Laboratory Protocol of PI

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Aims and Benefits of PI?

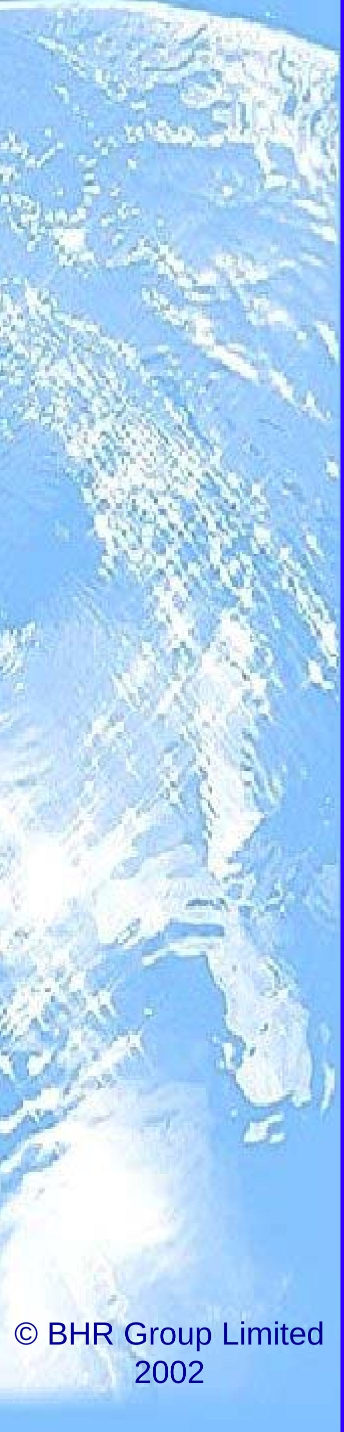
Provide equipment that enables the process chemistry to proceed at its optimum kinetic rate, with minimal by-products, resulting in plant which is: -

Safer, Smaller, Cleaner, Faster

Results in:-

- Higher product yield and less waste
- Smaller inherently safer plant
- Lower capital and running costs

Constraints of PI



Numerous constraints, restricting the use of Process intensification in industry:

- **Reaction kinetics**
- **Processing solids**
- **Process size (‘Biggest is best’ philosophy)**
- **Lack of awareness**
- **Limitations of product variability**
- **Uptake novel technologies**
- **Timescales**
- **Resistance to change**

PI Methodology

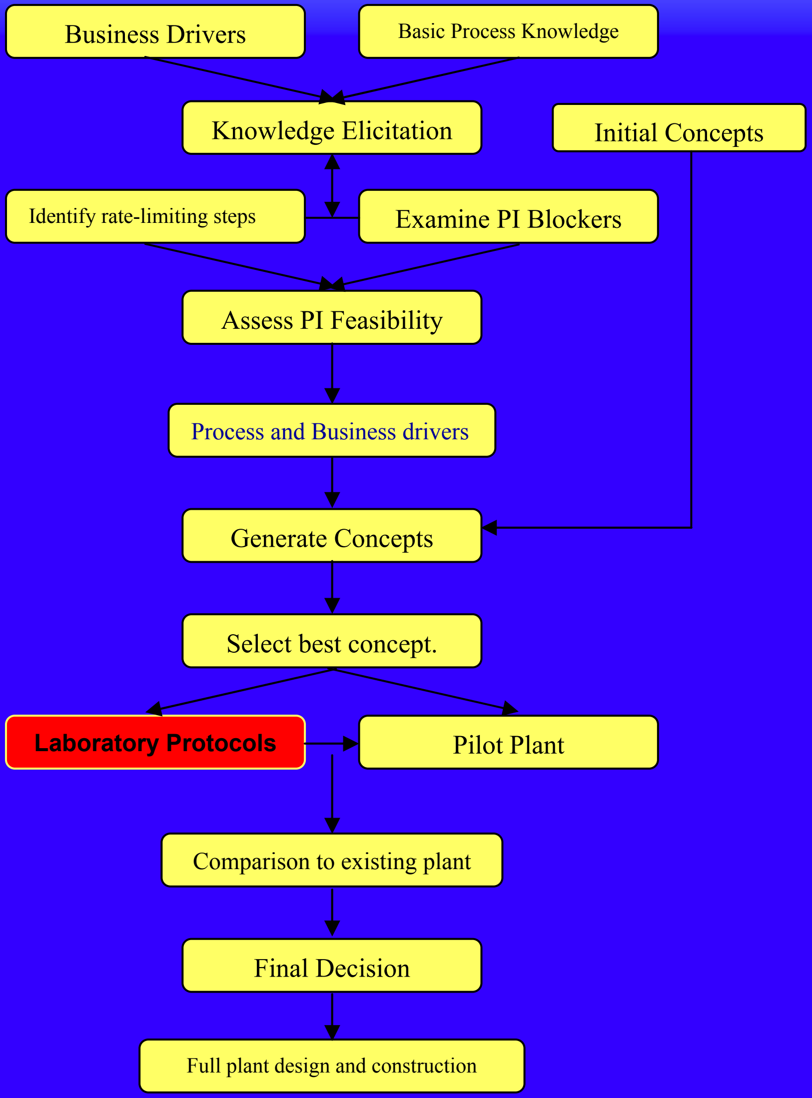
In an attempt to over come these constraints, BHR group developed a PI Methodology

This allows the suitability of process for intensification by assessing:

- Business Drivers
- Basic process knowledge
- Currently available or novel PI technology

Within the methodology was a ‘Laboratory Protocol’ section

PI Methodology



Business Drivers

Basic Process Knowledge

Knowledge Elicitation

Initial Concepts

Identify rate-limiting steps

Examine PI Blockers

Assess PI Feasibility

Process and Business drivers

Generate Concepts

Select best concept.

