NiTech® Pilot and Production Scale Equipment

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Contents

1. Principle of NiTech® scale up

NiTech® technology allows lab-scale (kinetic) reaction and crystallisation times to be achieved at production-scale. This is different to batch processing in stirred tanks, where the overall process time increases significantly with scale. Scaling up stirred tanks is also problematic because of heat and mass transfer constraints:

- Mass transfer constraints arise because mixing becomes less efficient at larger volumes leading to concentration gradients and poor interphase transfer.
- Heat transfer constraints arise because:
  - non-uniform mixing leads to thermal gradients which results in poor temperature control; and
  - surface area to volume ratio for heat transfer reduces dramatically at increasing scales – consequently, it takes longer to heat or cool fluid.

In addition, there has been no unanimously agreed rule or parameters for scaling up stirred tanks, meaning various parameters are often used, e.g.:

- impeller revolutions per minute, tip velocity or mean velocity
- averaged shear
- ratio of diameter to height
- stirred tank Reynolds number
- power density

NiTech® units have no heat and mass transfer constraints, plus have significantly increased specific surface to volume ratio; this gives predictable scalability. Geometric scale up is linear to maintain the flow velocity – weighted by solid/gas fraction – obtained from lab-scale work together with the required residence time (inherent kinetic time) and the production rate. Pressure drops for pilot and full scale units are also provided.

2. Literature on NiTech® scale up

The following literature papers are relevant to scale up.

Table 1 - Literature on NiTech® scale up

Title	Academic Paper PDF[1]	Work Carried Out	Key Conclusions
Scale-up correlation for mass transfer coefficients in pulsed baffled reactors by X. NI and S. Gao	Scale-up correlation for mass	Gas / liquid mass transfer scale-up using 50 and 100 mm inner diameter vessels.	Improved mass transfer in a larger diameter reactor is credited to: 1) The average bubble size was much smaller with a narrower size distribution, meaning an increase in interfacial area per cross-sectional area. 2) The gas residence times increased due to the oscillatory motion, more time for transfer. In addition, gas hold up was also significantly increased.
A Numerical Study on the Scale-up Behaviour in Oscillatory Baffled Columns by H. Jian and X. Ni	A numerical study on the scale up beh	3-D flow models of 50, 100 and 200 mm inner diameter vessels.	Scale-up is linear as the velocity ratios are largely independent of the column diameter. The results are in line with the experimental studies reported previously.
An Experimental Investigation into the Scale-up of Oscillatory Flow Mixing in Baffled Tubes by K. B. Smith and M. R. Mackely	An experimental investigation into th	Dispersion studies in 24, 54 and 150 mm vessels.	The technology can readily be scaled up from lab to pilot and to industrial scale, as lab experiments using <1 L can predict mixing in much larger vessels. Axial dispersion is not a function of tube diameter if key dimensionless groups are kept constant.

[1] Available on request

3. NiTech® scale up examples

The enhanced surface area to volume ratio for heat transfer and improved heat and mass transfer in NiTech® technology are the key elements in intensifying chemical and pharmaceutical processes. Example case studies are given in Table 2.

Table 2 - NiTech® scale up examples

Application	Size Indicators	Batch Equipment	NiTech® Continuous Equipment		
		Full scale	Lab scale	Pilot scale	Full scale
Liquid-Gas-Solid reaction	Reaction time	18 h [a]	9 min [b]	2.8 min [c]	1.7 min
	Equipment volume	Two 150 m^3 STRs	0.8 L	33 L	0.092 m^3
	Production capacity	40 L/min	0.09 L/min	12 L/min	54 L/min
Liquid-Liquid reaction	Reaction time	8 – 10 h [a]	4 min	4 min	*4 min* [d]
	Equipment volume	50 m^3 STR	2.5 L	40 L	*0.125 m^3* [d]
	Production capacity	32 L/min	0.6 L/min	10 L/min	*31 L/min* [d]

Notes:

a) Does not include the time to prepare and empty batches
b) 3 minutes to dissolve reactant gas and 6 minutes to complete the reaction
c) 85 seconds to dissolve reactant gas, and 85 seconds to complete the reaction
d) A production scale unit is being designed

