THE ADVANCED MULTIPHASE SYSTEMS

UCL

Complex ORAL health products (CORAL)
Characterisation, modelling and manufacturing challenges

EPSRC Future Formulation grant

Team

Chemical Engineering

Panagiota Angeli: Two-phase flows, intensified processing, laser based flow diagnostics (PIV, LIF)

Luca Mazzei: Two-phase flows, particulate systems, CFD, numerical modelling

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Mechanical Engineering

Stavroula Balabani: experimental fluid mechanics, particle suspensions, RheoPiv, microstructure

Manish Tiwari: nanotechnology, rheology, AFM

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Mathematics

Helen Wilson: rheology, non-Newtonian fluids, mathematical analysis

Liam Escott

Industrial support: GSK, Xaar

CPI

Overall vision

The main aim is to gain fundamental understanding and develop methodologies to aid manufacturing of complex formulations, including particle suspensions in non Newtonian fluids.

Issues include interparticle interactions, particle aggregation, particle wetting and swelling and inclusion of air bubbles.

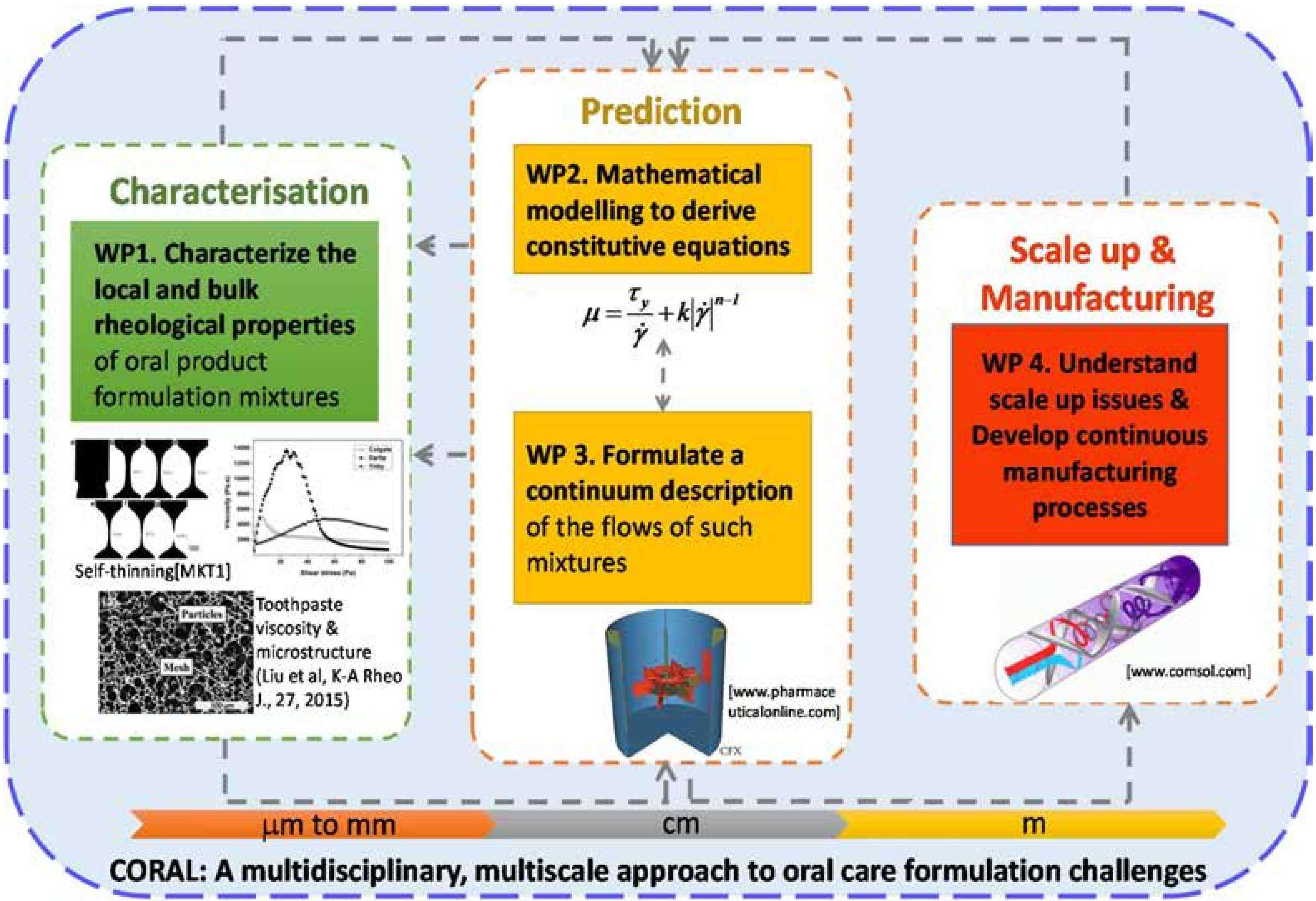
Aim of CORAL

We will follow a multiscale approach involving **mathematical analysis**, **numerical simulations** and **advanced experimentation,** to

- *elucidate* the dependence of the nonlinear rheology of the suspensions/pastes on microscale interactions
- *develop* the modelling tools, which describe the effects of process conditions on the suspension/paste rheology.

These will enable us to address **manufacturing** and **scale up** issues and to propose new **intensified continuous processes.**

Strategy/workpackages



WP1

Characterize the local and bulk rheological properties of oral product formulation mixtures

Rheology of suspensions

Provide insight into behaviour of non aqueous particle suspensions and inform mathematical and numerical modelling.

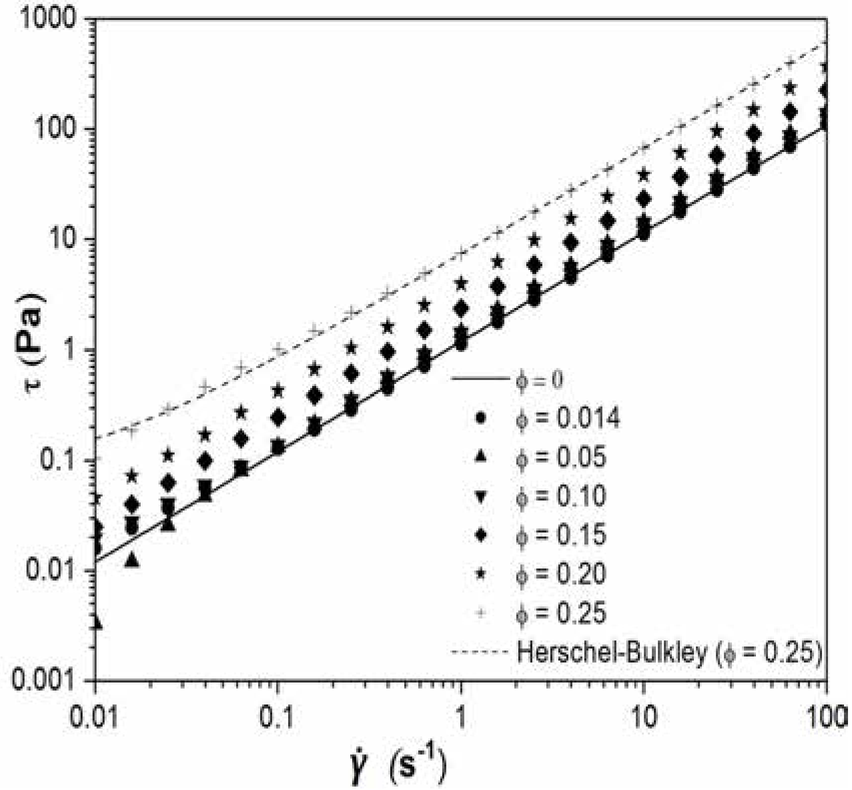
1. Characterise bulk rheology of particle suspensions in non aqueous matrices

    - monodisperse and polydisperse particle suspensions in glycerol
    - same suspensions in non Newtonian (glycerol+ polymer) matrix

2. Provide a link between microstructure and rheology through Rheo-optics
3. Probe interparticle forces of suspended particles in in non-Newtonian media.

