic aldehyde

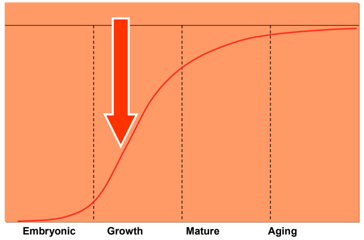
(Batch process)



(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

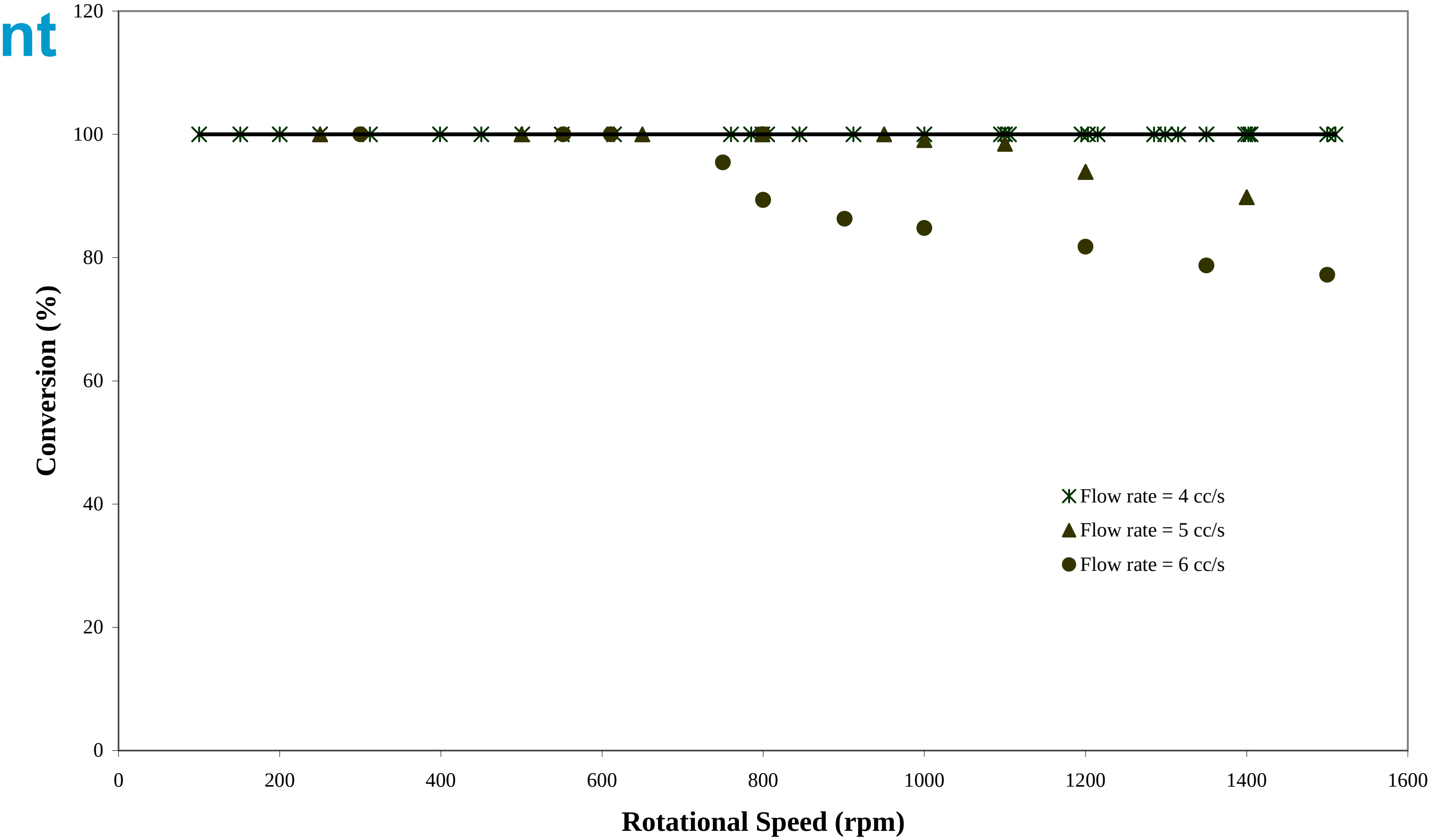
SDR – reaction applications

Fine chemical processes



rearrangement of α-pinene oxide to campholenic aldehyde