4. NiTech® scale up hardware

Scaled up designs are bespoke but based on the “fish-bone” stand arrangement using jacketed pipes. Diameters are rounded to the nearest nominal size; the length is rounded up to give an equal number of straight sections. Baffles are not welded to the pipe-work, but instead a “baffle string” is inserted into each pipe length and bend. As with lab units, there is versatility in terms of location and number of sight glasses, injection points, and sampling / analytics. Oscillation is typical driven by positive displacement pumps operating in alternating directions (e.g. gear pumps). The pumped oscillator hardware consists of a positive displacement pumps connected to a vertical fluid column and the main COBC/R body (see Figure 1):

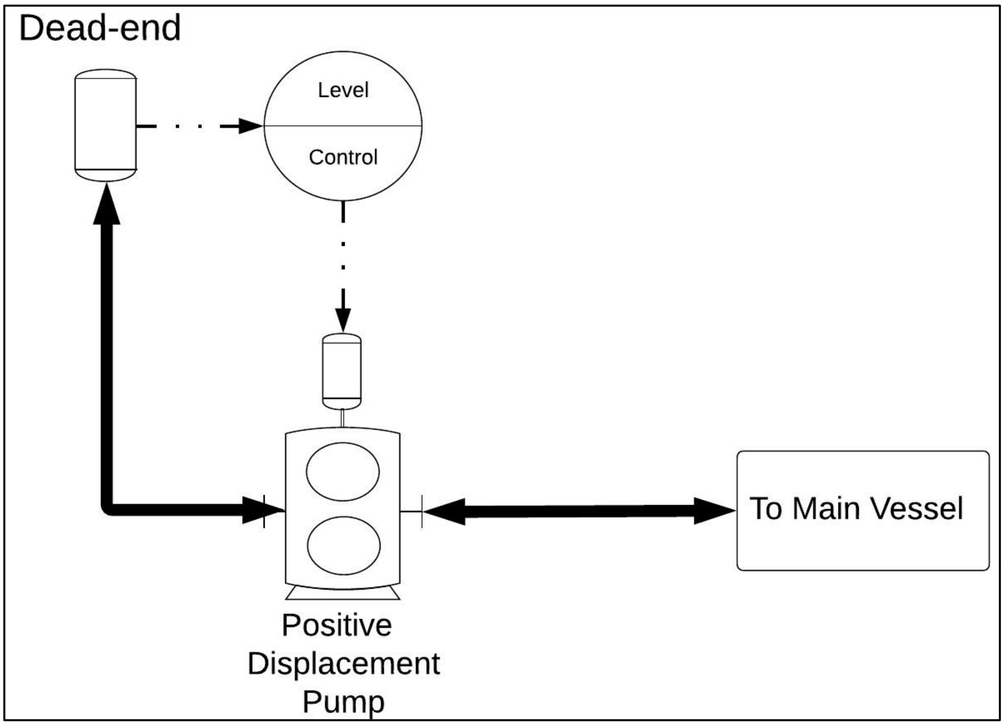

Figure 1 – Simplified Schematic of the Pilot and Production Scale Pumped Oscillator System

Positive displacement pumps (e.g. gear pumps) supply the oscillation, under variable discharge pressures, to give appropriate amplitudes in the main body. It is described as “fully-sealed” because the chosen pump is magnetically driven to remove dynamic seals, which would be a potential leak point. They change directions at a suitable frequency.

The vertical leg contains the fluid necessary to not dry run the pump on the forward stroke and to contain the liquid on the backward stroke. Level control is used to maintain the required liquid levels and net positive suction head (NPSH).

Reliability of this novel application of standard pumps was demonstrated in-house during development and has been used in previously built equipment.

a. An 11 L NiTech® Tubular Baffled Reactor

This unit is used for enhancing the mass transfer between a gas phase and a liquid phase; this step was identified as the rate limiting one. Tubular baffled reactors do not have an oscillator as intense mixing is provided by a slightly higher net flow (still under laminar flow condition) through a 15 mm inner diameter orifice baffled tube. Temperature control is provided in this case via electric heated jackets. It processes around 2.5 L/min of liquid and around 1 L/min of gas.