Rheology of suspensions

- Silica particles suspended in glycerol using using high shear mixer
- Parallel plate geometries used: 40 mm Parallel H/A Aluminium (DHR-3) (A)
  - 50 mm Sandblasted Stainless Steel (ANTON-PAAR MCR301) (B)

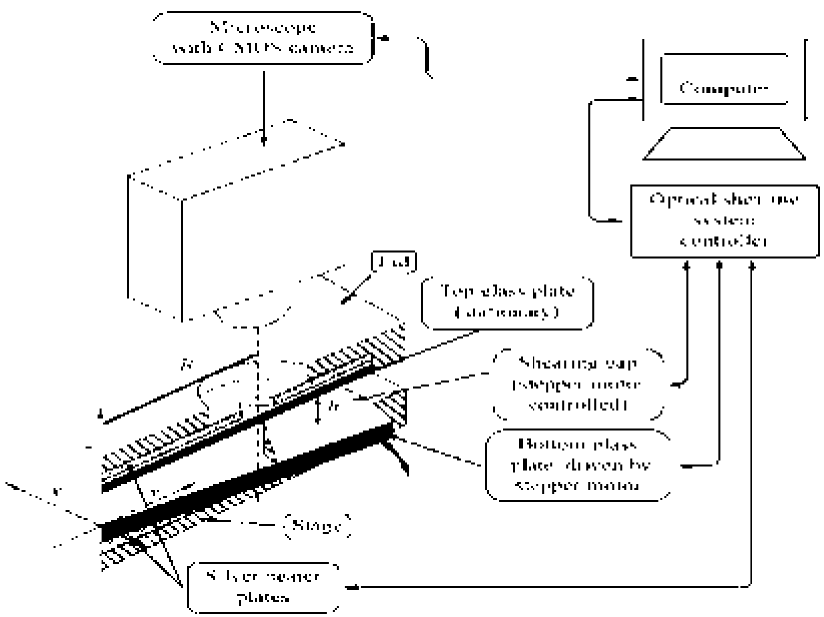


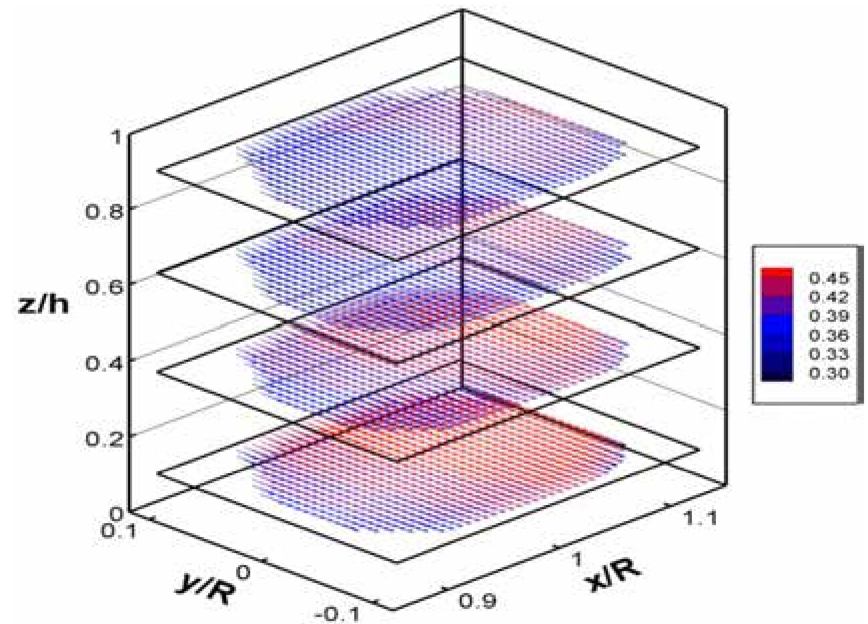
Non-Newtonian behaviour is observed only for **<u>φ = 0.25</u>**, since Herschel - Bulkley model showed:

n = 0.95 & $\boldsymbol{\tau_0}$ = 0.15 Pa

Optical shearing and mPIV (rheoPIV)

- Couette device (30µ*m* shearing gap);contant shear
- Image microstructure during shearing
- Characterise aggregation but also flow kinematics





Velocity field in different planes

AFM studies of inter-particle forces

Multimode 8 AFM, Bruker

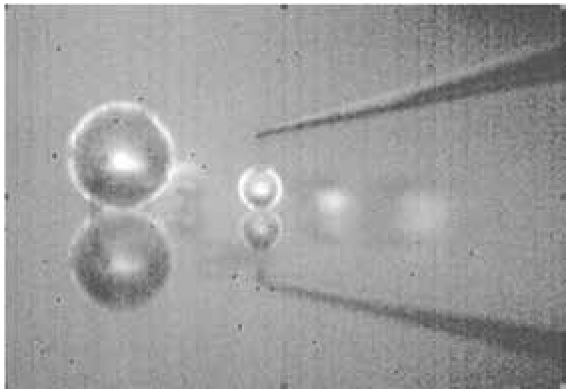


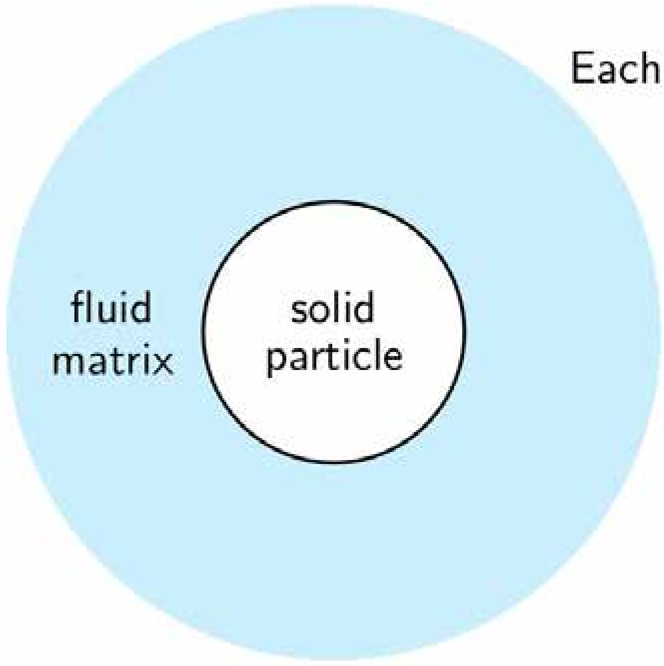
Image courtesy of Ian Armstrong, Bruker

WP2

Mathematical modelling to derive constitutive equations that describe the rheology of the systems studied above and incorporate microscale interactions

Solid particles in a viscoelastic fluid matrix
Effect of particles on macroscopic rheology.

