

Mixing time in a lab-scale stirred reactor with a catalytic basket

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The Company

■ IFP *Energies Nouvelles*



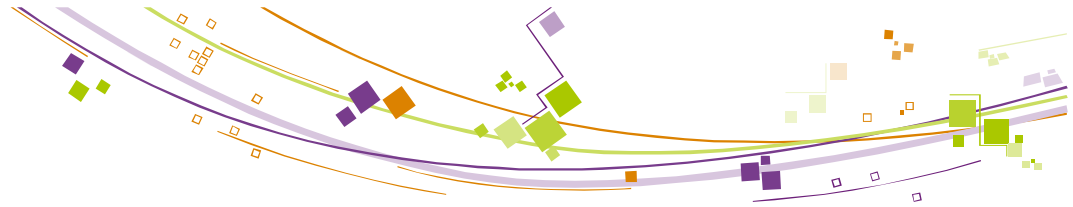
Lyon Site

Public research and training institute with international scope, covering the fields of **energy**, **transports** and **environment**

Transposing research to industry. **Technological innovation** is central to all its activities

Public interest missions:

- providing solutions to face society's challenges in terms of **energy and climate change**, promoting the emergence of a sustainable energy mix
- creating **wealth and employment** by supporting the French and European economic activity, and its competitiveness in related industrial sectors



Evaluation of commercially shaped catalysts

Petrochemical, Refining and New Biological Green industrial Processes



Beads



Extrudates

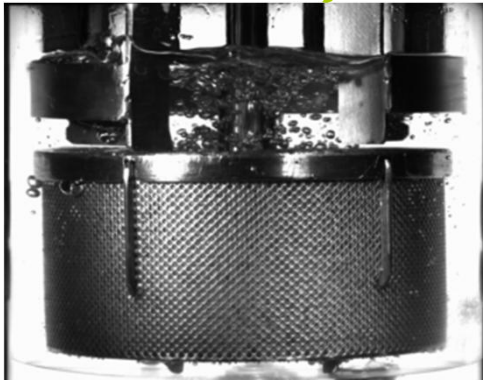
Particles (1-3 mm)

- Catalysts are evaluated/tested in their commercial shape
 - Spatial active site distribution in the particle
 - Multifunctional active sites



Evaluation of commercially shaped catalysts

**IFPEN possible choice:
Stirred reactor with stationary
catalytic basket**



Hollow self-inducing impeller



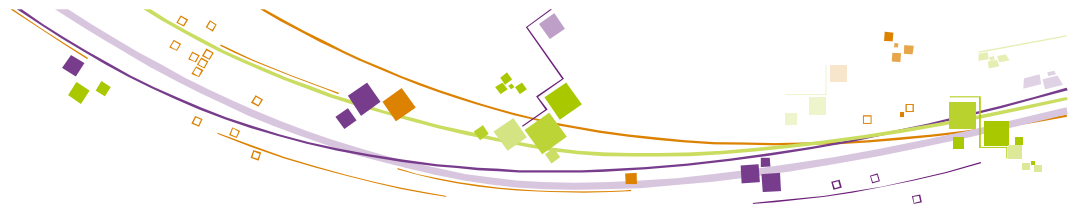
Catalytic basket



Catalytic basket + Stirrer

- Lab-scale baffled stirred tank reactor (STR)
 - Volume = 300 cm³
- Stationary catalytic basket
 - Perforated metal walls
 - Support the catalyst particles (fixed bed)
 - Gap between basket walls = 9 mm
 - Diam. catalyst = 1 – 3 mm
- Self-inducing radial flow impeller
 - Hollow shaft with orifices
 - Recirculation of gas in a closed loop without the need for additional equipment
 - Mixing of the gas/liquid phases
 - D=20-25 mm, 800-2000 rpm

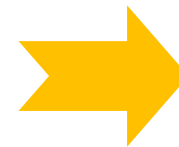
**Highly complex gas-liquid multiphase
turbulent flow (8 000 < Re < 20 000)**



Characteristic times



- Constant development of catalysts with better performance
- Faster reactions
- Mass transport limitations between the phases can compromise kinetic tests



Need to know the reactor's domain of applicability
+ Design optimization

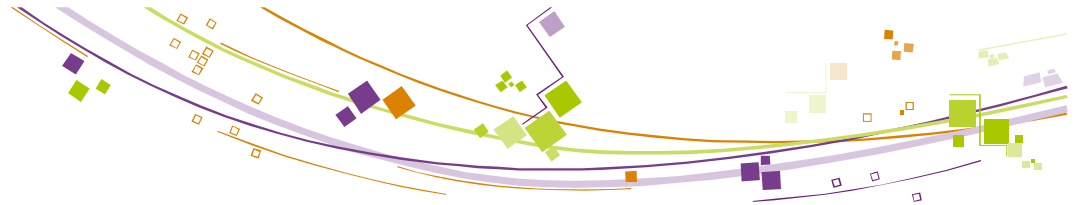


General objectives of the project

- Having deeper knowledge on the **hydrodynamics of STRs with stationary catalytic baskets**
 - Complex multiphase flow
 - Difficult to observe experimentally the flow inside the catalytic basket
 - Only a few papers exist in the literature (for similar, but different, setups)

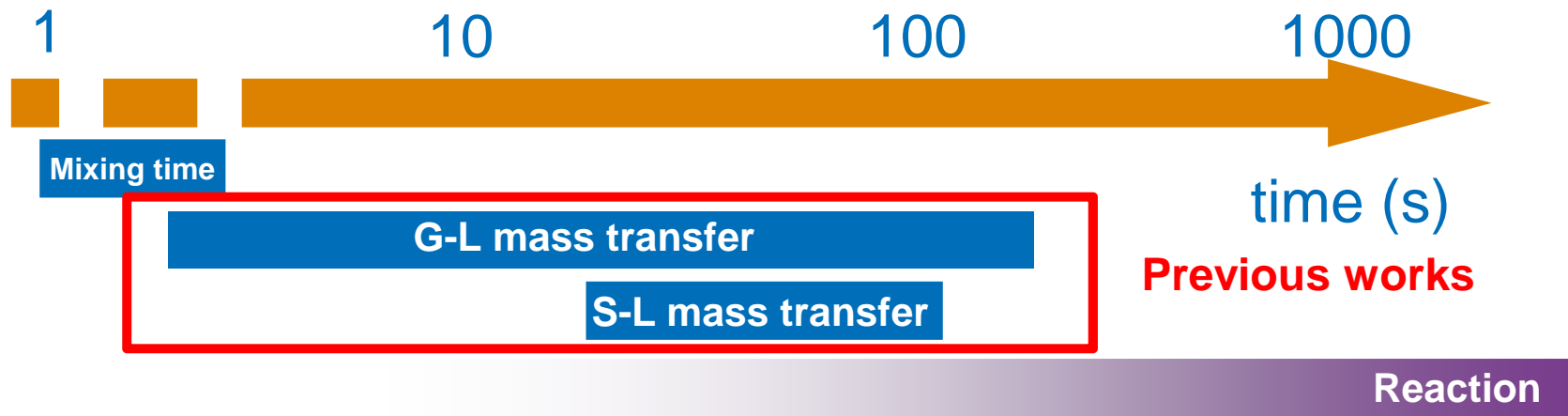
- Knowing the conditions for which the **reaction kinetics is the controlling rate** (for fast reactions).