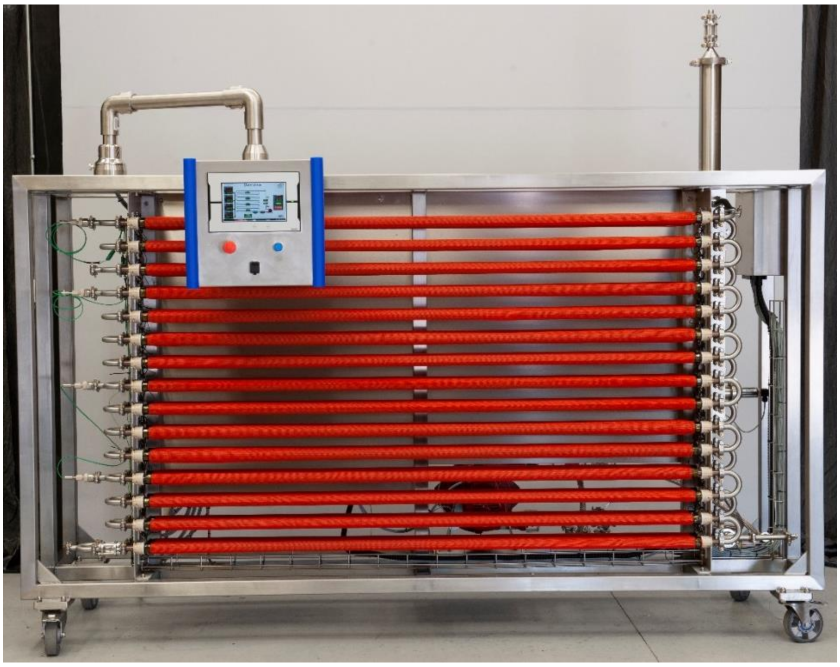

Figure 2 – The Front Side of a 11 L, 64 m long Stainless Steel NiTech® DN15 Tubular Baffled Reactor for Enhancing Gas-Liquid Mass Transfer, showing the Process Tubing with Electric Heating

Figure 3 - The Reverse Side of a 64 m long Stainless Steel NiTech® DN15 Tubular Baffled Reactor for Enhancing Gas-Liquid Mass Transfer, showing the Liquid Pump and Gas/Liquid Separator

b. A 20 L NiTech® Reactor

A 20 L, DN36, 20 m stainless steel, jacketed tubular-baffled system is shown below (Figure 4 and Figure 5) for a liquid-liquid reaction at a throughput of 10 L/min. It includes space for an extra 20 m and integrated feed pumps.

Figure 4 – A 20 L, 20 m long Stainless Steel DN36 NiTech® Continuous Oscillatory Baffled Reactor for a Liquid-Liquid Reaction

Figure 5 – A Close-up of the Tube Arrangement on a NiTech DN36

c. A 58 L NiTech Unit

A 58 L, 40 m long, DN43, 316L stainless steel, jacketed, baffled-tube network for a solid-liquid reaction at 1.5 L/min is shown below (Figure 6 and Figure 7), and annotated with key features:

- The Jacketed, Baffled Pipework = Red Square
- The Oscillator Pumps and Back-leg = Blue Ovals
- The Main Feed Point and Safety Pressure Sensor = Green Triangle (Reverse-side)