- Slow flow expansion (weak elasticity)
- Mean-field "cell model" as a proxy for solids concentration:



Each particle uses identical "effective volume"

Particle radius a

Cell radius b

Effective concentration $\phi = \frac{a^3}{b^3}$

Objective: deduce best possible one-phase constitutive model of the two-phase system (matrix and particles).

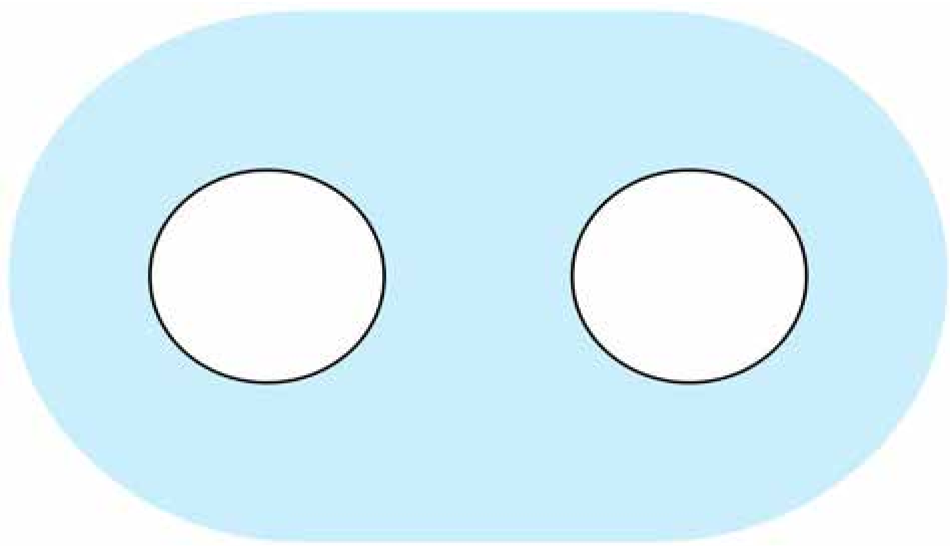
Analytical work – follow up steps

1) Better matrix models, different inclusions.

- Background fluid rheology is **not** weakly viscoelastic! Need more realistic VE models
- There may be bubbles

2) Account for particle-particle interactions

- Incorporate inter-particle forces from AFM experiments
- Quantify local effects of fluid layer between close particles



WP3

Formulate a continuum description of the mixture flows

Objectives

- Simulate the mixture fluid dynamics and the particle mixing using CFD
- Implement the mixture rheology (obtained by means of mathematical modelling or experiments) for validation
- Perform a sensitivity analysis on the most important parameters affecting the fluid dynamic behaviour of the mixtures

Challenges

- Polydisperse, non-spherical particles with high concentration
- Different effects of different particles on the rheology of the mixture

Simulation approaches

1. Eulerian-Eulerian model

Solves mass and momentum balance equations for each phase

2. Mixture model

- Solves the mass and momentum balance equations for the mixture, a mass balance equation for the solid phase and an algebraic equation for the relative velocity between the fluid and solid phases
- Allows us to use a mixture viscosity obtained from experiments

Current work

Mixture viscosity in mixture approach: 1) Batchelor's equation, 2) Experiment

Particles are assumed to be monodisperse and spherical

Eulerian-Eulerian approach	Mixture modelling approach
Newtonian liquid + particles	Newtonian liquid + particles
Non-Newtonian liquid + particles	Non-Newtonian liquid + particles

We do not recover the non-Newtonian behaviour in the dense limit from the E-E approach by using the default formulation in Fluent.

Use the **mixture approach** with viscosity obtained by rheological measurements or mathematical modelling. These will include polydisperse particles and non-Newtonian matrices

WP4

Scale-up and manufacturing issues

Characterisation of industrial mixer

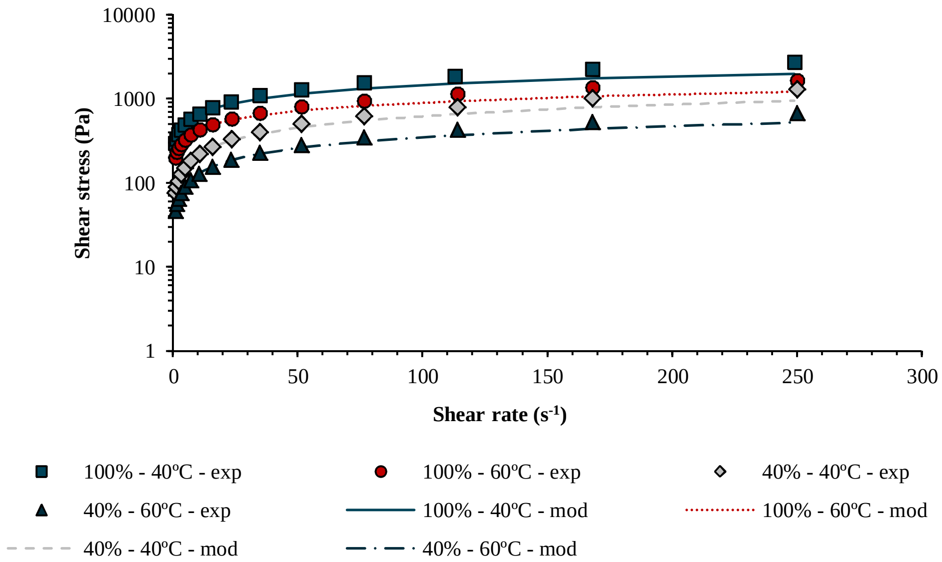
Initial studies without solids.