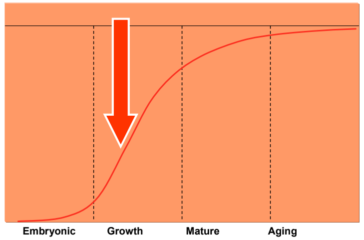
(SDR)



(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

SDR – reaction applications

Fine chemical processes



rearrangement of α-pinene oxide to campholenic aldehyde

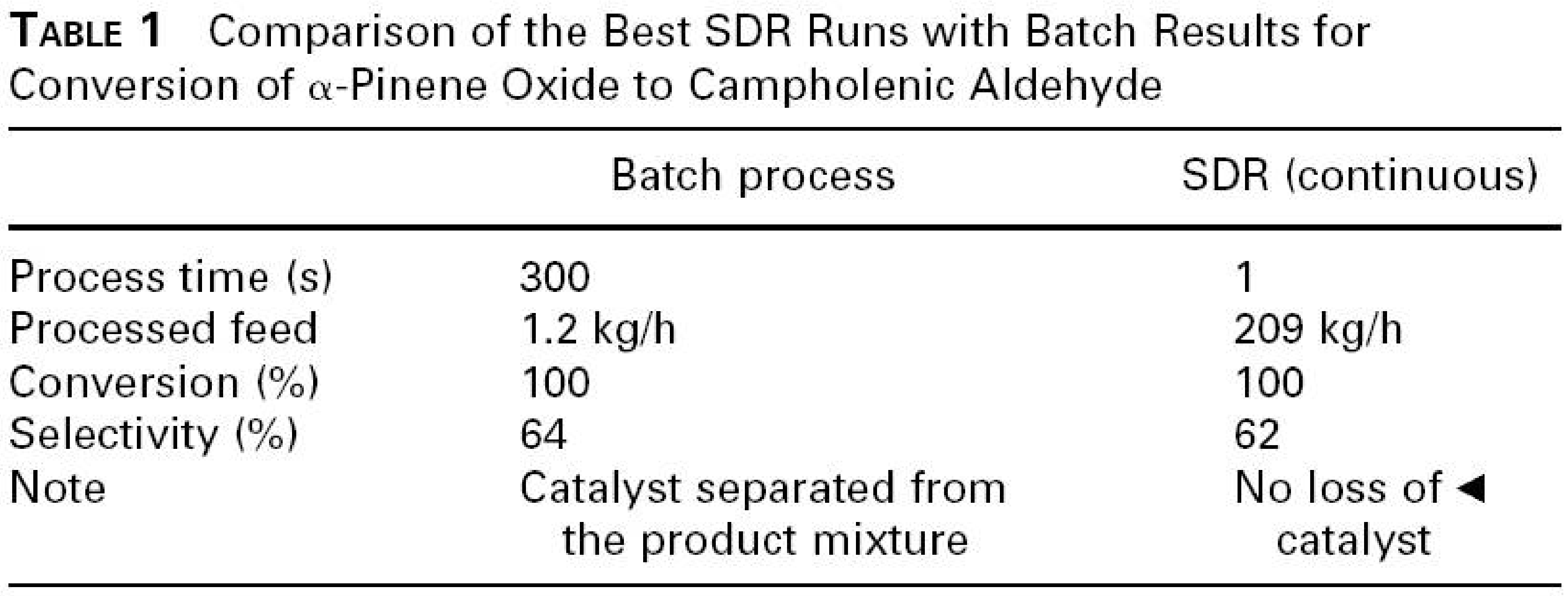
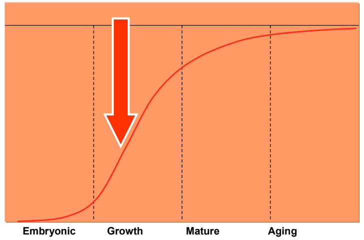