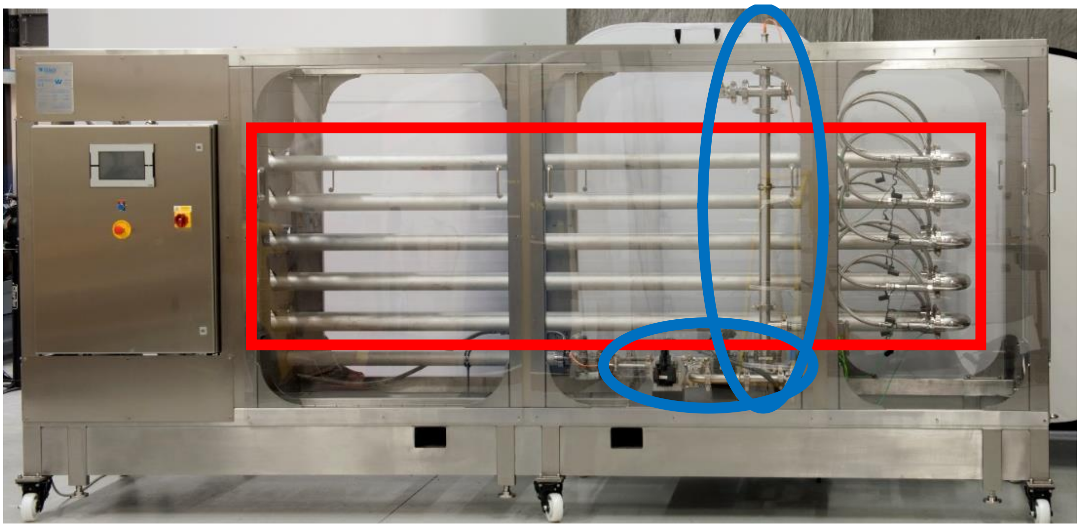

Figure 6 - The Front Side of a 40 m long Stainless Steel NiTech DN43 Continuous Oscillatory Baffled Reactor for a Precipitation Reaction

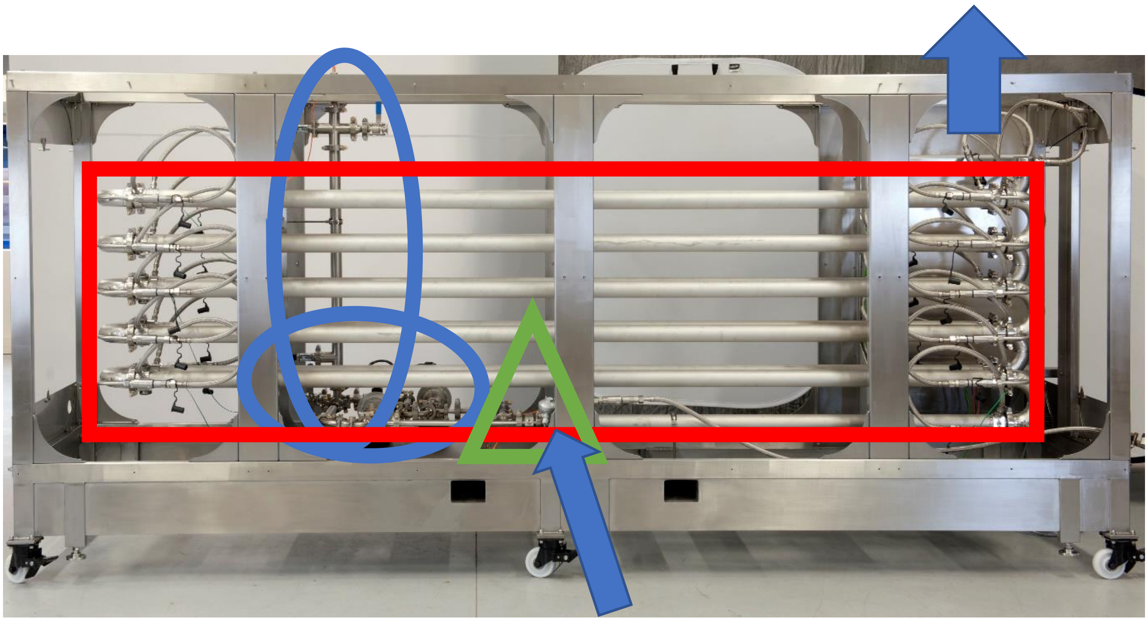

Figure 7 - The Back Side of a 40 m long Stainless Steel NiTech DN43 Continuous Oscillatory Baffled Reactor for a Precipitation Reaction with Annotations for Key Features

Figure 8 shows the oscillator pump protection loop to avoid contact between the pumps and the coarse solids being processed:

Figure 8 – Oscillator Protection Loop Installed on a NiTech DN43 COBR