Objectives

- Develop and validate the CFD code
- Investigate the rheology of the mixtures

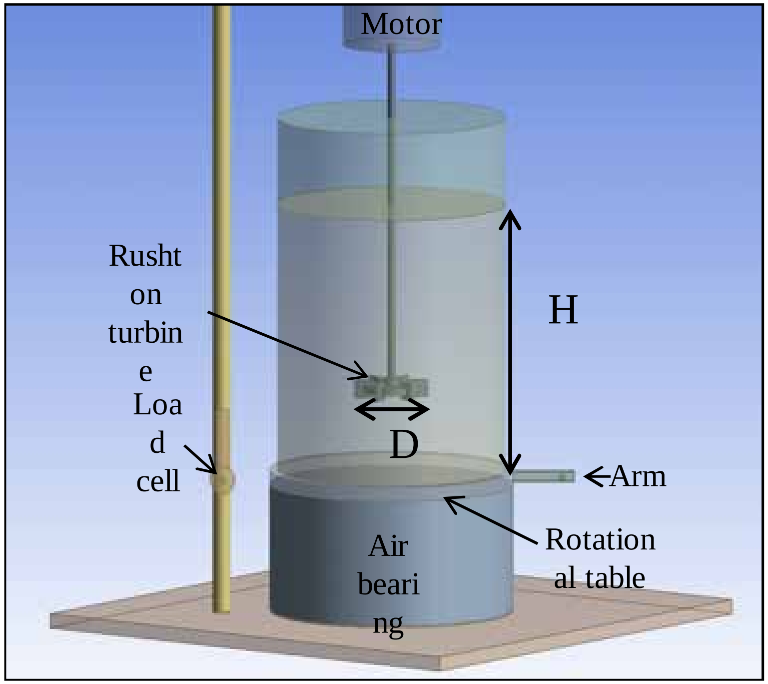
Mixtures of glycerol with activated gel

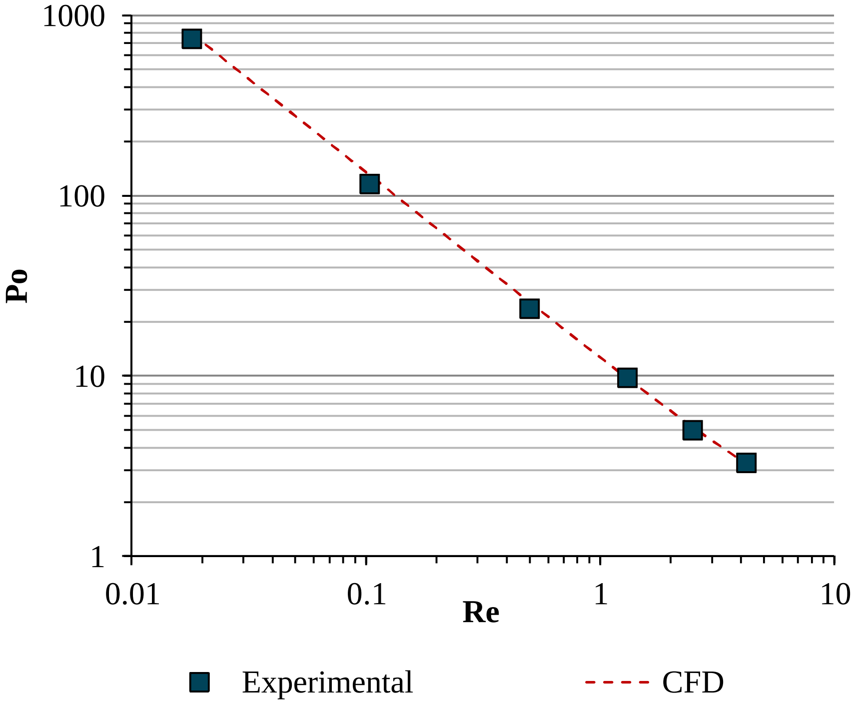
Detailed rheology measurements for different temperatures and phase fractions.



CFD model validation

- Simple mixer configuration
- Accurate torque/power measurements using a air bearing



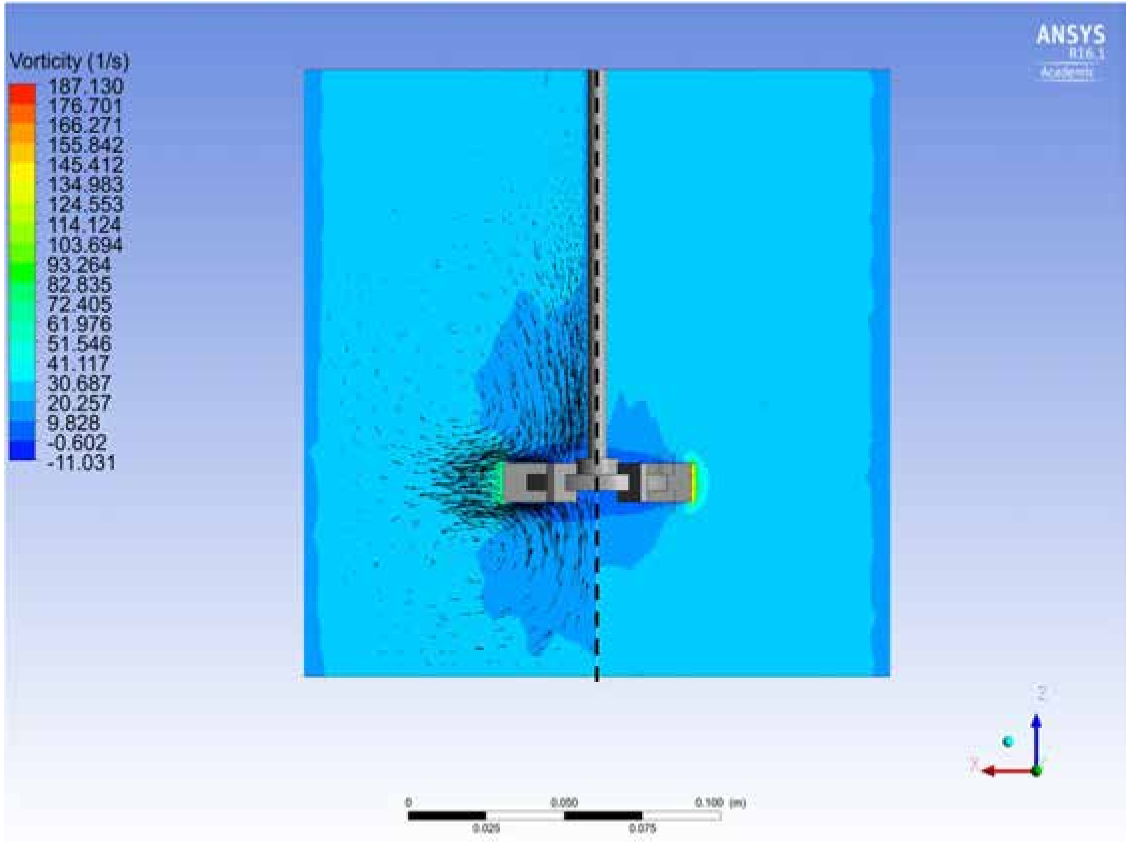


Gel 80%/Glycerol 20% at 60 ºC

Cortada-Garcia, Dore, Mazzei, Angeli, ChERD, 119, 171, 2017.