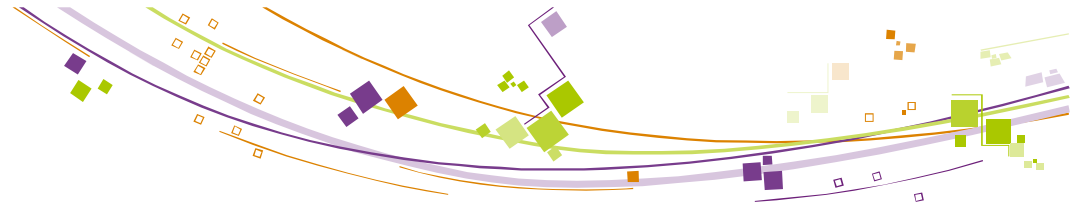
- Finding **more efficient and robust reactor/basket designs** reducing mass transfer limitations:
 - Homogeneous distribution of the gas in the liquid phase
 - Optimal gas absorption rate into the liquid phase ($\uparrow k_L a$)
 - Maximize velocity of the liquid through the catalyst porous medium ($\uparrow k_S$)



Previous works

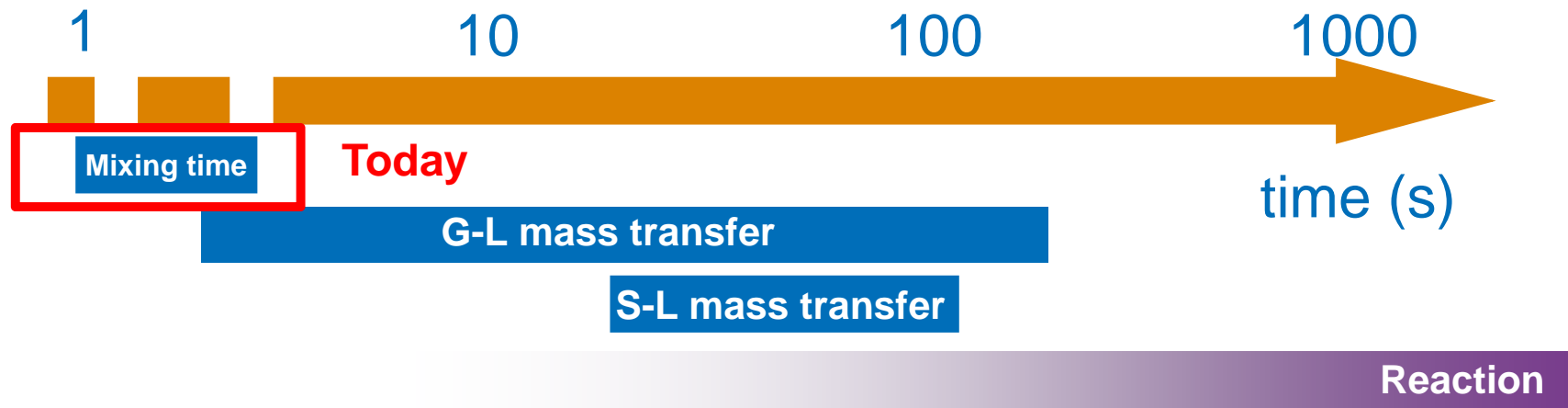
Characteristic times





Previous works

Characteristic times



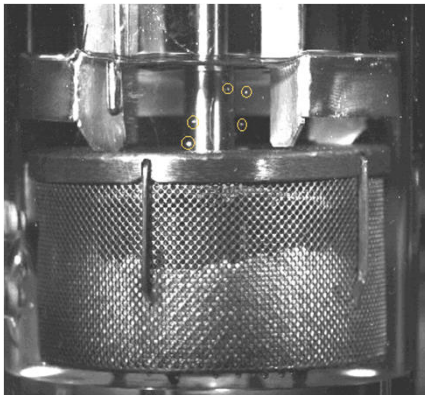


Flow regimes

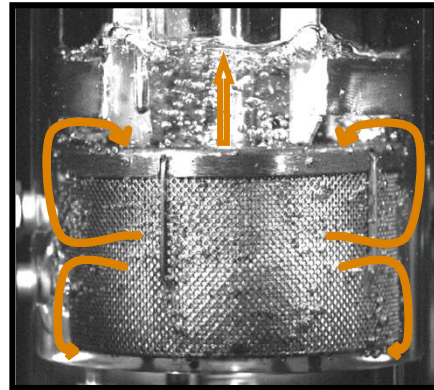
Stirring (800-2000)



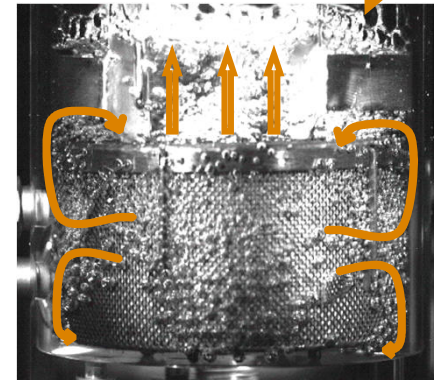
50%



First bubbles regime

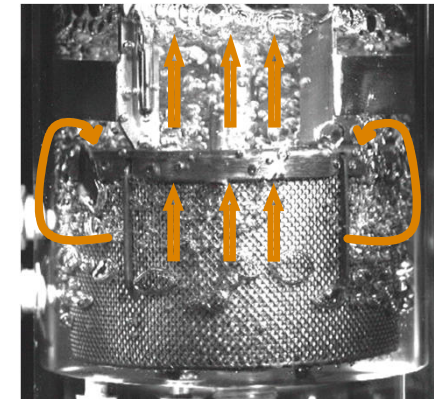
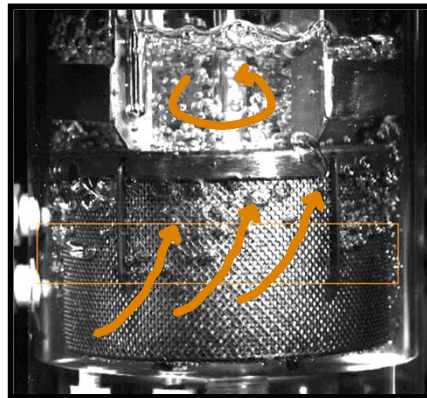
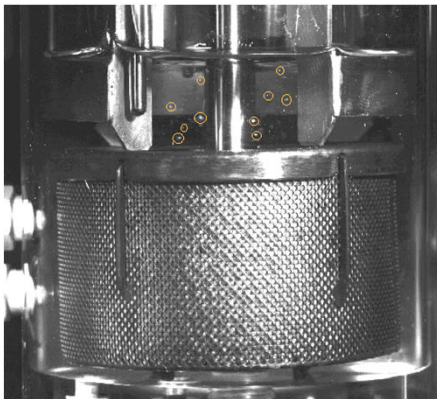