Laboratory Protocols

Pilot Plant

Comparison to existing plant

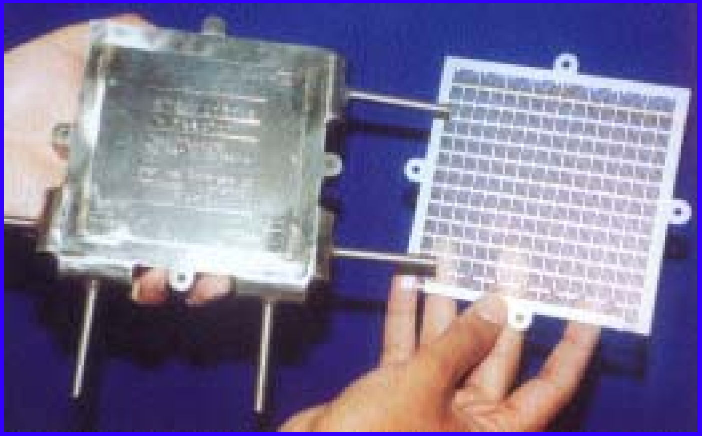
Final Decision

Full plant design and construction

Laboratory Protocol

PI is difficult to model within the laboratory as there exists an inability of small scale experiments to recreate the conditions experienced in a PI plant.

Design a small high intensity stirred tank reactor...



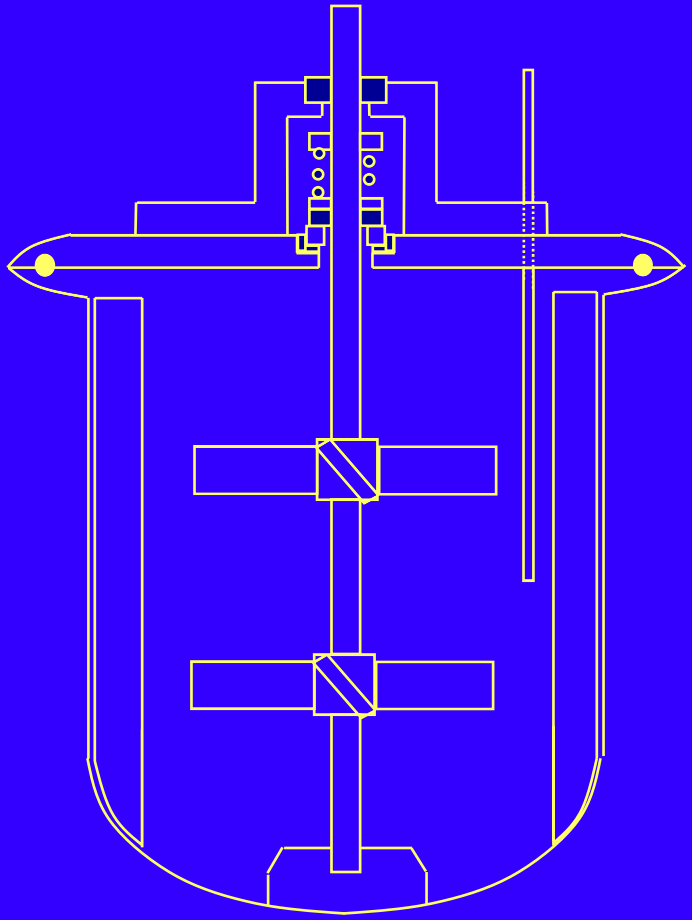
…to try and simulate and model intensified reactor systems

Design of the Vessel

Design considerations for construction of the protocol vessel: -

- **High intensity mixing**
- **Homogenous mixing**
- **Heat transfer capabilities.**
- **Simple to use**
- **Safe to use**
- **Well understood**

Protocol Vessel



Protocol vessel consists of:-

- **733ml glass vessel**
- **baffles**
- **Dual 45º PBT’s**
- **0- 4000 rpm Motor**
- **Mechanical shaft seal**

Why use Laboratory Protocol?

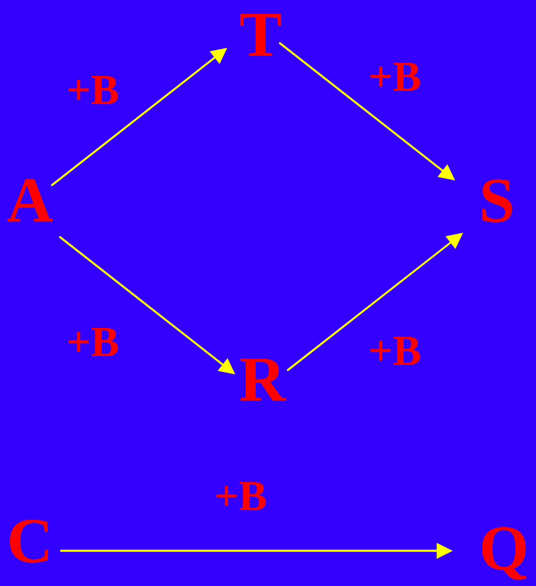
Industry needs to be shown that PI is a viable option for its processes but why use laboratory protocols, rather than demonstration rigs of intensified equipment?

- **Less process fluid requirement.**
- **Quick and Simple**
- **Uses ‘known’ equipment.**
- **No risk of fouling or blocking.**

Bourne Reaction Scheme

The mixing characteristics of the vessel were tested using the ‘mixing sensitive’ Bourne reaction scheme.