Characterisation of industrial mixer

CFD simulations reveal dependence of mixing on rheology

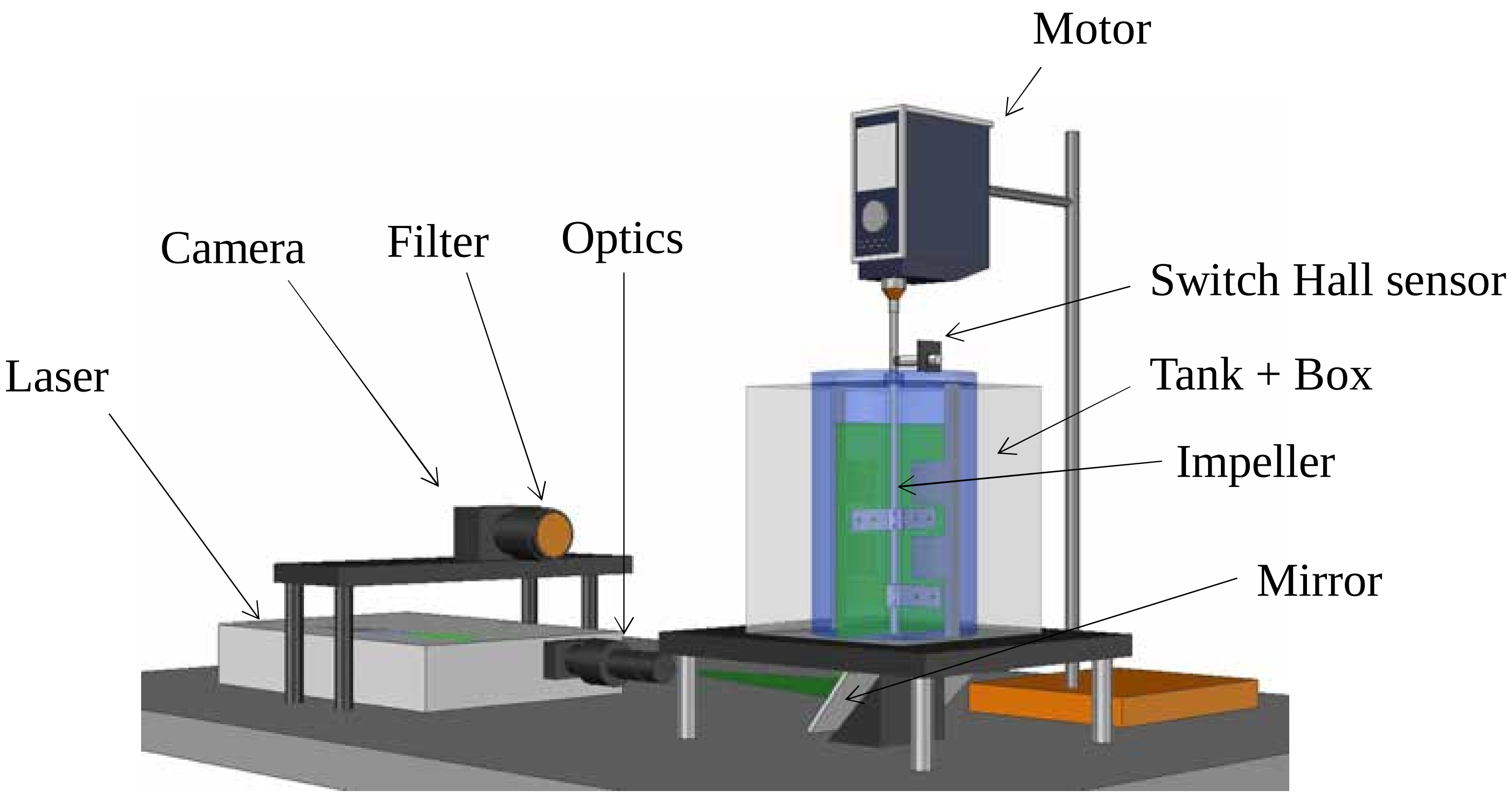


Impeller type used not effective

Vorticity profile and velocity vectors.
LHS glycerol 20 ºC 100 rpm
RHS gel 80% 60 ºC 100 rpm

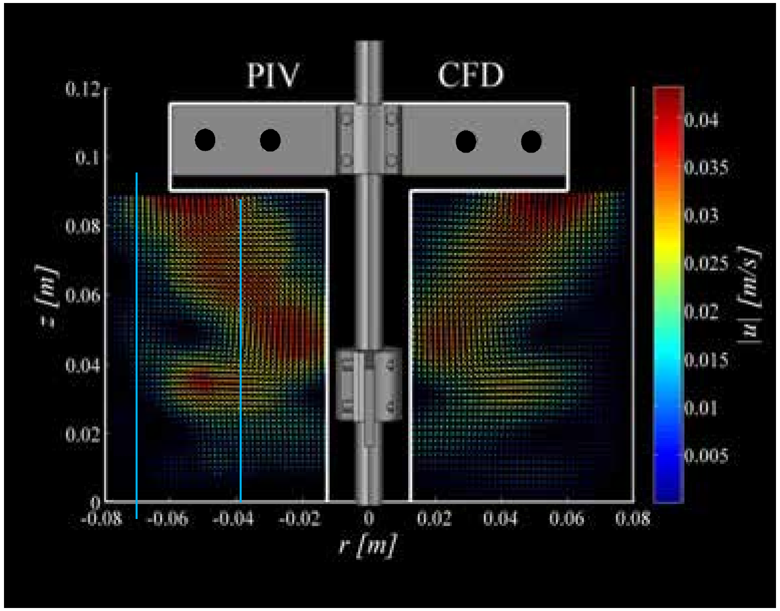
Characterisation of industrial mixer

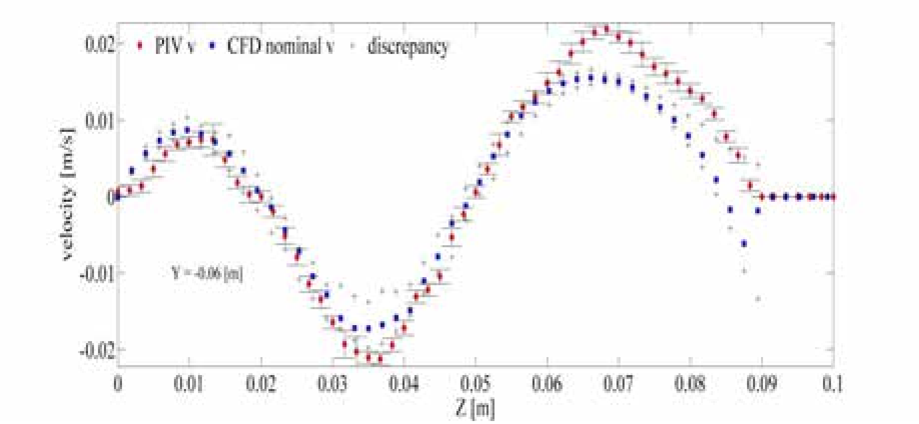
Improved impeller/baffles geometry to reflect industrial mixer and improve mixing