Bubble crossing regime



Coalescence regime

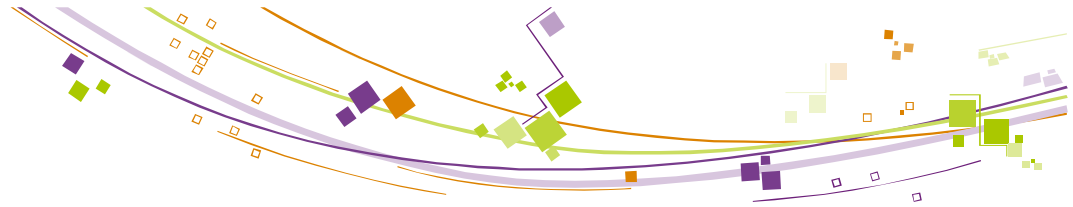
100%



Mixing time ?



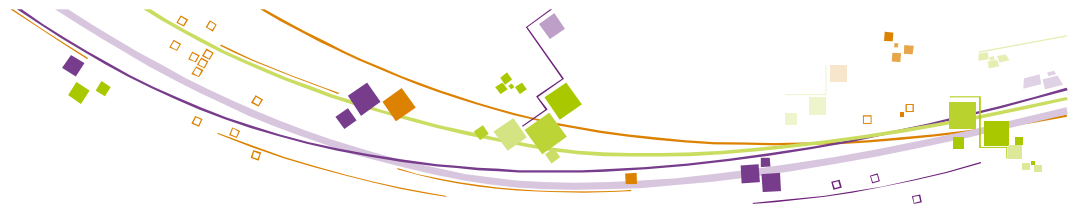
Mixing time



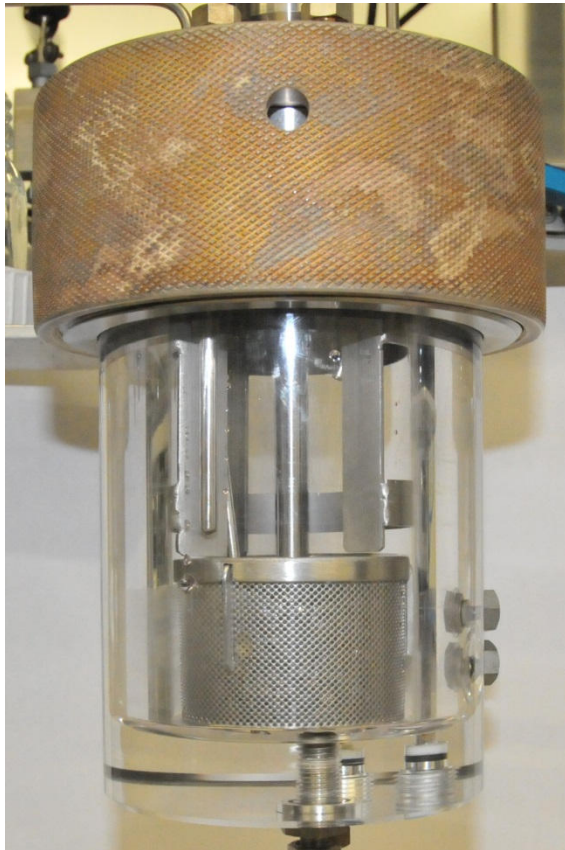
Mixing time, Technique ?

Small lab-scale baffled stirred tank reactor, 300 cm³

Method		Advantages	Disadvantages	Interest for the study of small reactors
Invasive	Conductimetry pHmetry Refractometry	Use of simple and low price probes Easy set-up & implementation	Need of several probes to study the local mixing times High consumption of fluids (products and tracer) Intrusive method	★ ★
	Thermal	No product consumption. Low changes of the nature of the fluids if a low quantity of tracer is employed	Need of reactor insulation High cost	★
Non-Invasive	Radioactivity	Accurate	High cost, Safety issues	★
	Colorimetry with or without chemical reaction	Visualisation and comprehension of the flow pattern in the reactor Possibility to have the mixing times in different parts of the reactor in a single experiment	Impossible to apply to the study of opaque fluids Difficulty to identify the t_0 High consumption of fluids (fluids and tracer)	★ ★ ★

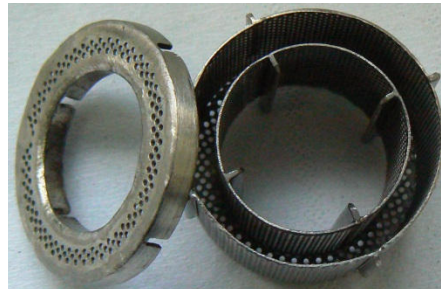


Mock-up



PMMA Mock-up

Inner diameter of 58 mm and height of 118 mm (design from Top Industrie)



Basket (50% and 100% loading)