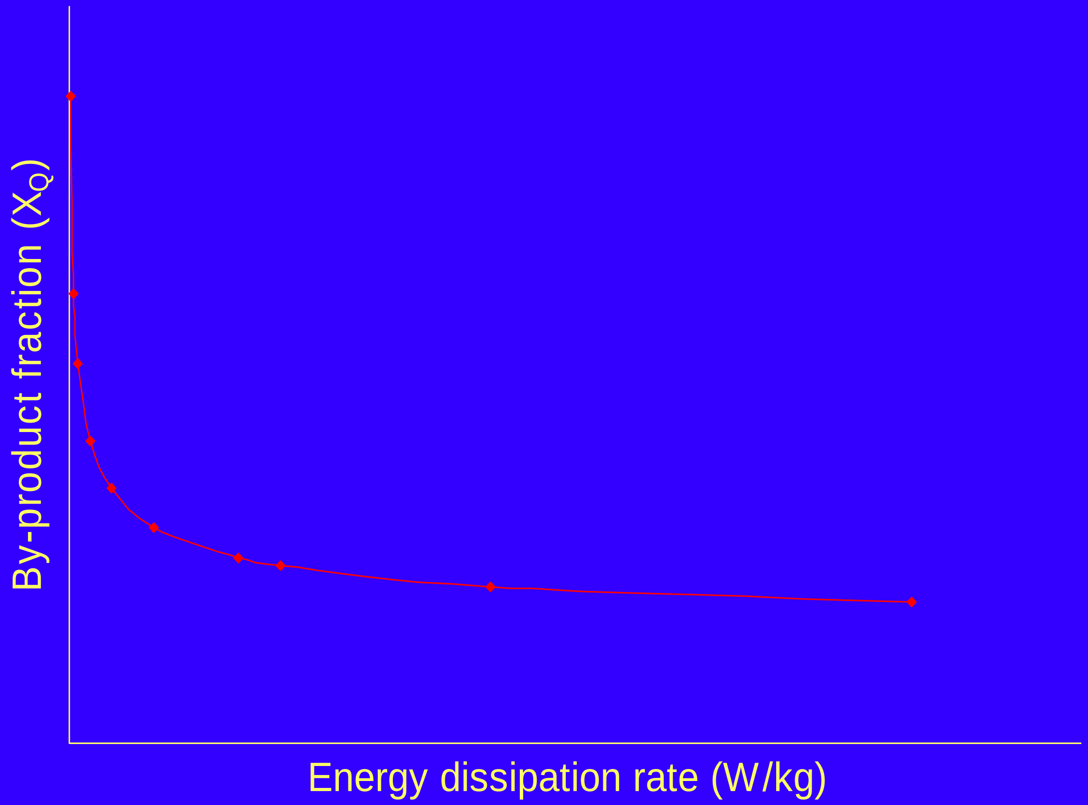
Involves mixing of a solution of 1-Naphthol (A) and 2- Naphthol (C) and diazotised sulphanilic acid (B)



- **If mixing is perfect, the faster reactions are favoured, so mostly R will be formed**
- **If mixing is not perfect, the faster reactions are mixing-limited and more by-products, S and Q, are formed.**

Bourne reaction scheme

The by product fraction, X_Q, is used as a measure of the degree of mixing.