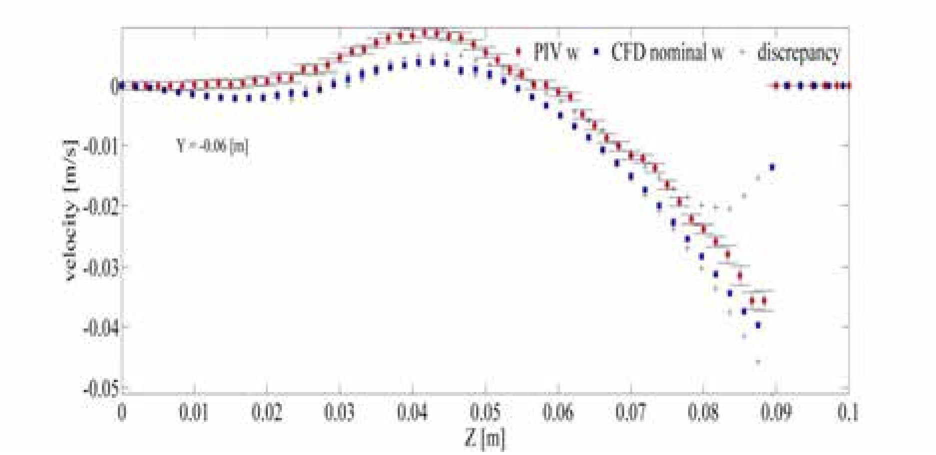


Characterisation of industrial mixer

Comparisons between experimental and CFD results



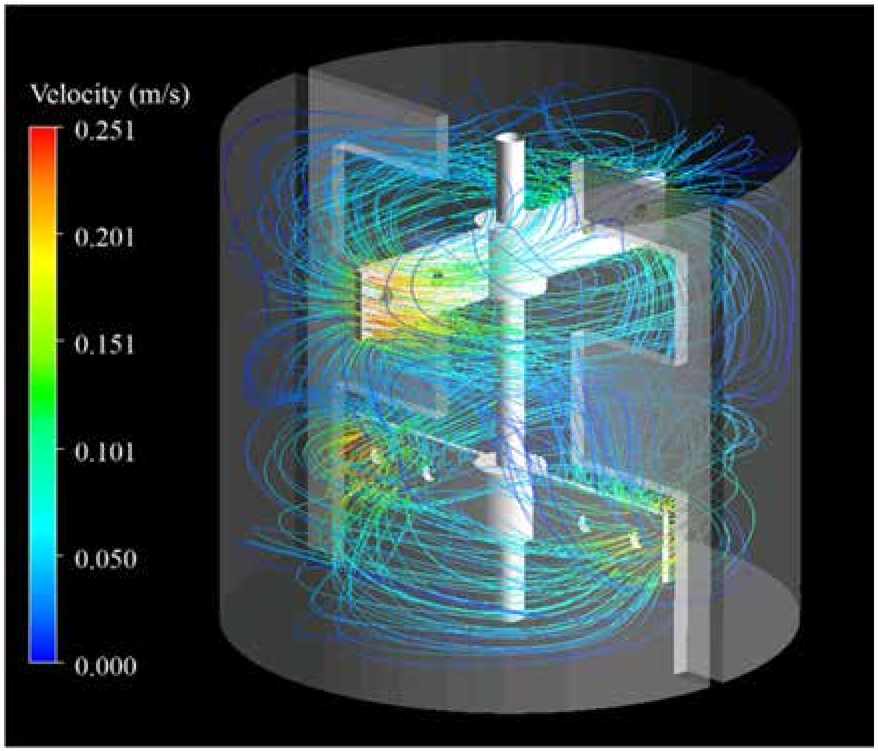


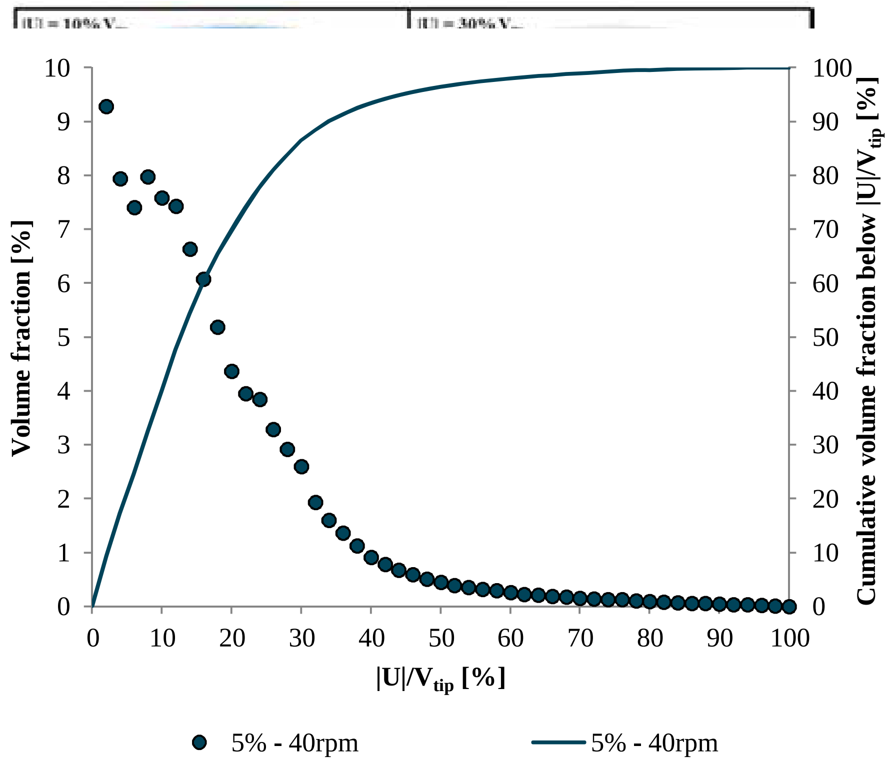


Gel mas fraction (%)	5
N (rpm)	40
Ti (ºC)	50
Te (ºC)	48
T avg (ºC)	49
Δt (ms)	15
Tip speed (m/s)	0.25