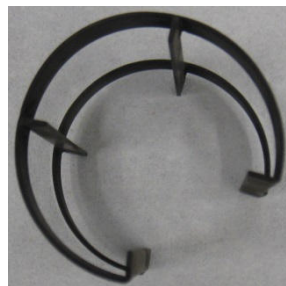


R25



R20

Basket + Stirrer



Baffles

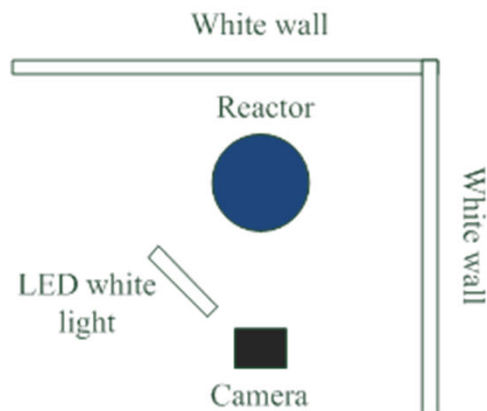


Hollow self-inducing impeller

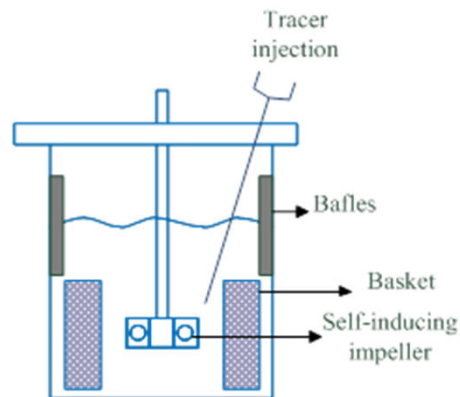


Experimental Setup

Colorimetry without chemical reaction



Global setup



Reactor

Conditions

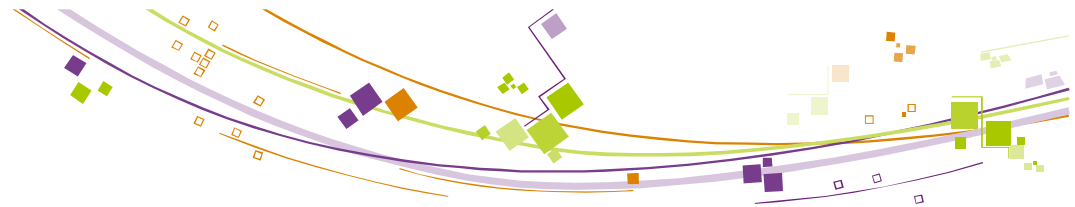
- Room temperature (20°C) and atmospheric pressure
- Liquid phase: Hydrocarbon media- Heptane, 300 ml
- Gas phase: Nitrogen
- Liquid tracer: Methyl red dissolved in ketone and heptane (yellow color), 0.3 ml
- Images of the tracer dispersion in the reactor: Reflex Nikon D300s, with the ability of capturing 1280×720 pixels images at a frame rate of 25 images/s
- White light LED was used to avoid external light interferences

Optimization of the volume of injection

Position of the injection

Effect of the time of injection

Determination of the mixing time at different conditions: with and without the basket, different agitation, liquid height, solid load, etc.



Determination of the mixing time

Methodology

- Sequences of images of the dye dispersion
- Definition of regions of interest (ROI)
- Following the pixel color change in ROI in function of the time until steady state
- Quantification of the color changes in each ROI for each image (Halcom software)

- Subtraction of the first image, without tracer
- Measure of the average pixel intensity on the blue channel
- Computation the final intensity by averaging the pixel intensity on the last 20 images
- Plot the intensity normalized by the final intensity against time

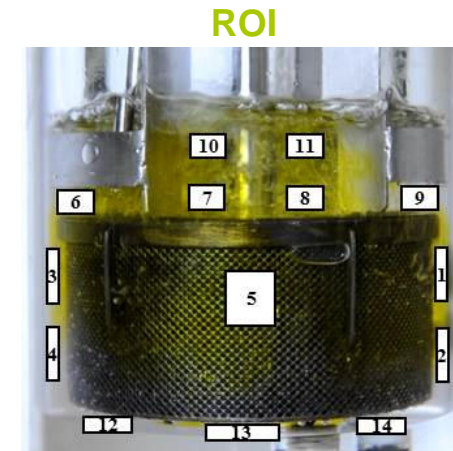
$$C_r = \frac{C_i - C_0}{C_\infty - C_0}$$

- Plot the variance in fonction of the time (M is the number of ROI)

$$\log \sigma^2 = \log \left(\frac{1}{M} \sum_1^M \sigma_M^2 \right) \text{ where } \sigma_M^2 = (C_r - 1)^2$$

Mixing time at 95% is defined as the time required to reach steady state in all ROI

$$\log(\sigma^2) = -2,6$$



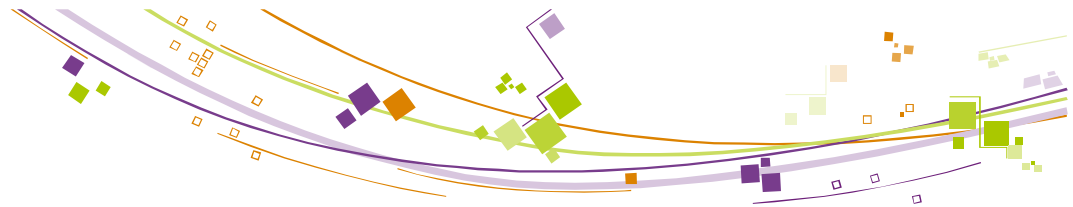
With basket



Without basket

¹ Average pixel intensity is an integer number comprised between 0 and 255.

² Blue channel was chosen as it was the one leading to the best signal to noise ratio.



Example

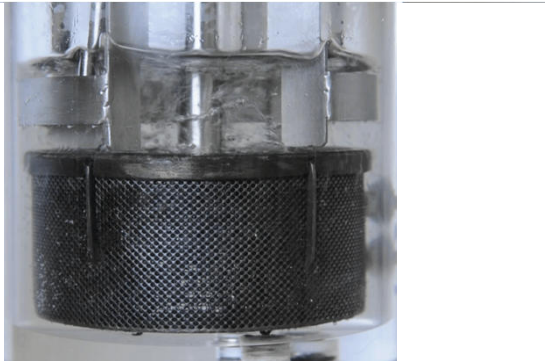
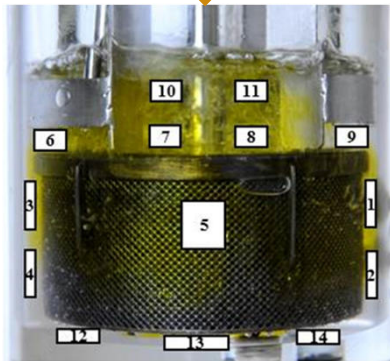
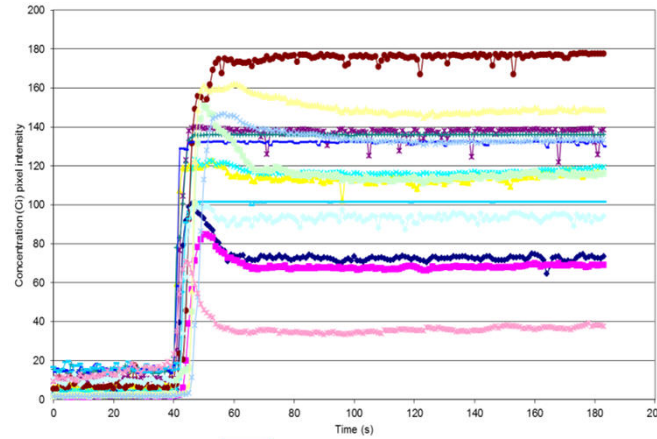