TABLE 1 Comparison of the Best SDR Runs with Batch Results for Conversion of α-Pinene Oxide to Campholenic Aldehyde

	Batch process	SDR (continuous)
Process time (s)	300	1
Processed feed	1.2 kg/h	209 kg/h
Conversion (%)	100	100
Selectivity (%)	64	62
Note	Catalyst separated from the product mixture	No loss of ◀ catalyst

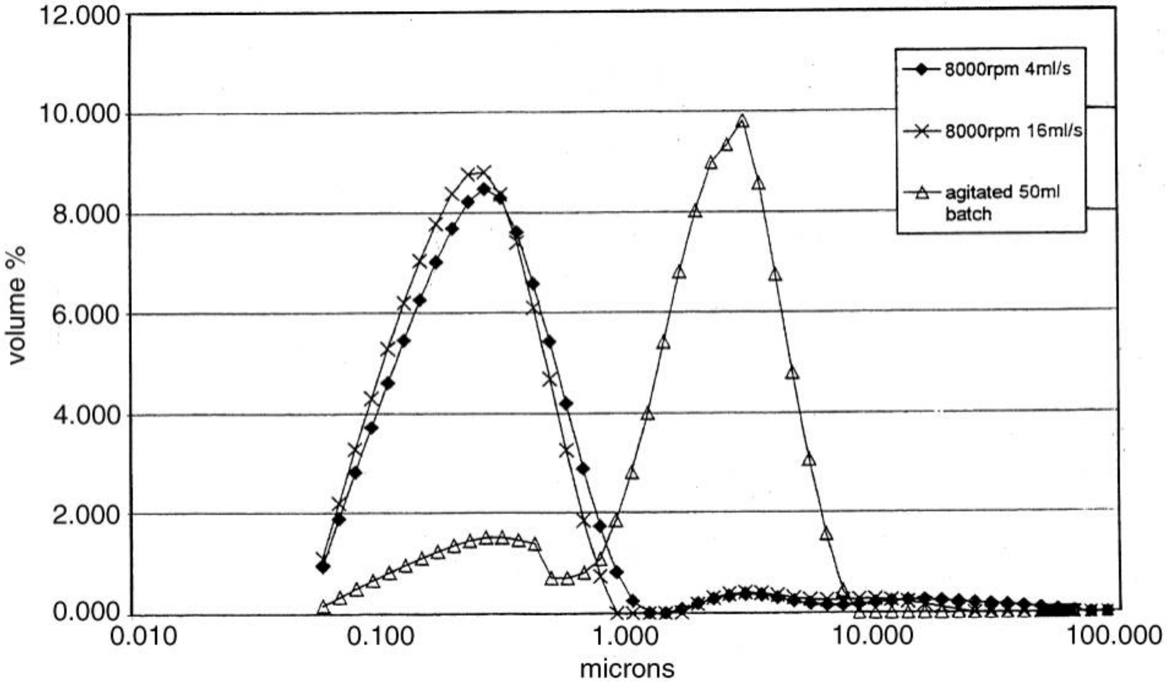
(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