Characterisation of industrial mixer

Hydrodynamics in the stirred vessel

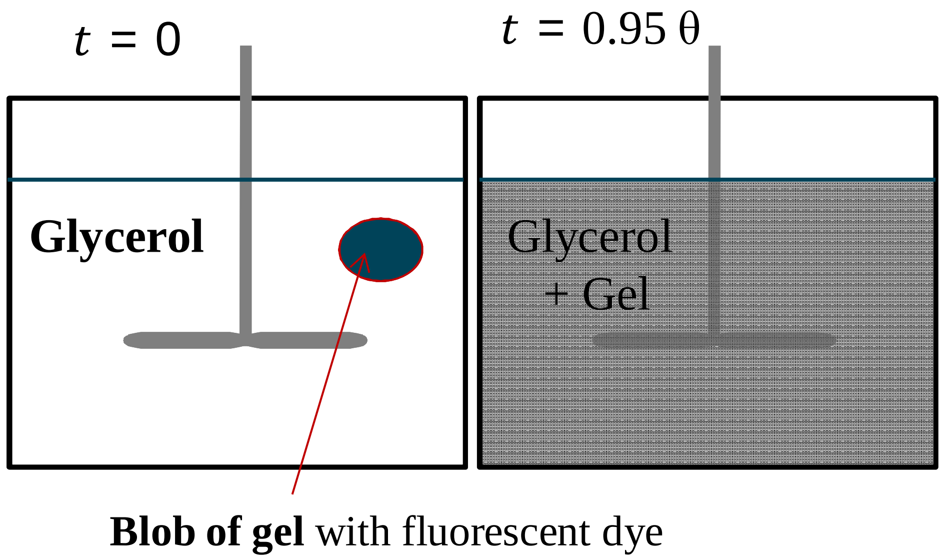




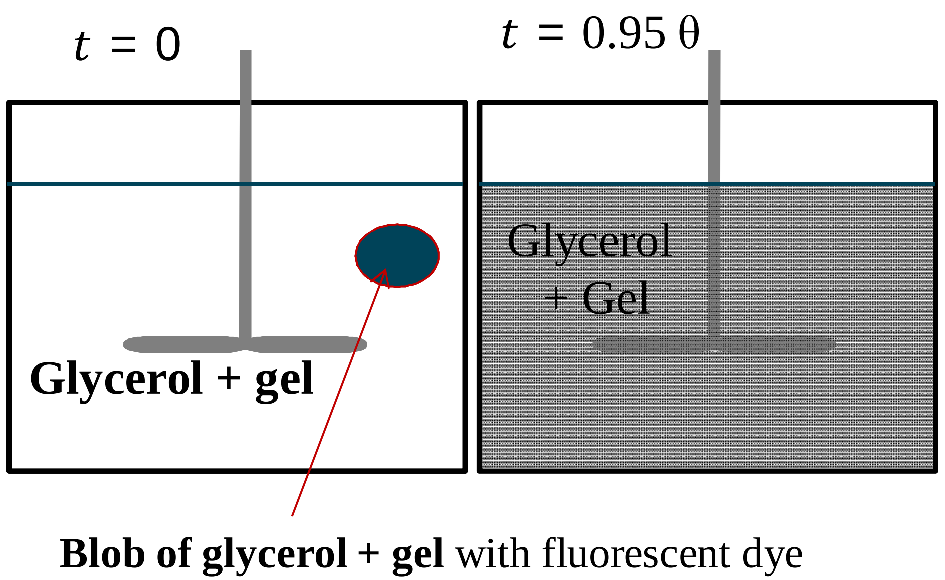
Characterisation of industrial mixer

Characterisation of mixing performance

Option 1

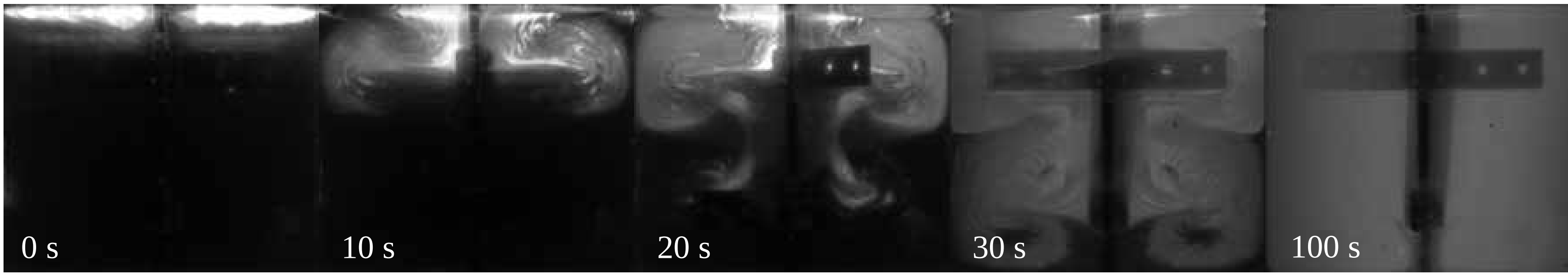


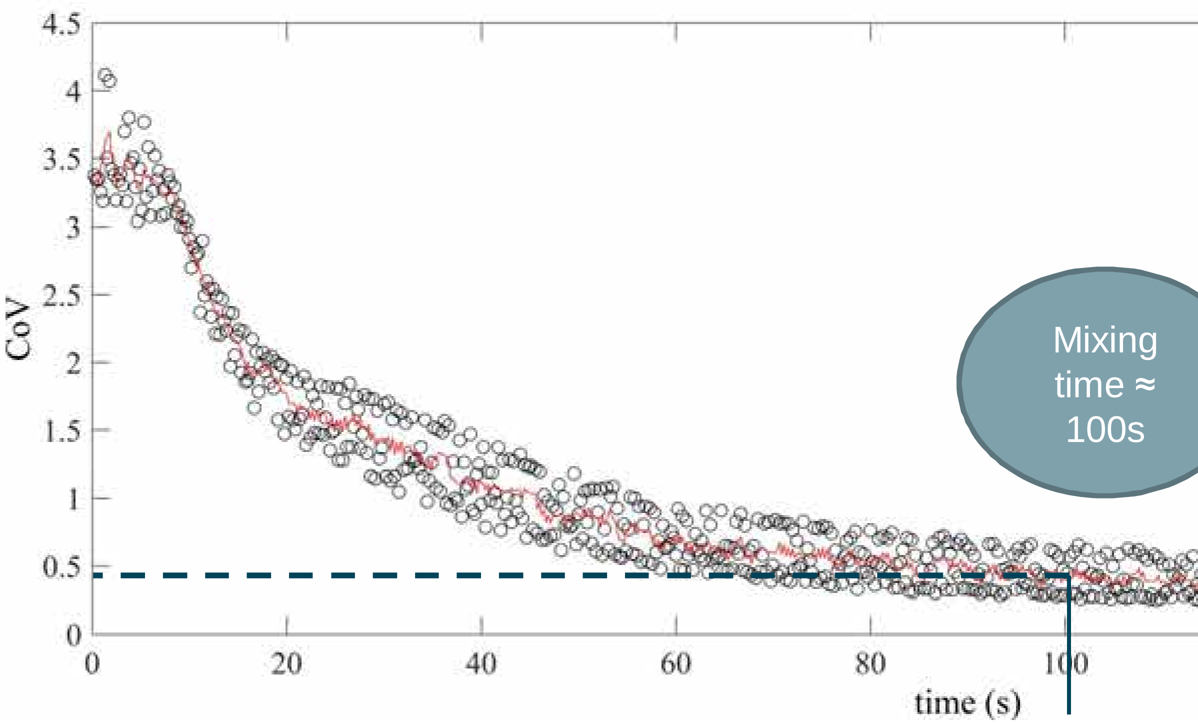
Option 2

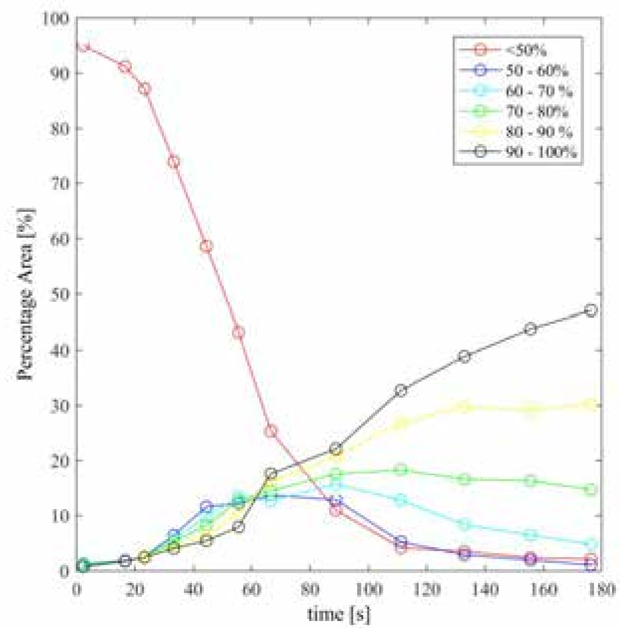


Characterisation of industrial mixer

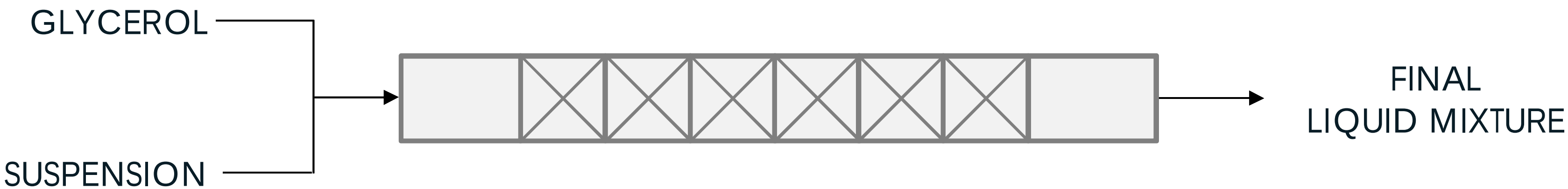
% gel	% glyc	T	Imp Speed	Re
0	100	60	60	18.9







A continuous approach



\+ Lower viscosity ratio
\+ Room temperature

- Unknow rheology of the final mixture
- Possible change of rheology during mixing

WHY STATIC MIXERS?