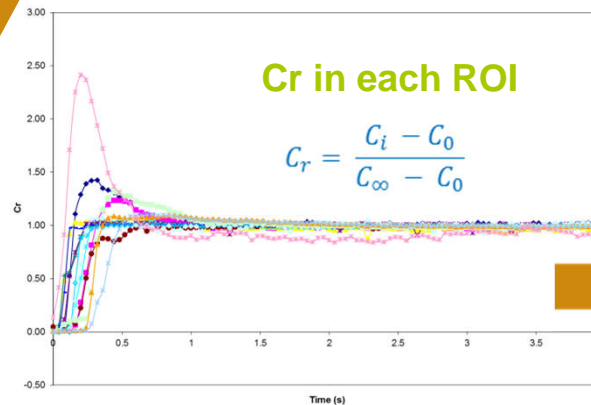
Image sequence acquisition



Post-treatment of the images



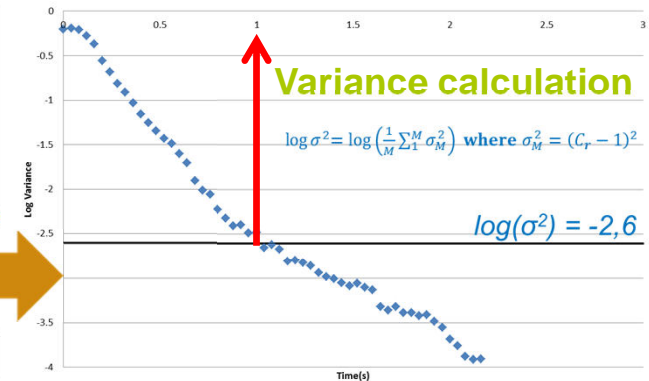
Ci in each ROI



Cr in each ROI

$$C_r = \frac{C_i - C_0}{C_\infty - C_0}$$

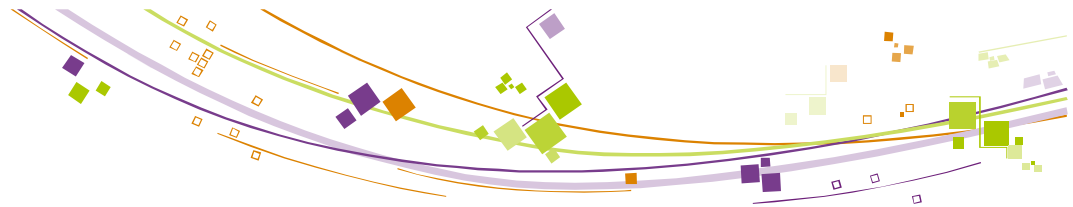
Mixing time at 95%



Variance calculation

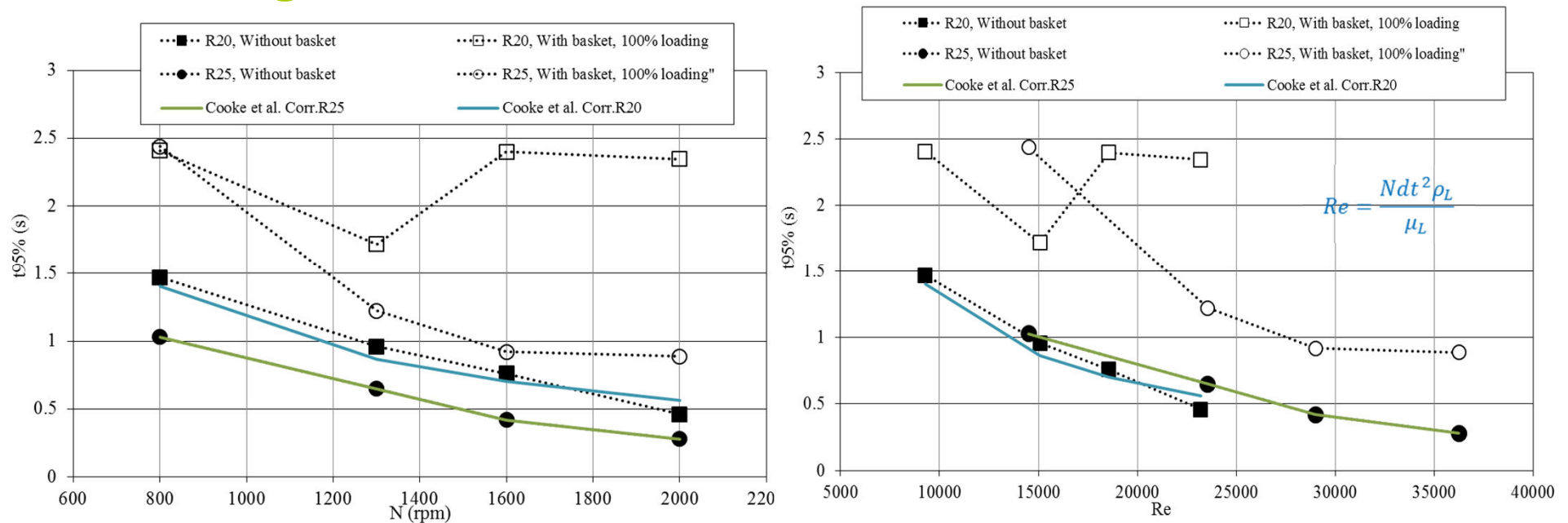
$$\log \sigma^2 = \log \left(\frac{1}{M} \sum_{i=1}^M \sigma_M^2 \right) \text{ where } \sigma_M^2 = (C_r - 1)^2$$

$$\log(\sigma^2) = -2,6$$



Results

Mixing time, with and without the basket



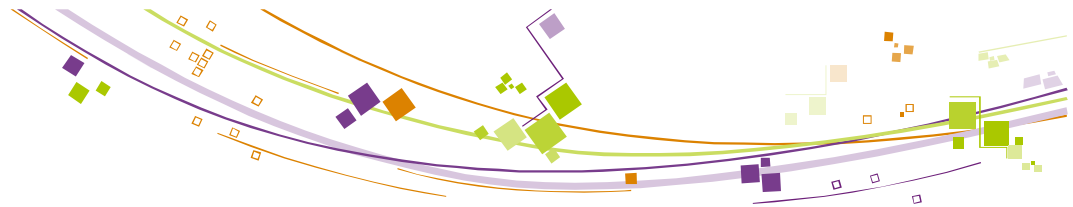
■ Without basket

- Short mixing times, good agreement with Cooke and Middleton correlation (method validation)
- Mixing time is a function of the hydrodynamic conditions of the system (Re)