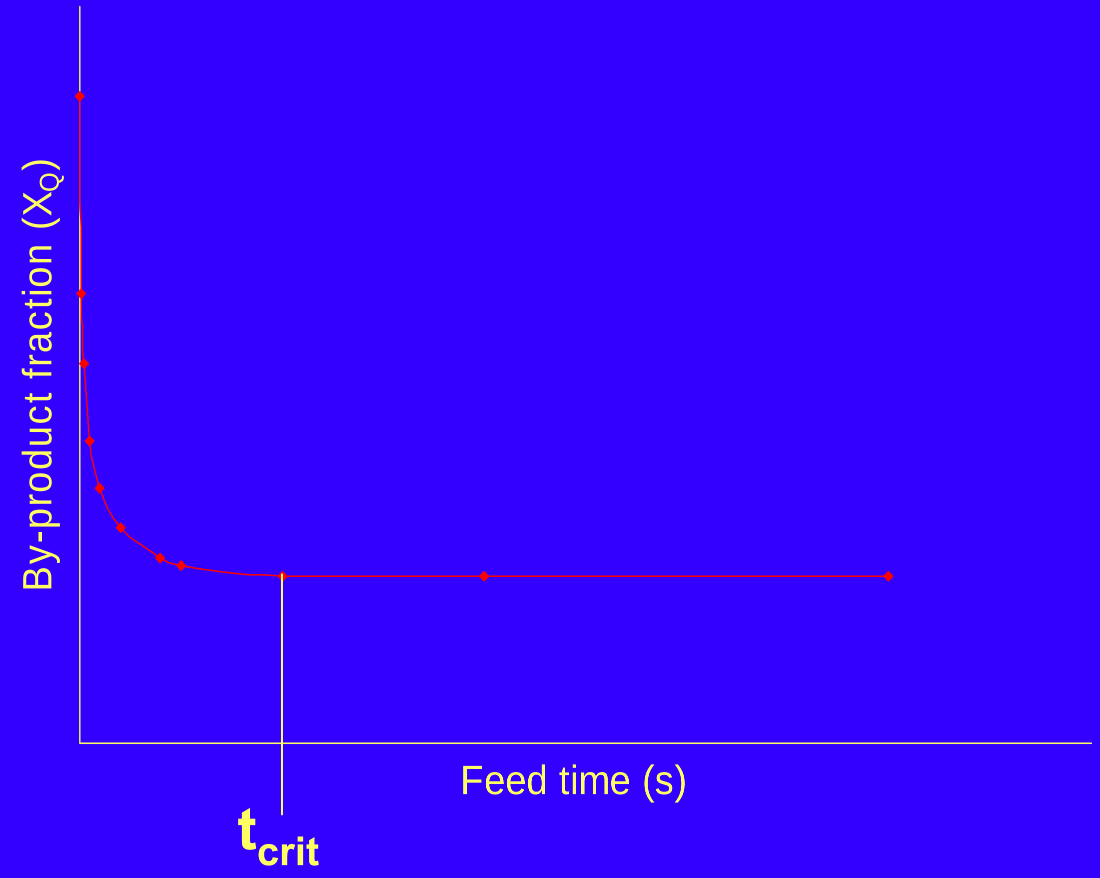
Characterisation

Protocol vessel was characterised: -

- **Power number determination for dual impeller.**
- **Heat transfer capabilities.**
- **Effect of impeller arrangement.**
- **Effect of impeller pumping direction.**
- **95% mix time determination.**
- **Local point energy dissipation rates (ε).**
- **Critical feed time analysis.**

Critical Feed time

Experimental determination of the critical Feed time:



Intensified Reactors

Why choose static mixers?

- Very common form of intensified plant/process.
- Used as both mixers and reactors.
- Well understood.
- Lot of data from HILINE experiments to model them with.

Static Mixers

What are they?

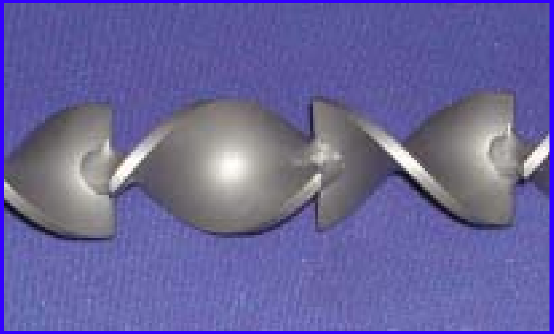
- Series of stationary guiding elements inside a pipe.

How do they work?

- Pumps delivering components supply the energy for mixing
- Stationary guiding elements in the pipe, split, divide and remix the fluid, at the expense of a pressure drop.

Static mixer simulation

Simulated four types of static mixer:



Kenics

SMXL

SMV

HEV

Process of simulation

- Bourne reaction scheme in a static mixer system.
- Calculation/determination of mixing characteristics of Static mixer system
- Determination of process variables required to match those calculated for static mixer system.
- Bourne reaction scheme in Protocol vessel at determined process variables.
- Comparison of by product fractions obtained

Simulating static mixers

Simulation involved matching one or several of the following characteristics:

- Energy dissipation rates (ε)
- Reynolds number (Re)
- Mixing timescales
    - Macromixing (τ_{macro})
    - Mesomixing (τ_{meso})
    - Micromixing (τ_{micro})

Simulating static mixers

Matching characteristics within the two systems:

	Static Mixer	STR
Energy dissipation rate	$\varepsilon_{AV} = f\left(\overline{u}^3\right)$	$\varepsilon_{AV} = f\left(N_i^3\right)$
Reynolds number	$Re_H = f(u)$	$Re_i = f(N_i)$