ü Approach plug flow

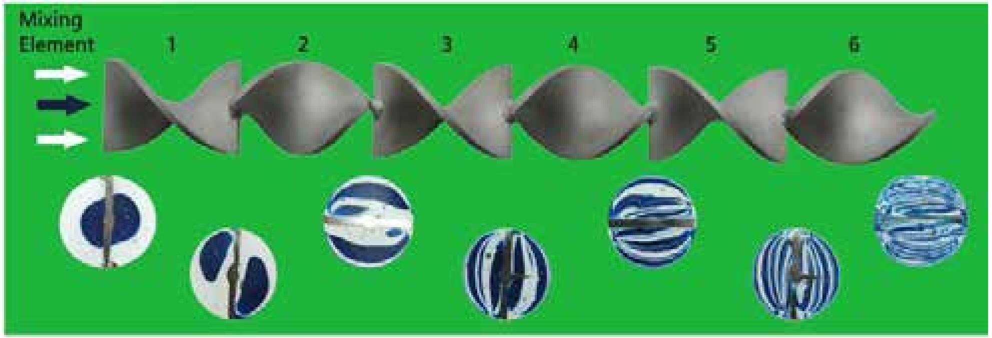
ü Good mixing at low shear rates

ü Short residence time

ü Small space required

ü Low maintenance, operative and equipment costs

WORKING PRINCIPLE



http://www.stamixco-usa.com/principles-of-operation

A continuous approach

Design steps

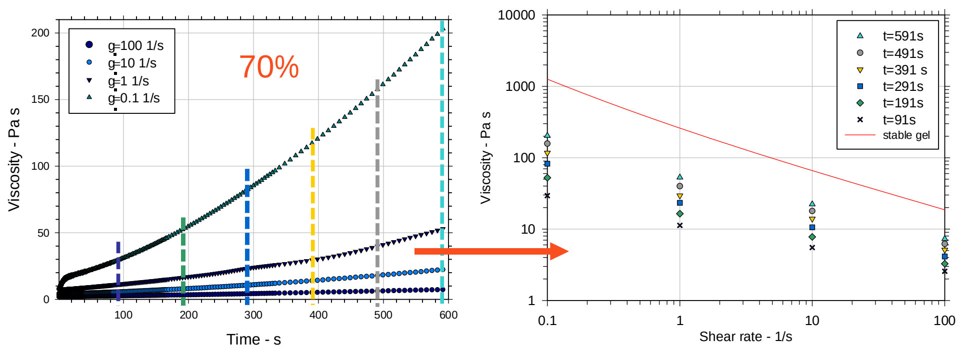
- PRESSURE DROP
- MIXING EFFICIENCY

CFD Modelling

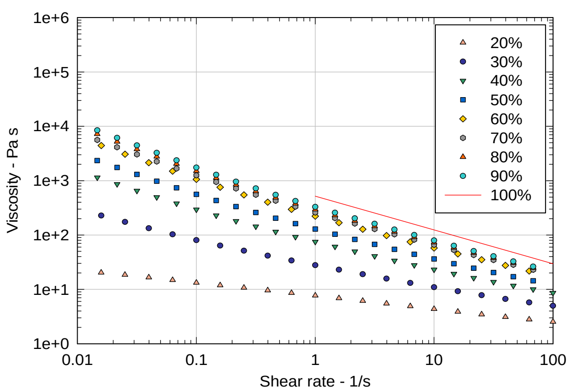
- v MIXER GEOMETRY
- v LENGTH
- v FLOW RATES

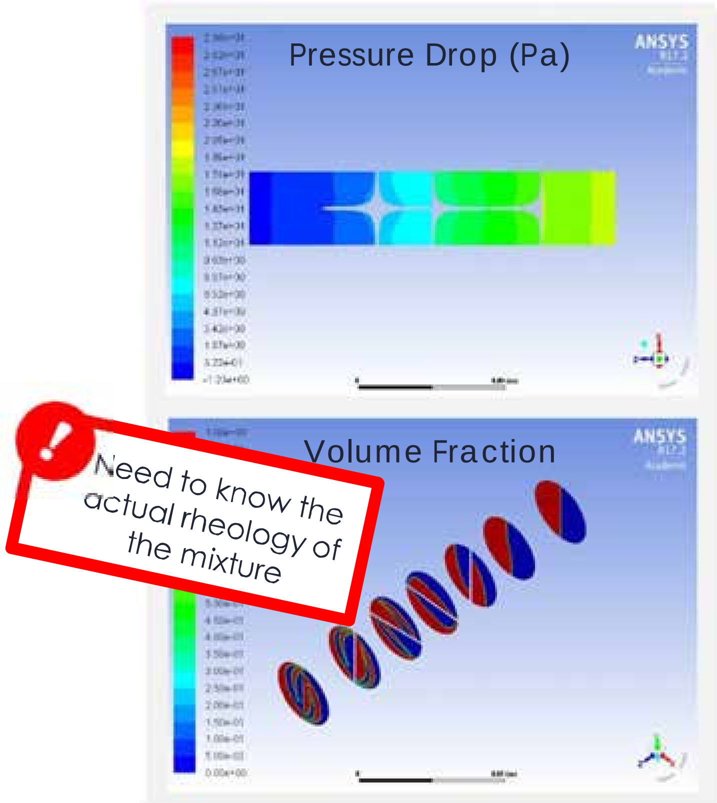
PRELIMINARY RHEOLOGICAL MEASUREMENTS

Glycerol/suspension mixtures



Final gel



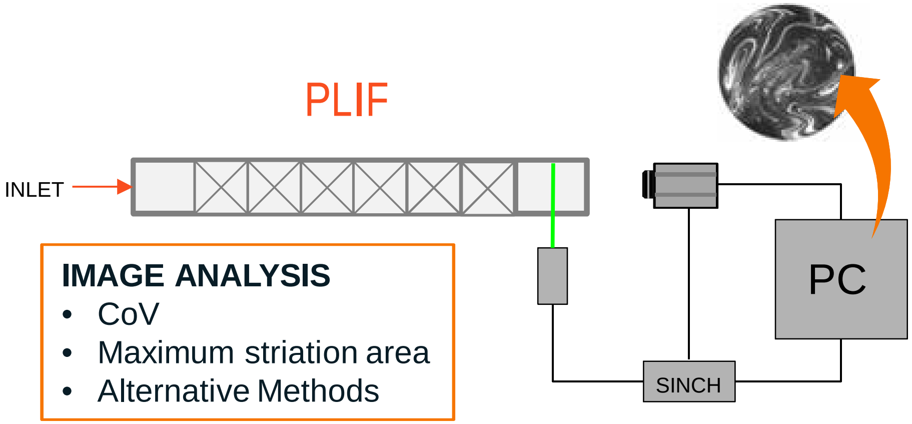


A continuous approach

§ **CFD simulations of the two-liquid flow system**

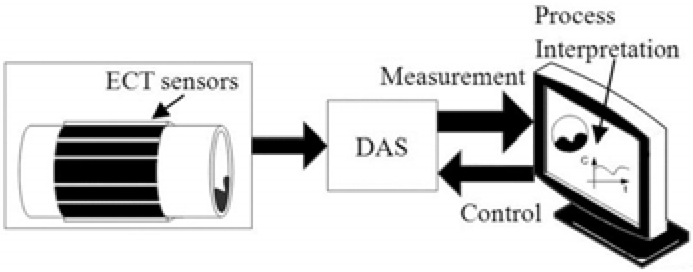
§ **Experimental campaign**

PLIF



Assessment of the final product quality

ECT (Electrical Capacitance Tomography)



Concentration gradients along the mixer

+ Non-intrusive technique
+ In-line concentration profiles
+ High image capture rate

- Need to design proper electrodes
- Need different permittivities for the two phases

Future steps

- Characterise rheology of mixtures
- Study mixing efficiency using known fluids
- Experimental studies on hydrodynamics and on mixing efficiency
- Computational fluid dynamics simulations
- Addition of solids

Acknowledgements

EPSRC Future Formulation – CORAL
GSK, Xaar, CPI

MEMPHIS project: http://www.memphis-multiphase.org/

ThAMeS Multiphase
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