■ With basket

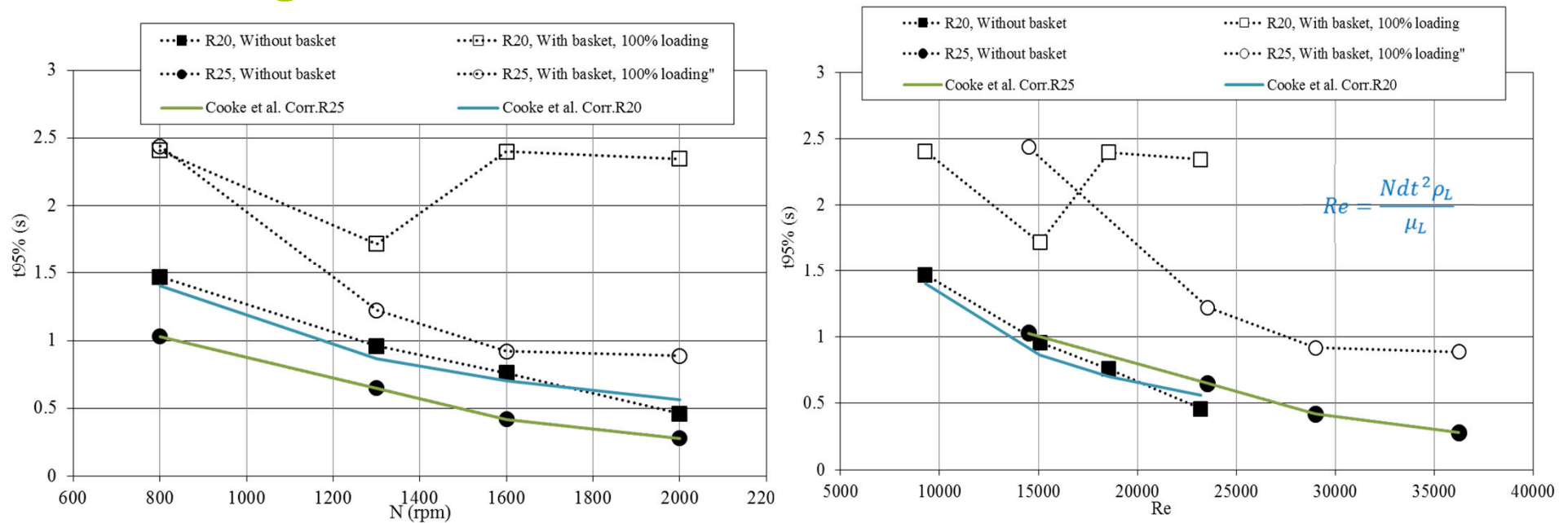
- Short mixing times but increase (x2 or 3) in relation to without basket
- Influence of the reactor configuration and not only the hydrodynamic conditions of the system

$$t_{95\%} N_{pg}^{1/3} N \left(\frac{d_t}{d_R} \right)^{2.4} \left(\frac{d_R}{h_L} \right)^{2.4} = 3.3$$



Results

Mixing time, with and without the basket



■ With basket

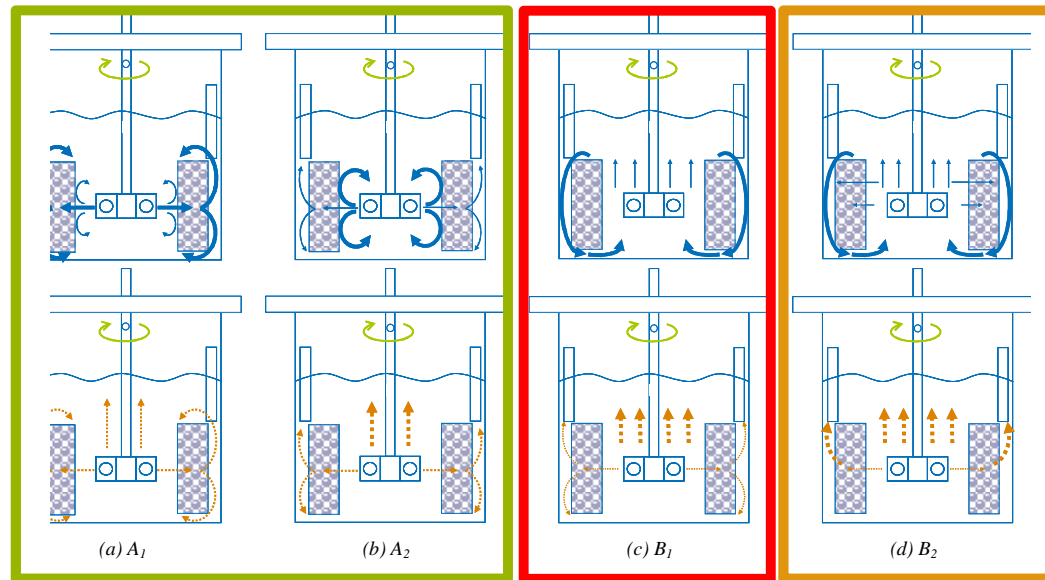
- Increase of stirring rate: Different effects for R20 and R25, Different flows regimes
- Increase of stirrer diameter: Increase of the homogeneity of the fluids



Flow regimes (PIV and visualisations)