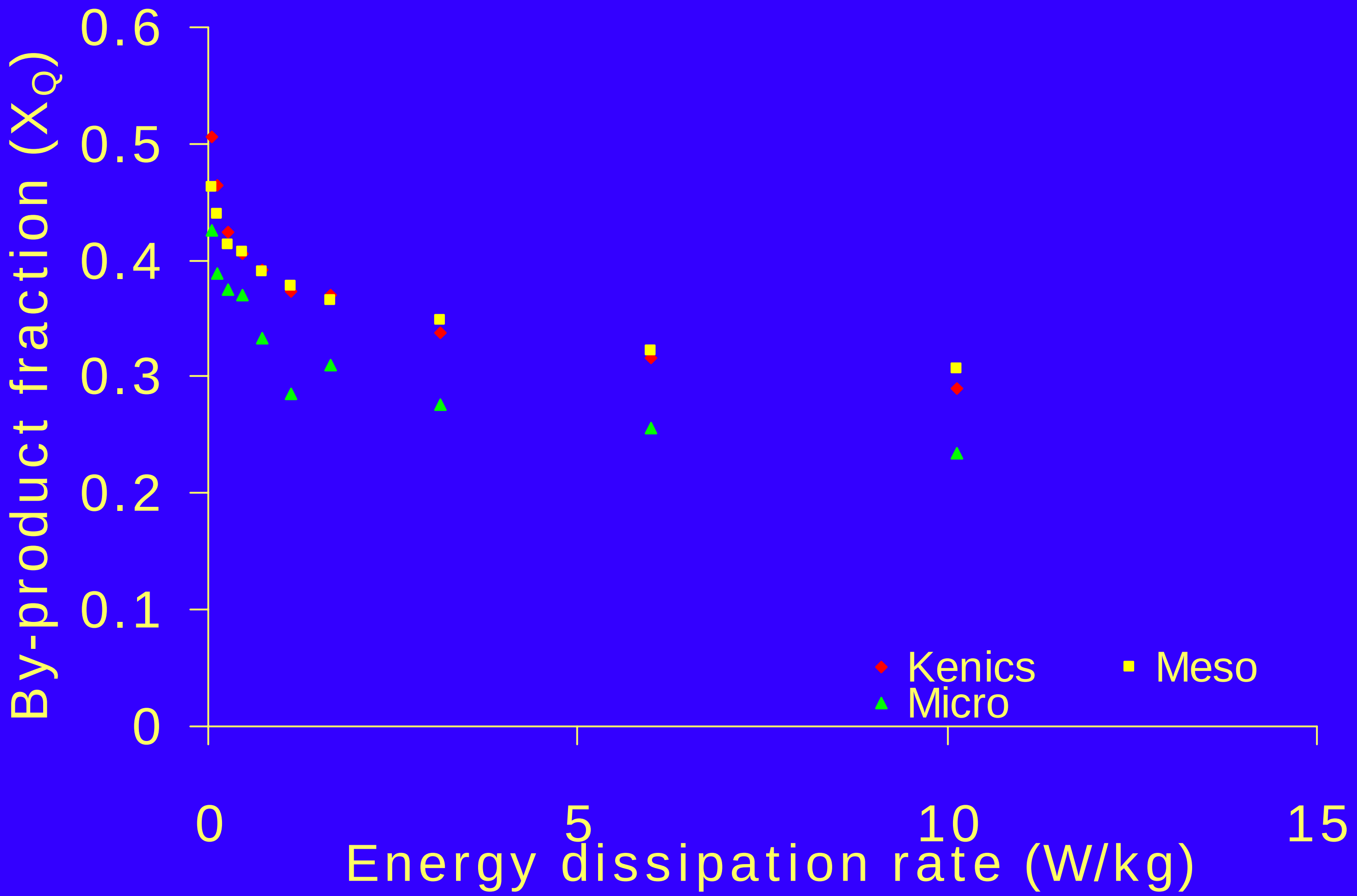
Simulating static mixers

Matching characteristics within the two systems:

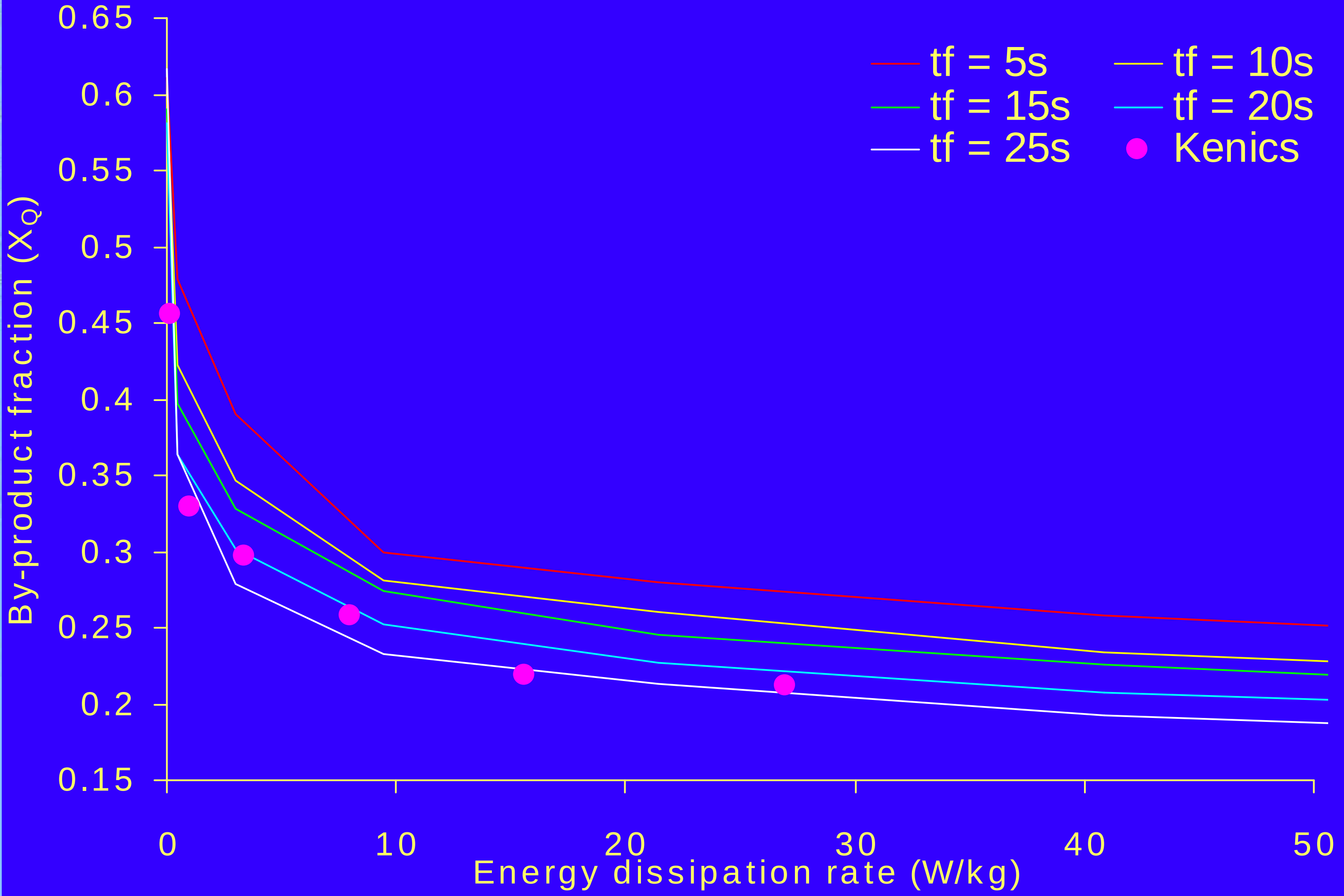
	Static Mixer	**STR**
Mesomixing timescale	$\tau_{meso} = f\left(Q_B \bar{u} \varepsilon\right)$	$\tau_{meso} = f(t_f, N_i)$
Micromixing timescale	$\tau_{micro} = f(\nu, \varepsilon)$	$\tau_{micro} = f(\nu, N_i)$