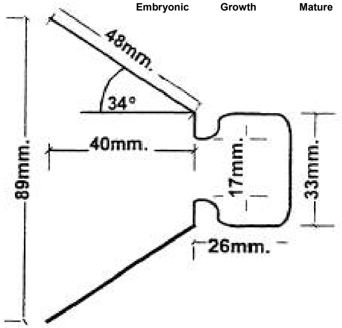
SDR – reaction applications

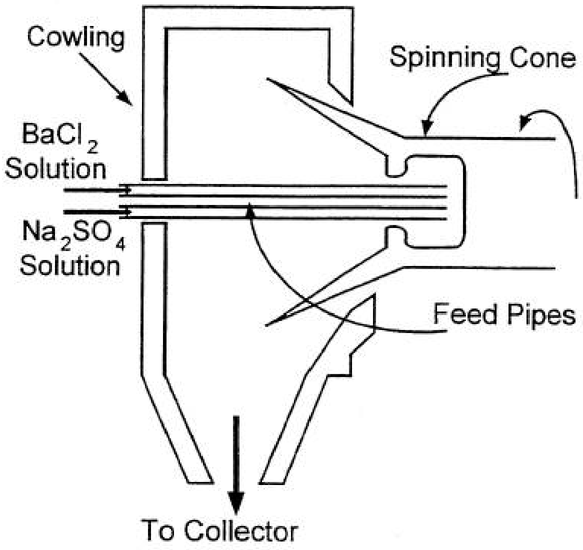


Precipitation

$BaSO_4$ from $BaCl_2$ and Na_2SO_4

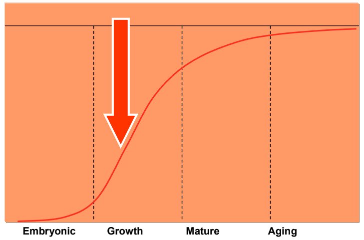






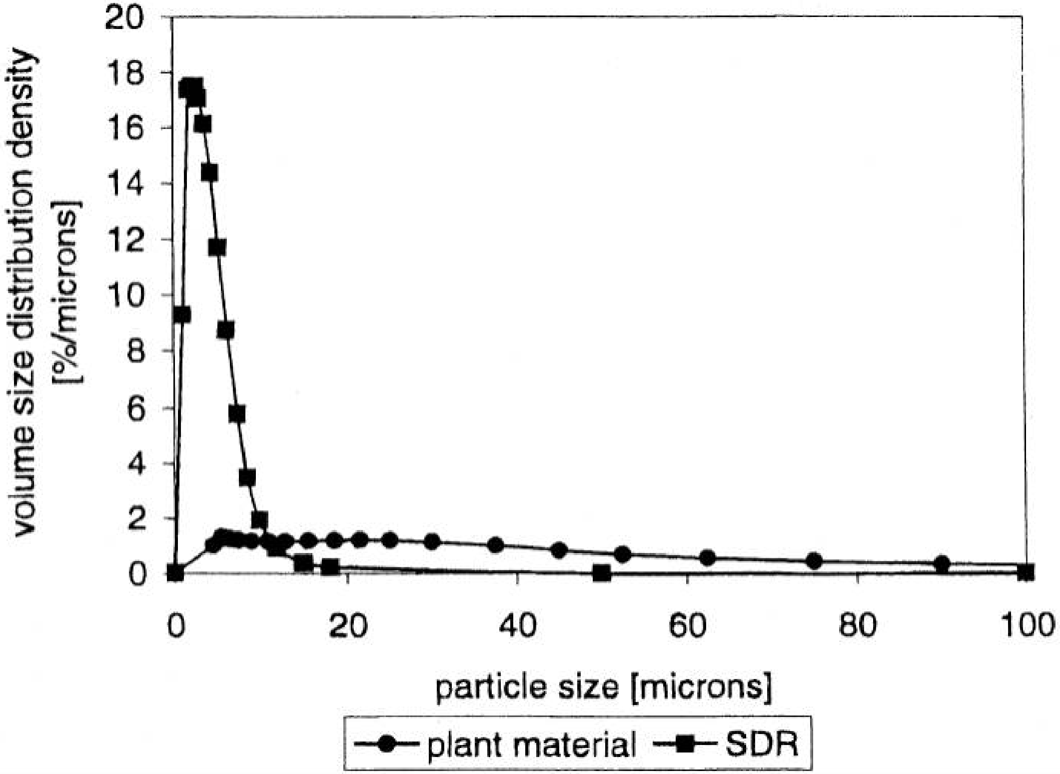
(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)

SDR – reaction applications



Precipitation

Recrystallization of API (unnamed product) by SmithKline Beecham



(source: C. Ramshaw, in: *Re-Engineering the Chemical Processing Plant*, Marcel Dekker, 2003)