Liquid

Gas



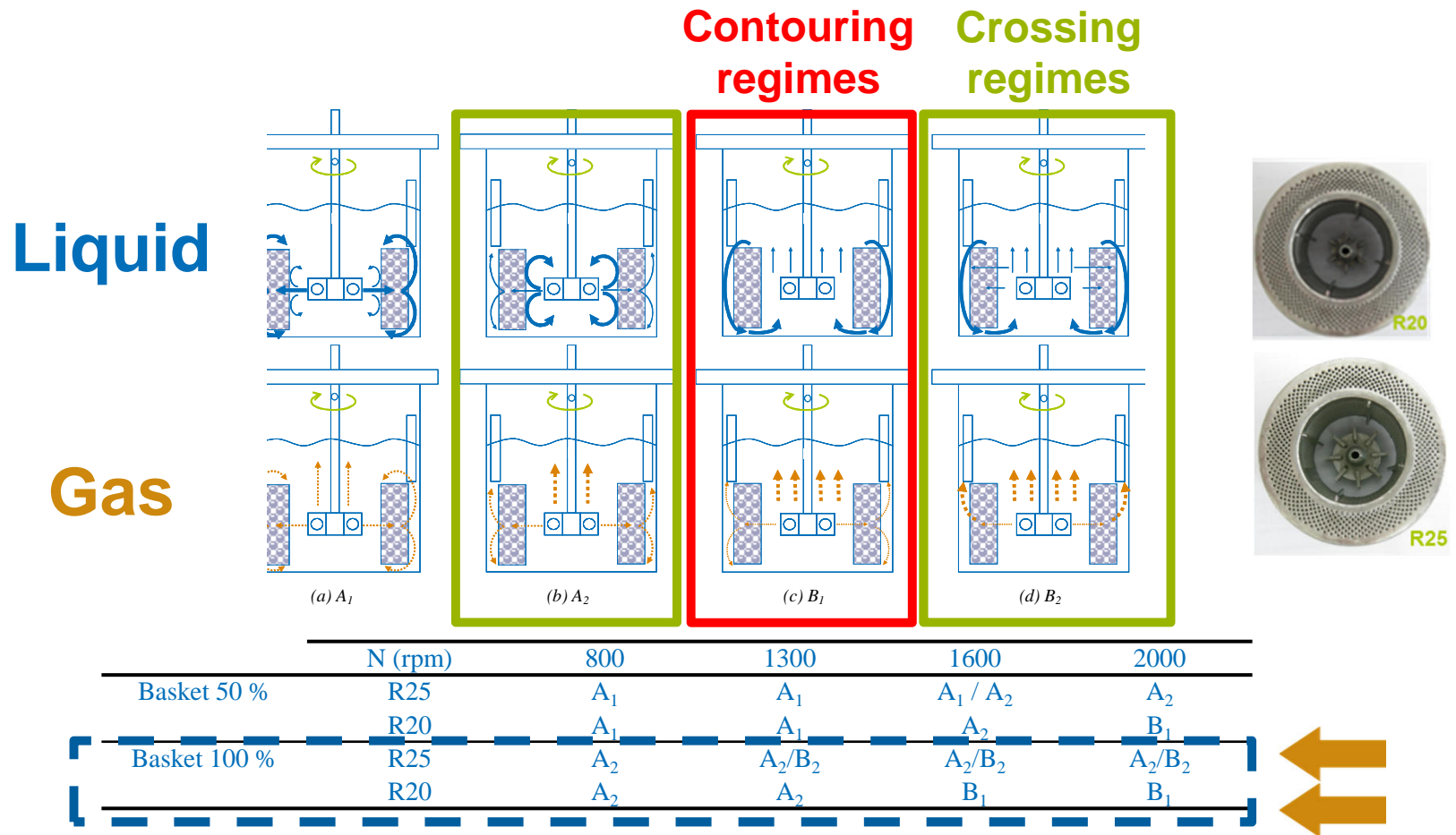
Crossing regime

Liquid Contouring regime
Avoid !!!

Crossing and Contouring regime



Flow regimes (PIV and visualisations)

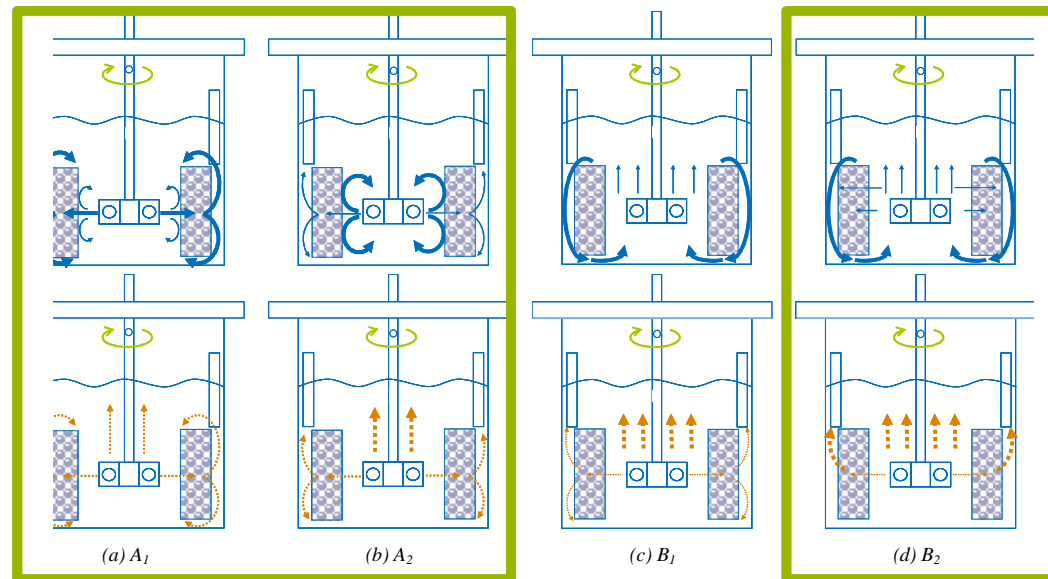




Flow regimes: PIV and visualisations (R25 turbine)