Initial Modelling

Initial modelling attempts:

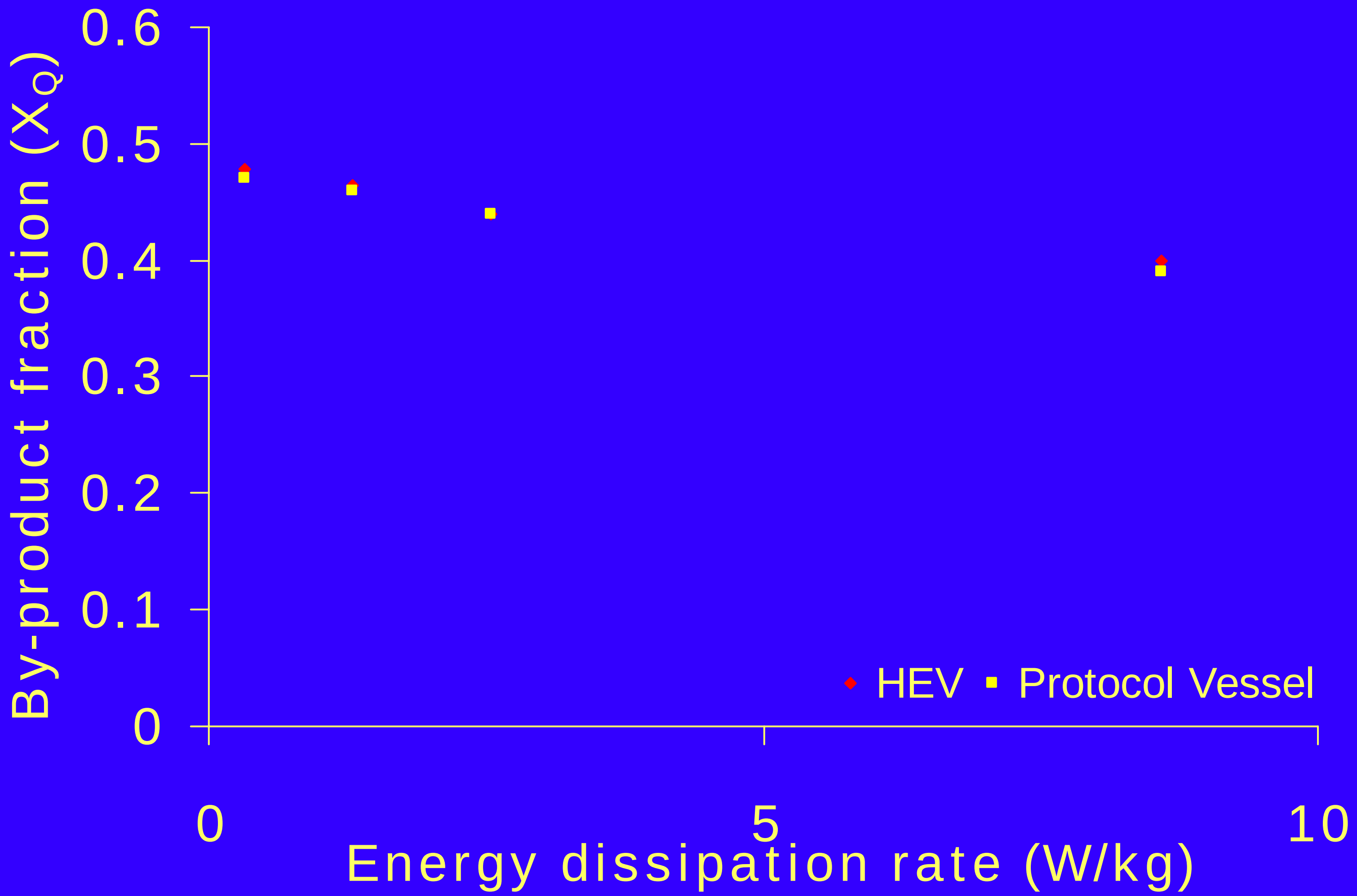


‘Matrix’ approach



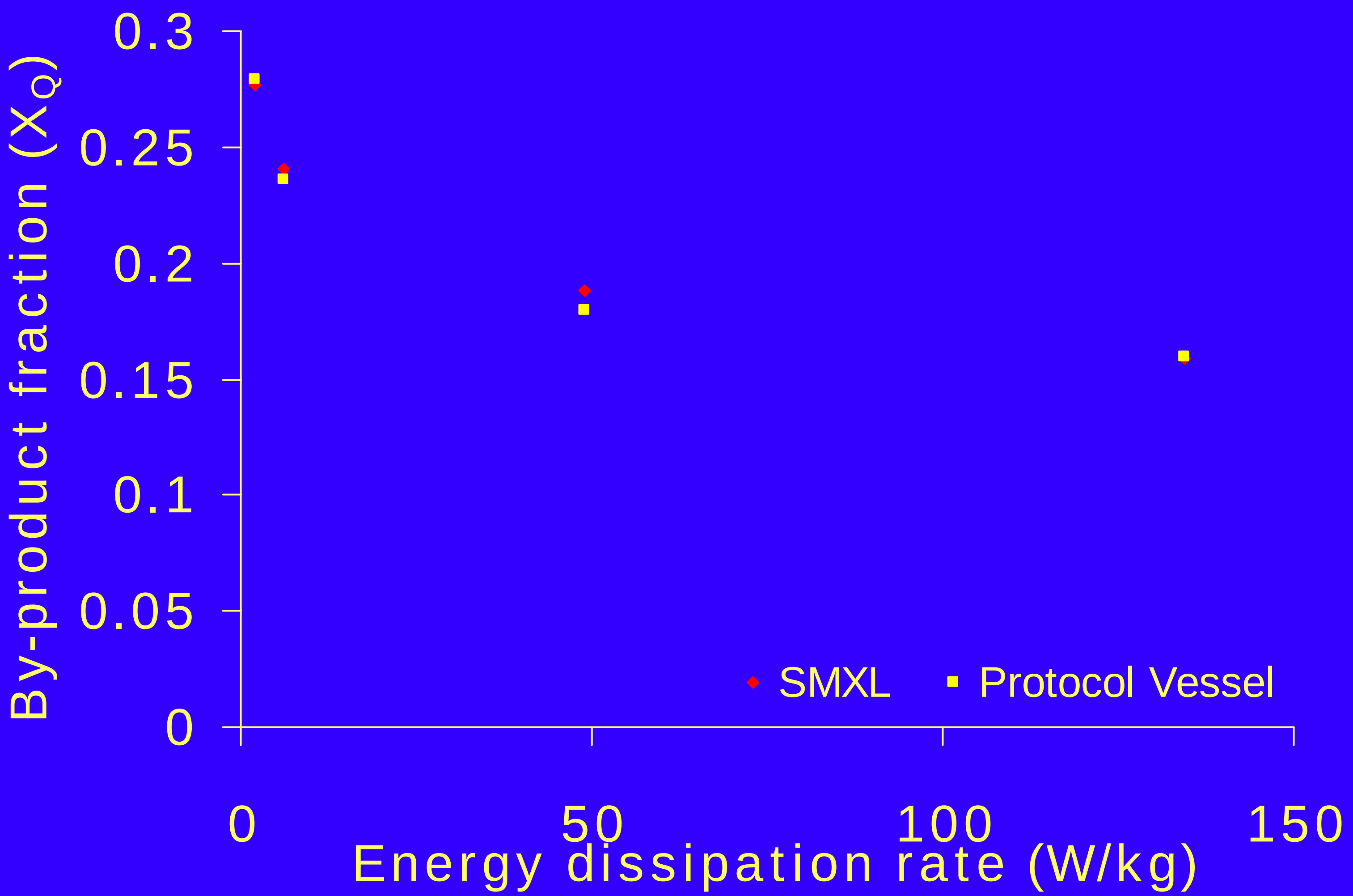
Static mixer simulation

HEV Static mixer - 7 Tab arrays



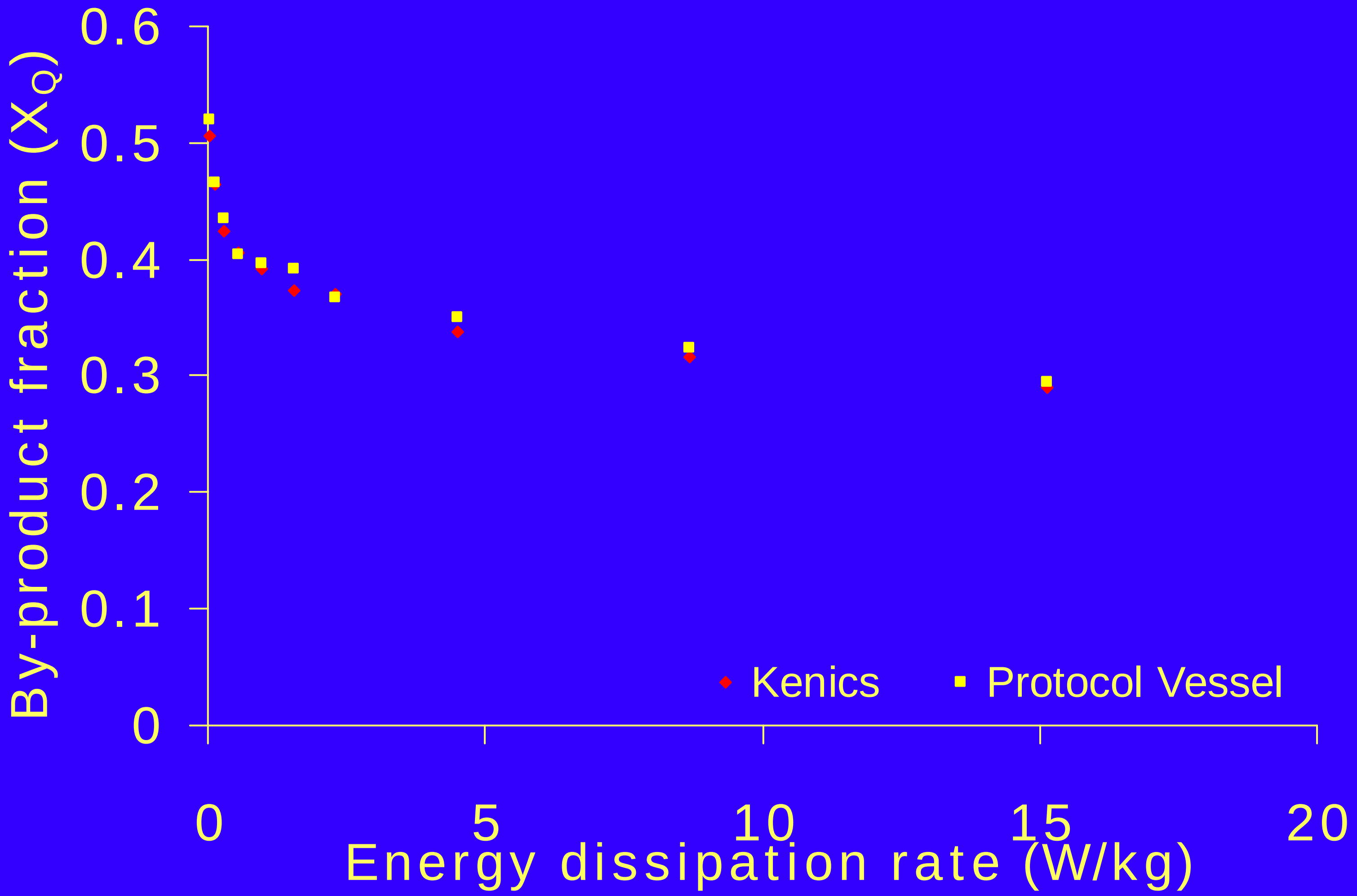
Static mixer simulation

SMXL Static mixer - 3 elements



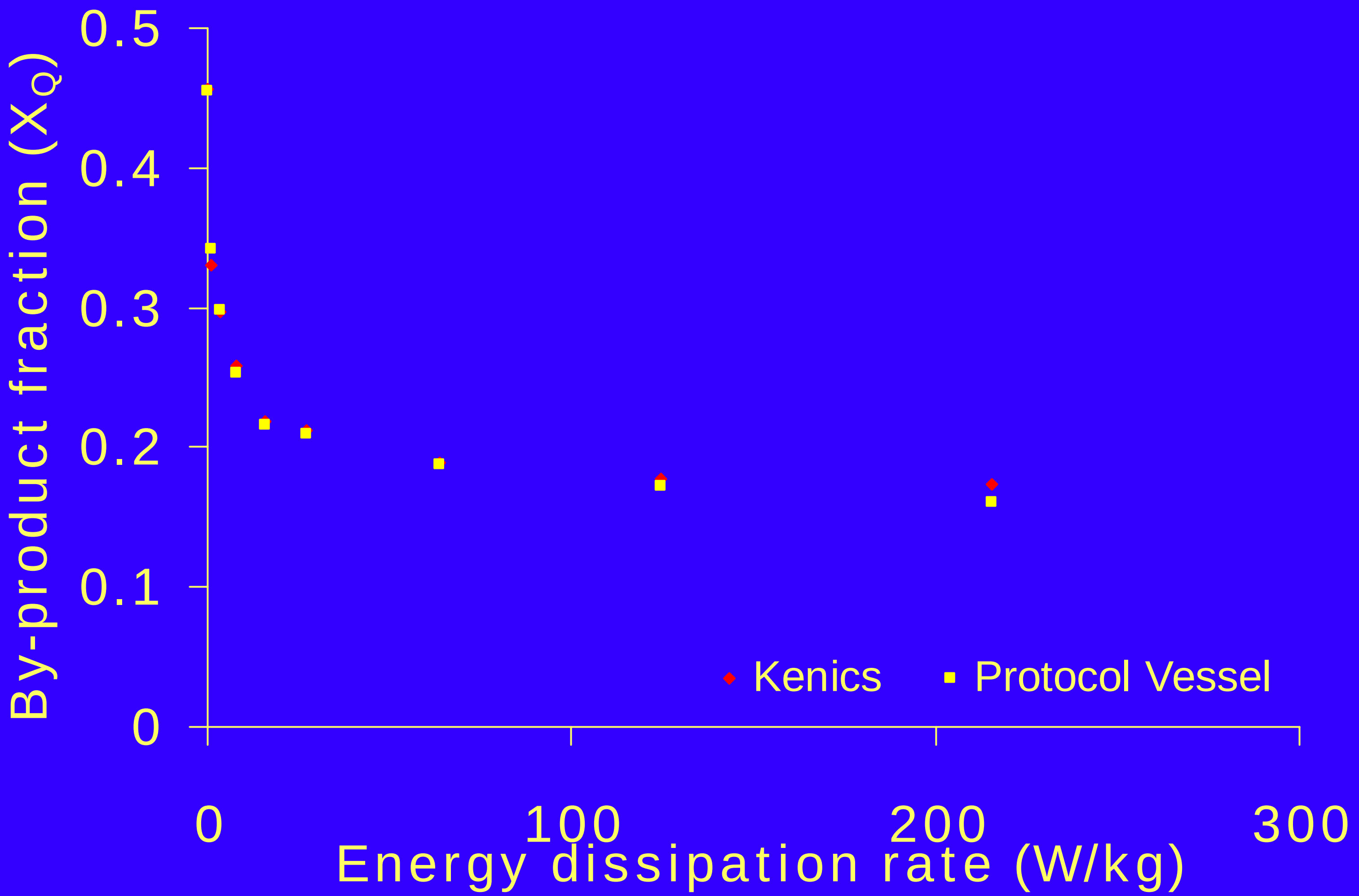
Static mixer simulation

Kenics Static mixer - 11 elements



Static mixer simulation

Kenics Static mixer - 9 elements



Conclusions

A reasonable match of the by-product fractions between the two systems can be obtained by:-

- **Setting Energy dissipation rate in the protocol vessel equal to that in the Static mixer system**
- **Estimation of a feed time from experimental matrix**

Future Work

- Determine correlations between the two systems.
- Construction of methodology
  - Allow selection of static mixer system from a series of bench scale STR tests
- Use of other reaction schemes
  - Single phase
  - Two phase
- Simulation of other continuous systems
  - HEX Reactors
  - spinning disks