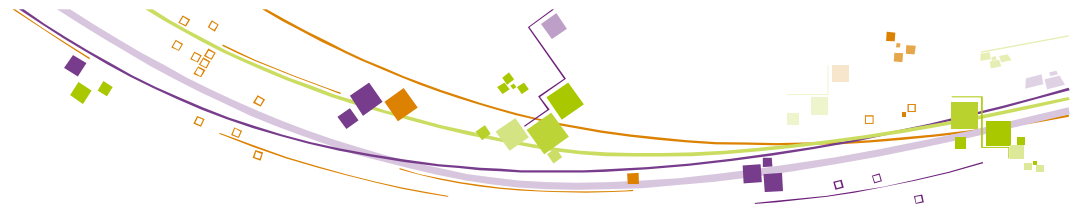
Liquid

Gas



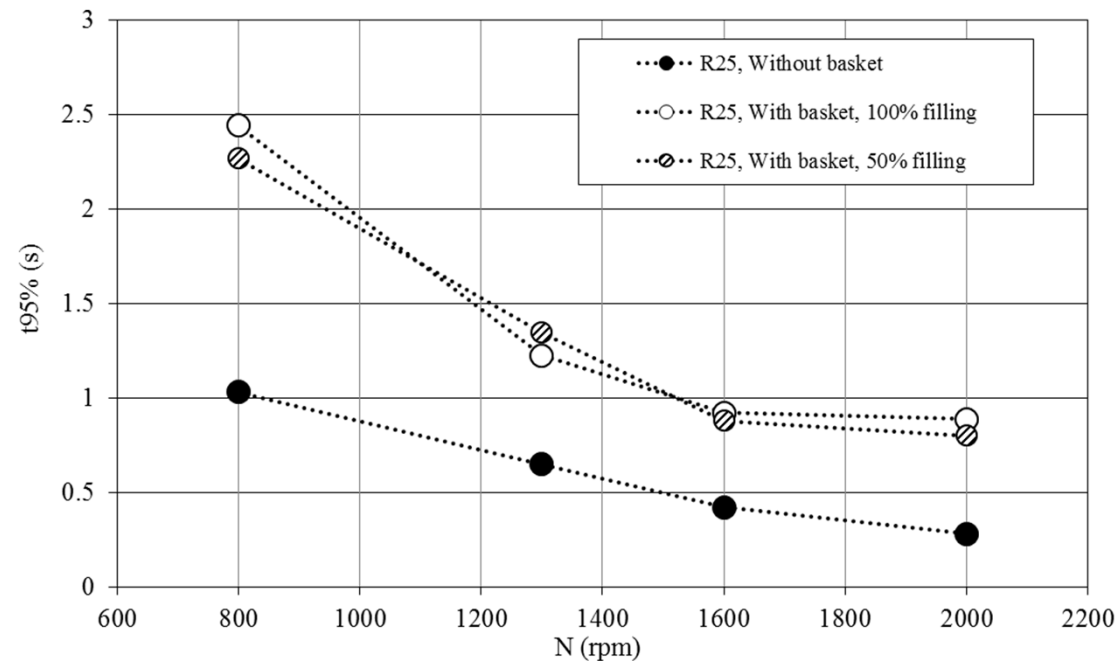
Crossing regimes

	N (rpm)	800	1300	1600	2000
Basket 50 %	R25	A ₁	A ₁	A ₁ / A ₂	A ₂
Basket 100 %	R25	A ₂	A ₂ /B ₂	A ₂ /B ₂	A ₂ /B ₂



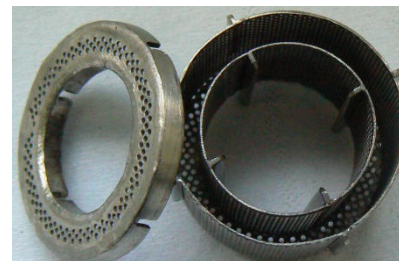
Results (R25 turbine)

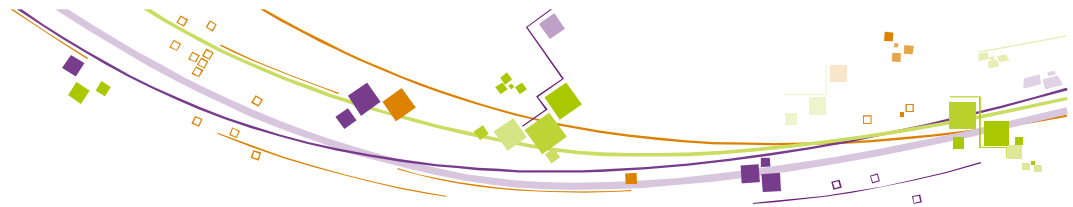
Mixing time, with and without the basket and % of filing



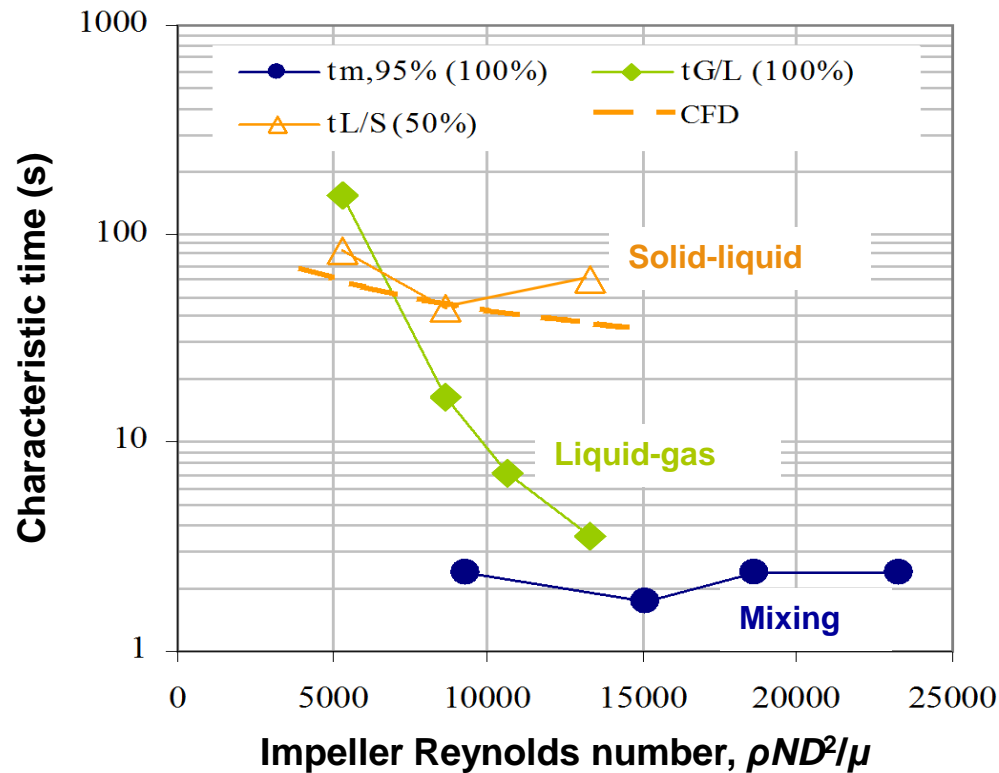
■ Filing

- No impact of the filing
- Impact of the presence of the basket
- Similar regimes





Characteristic times (R25 turbine)



Mixing time is not the limiting factor for stirred reactors with catalytic basket

Braga, M., 2013, PhD Thesis. « Etude des phénomènes de transfert et de l'hydrodynamique dans des réacteurs agités à panier catalytique (Study of transport phenomena and hydrodynamics in stirred reactors with a catalytic basket). » Université Claude Bernard Lyon 1, Lyon, France.



Conclusions

- **Methodology to estimate mixing times in stirred reactors**
 - Simple method based in image processing
 - Non intrusive method
 - Hydrocarbon media
 - Presence of gas
 - Small size reactors
- **Limitations**
 - Light control
- **Estimation of the mixing time in stirred reactors with and without catalytic basket**
 - Presence of the basket increases the mixing time
 - Small mixing times compared with G/L and L/S mass transfer
 - Flow regimes (liquid flow regimes) correlated with mixing time
 - Two small stirrers can induce a basket contouring regime of the liquid



Work in progress

- Other techniques to get mixing time in hydrocarbon media in small “exotic” reactors
 - UV-Visible spectroscopie



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**Thank you for your
attention**

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