

TITLE **A BAFFLING ROUTE TO SIMPLICITY**

D Wray Centre for Process Innovation

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Introduction

The chemical industry in the UK is under pressure from influences in the far east and other developing regions of the world where labour costs are lower. The use of novel processing routes should enable both capital and revenue costs to be reduced and increase process efficiency, helping to offset labour cost advantages enjoyed by these economies

The Centre for Process Innovation (CPI) began operation on 1st April 2004, one of its remits being to help the chemical industry in the UK become more competitive in world markets.

One project undertaken by CPI was the conversion of a traditional batch process using a stirred tank reactor (STR) to a continuous process using NiTech's Oscillatory Baffled Reactor (OBR™).

An OBR is a tubular reactor containing a series of baffles in the form of annuli; if a liquid is pulsed through the annuli in an OBR, eddies and vortices are formed above and below the annulus to give radial mixing characteristics far in excess of that observed in a conventional STR. Consequences of this enhanced mixing include:

Improved heat transfer

Fluid forces near the wall are as strong as those in the centre of the column, thus fluid renewal over the surfaces of an OBR is more frequent than in a STR, removing thermal gradients and ensuring more efficient heat transfer.

Improved mass transfer

As a result of the improved radial mixing, gas/liquid mass transfer coefficients can be increased by up to six times those encountered in conventional mixing. Gas residence time within the liquid phase is also increased considerably as the mixing properties enhance gas hold up.

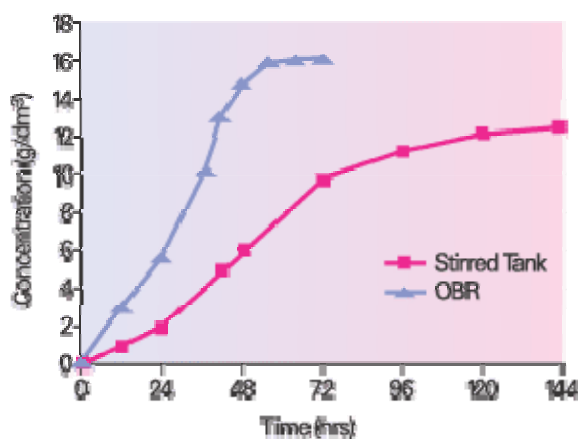


Figure 1: Comparison of Biopolymer fermentation products between an OBR and an STR

A further benefit is that bubble size decreases and becomes more uniform therefore increasing the interfacial area between the two phases, increasing mass transfer accordingly. Similarly in liquid/liquid systems droplet size can be controlled to deliver enlarged interfacial area. Figure 1 demonstrates that desired concentration levels of fermentation production could be achieved with an OBR in less than half of the time of the traditional process.

Plug Flow

Within the Oscillatory Baffled Reactor, conditions very near to plug flow can be attained even at low flow rates through the reactor tube, resulting in consistent product quality. The correct combination of oscillation amplitude and frequency creates a well-mixed regime and less waste. When a net flow is applied this well mixed plug moves along the length of the reactor with minute axial spread. The near plug flow is achieved at less than a tenth of the flow of connected tubular reactors.(see visual plug flow at www.nitechsolutions.co.uk/solid_sus_dia.htm) Figure 2 shows the progression of a slug of KNO_2 along an OBR. It can be seen that as the area under the peaks is constant, the KNO_2 moves down the reactor without significant axial spread, thus approximating to plug flow.

Probe 1

3.7 meters away from injection

Probe 2

7.9 meters away from injection

Probe 3

10.1 meters away from injection

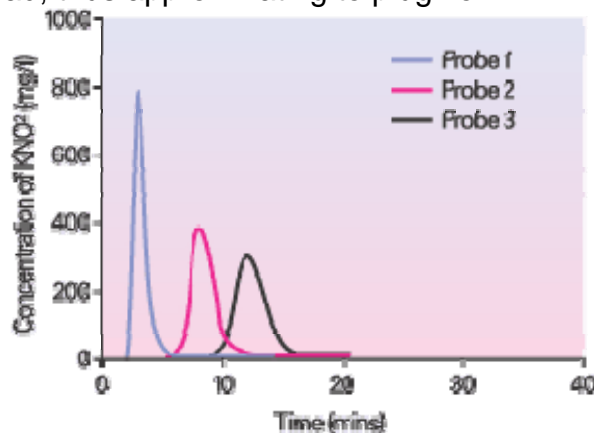


Figure 2: Residence Time Distribution Measurement in an OBR

Potential Commercial Benefits of the Oscillatory Baffled Reactor

The OBR naturally has a low inventory: this minimises material charges for a particular production run, and lowers material costs, significantly less material loss occurs in clean down and changeover of equipment. Also, the continuous process ensures that greater control over reactions is achieved, resulting in less out-of-spec product and less by-products, which in turn lead to less waste management and production costs as well as lower overheads. Compared to traditional batch processes greater plant uptime is also achieved. Continuous production also leads to savings in utility costs and space requirements, again

lowering general overheads. Production is geared to customer demand and not the size of a STR, thus reducing levels of product inventory.

Safety, Health and Environmental considerations.

There are several factors which contribute to better Safety Health and Environmental performance using an OBR:

From a safety point of view, the OBR can be considered as being a small pipe with liquid slowly passing through, consequently there is never more than a few litres of reactants at temp and pressure, compared to a STR which usually, contain large amounts of reactants and solvents at elevated temperature to achieve equivalent production rates.

Environmentally, the enhanced mass and heat transfer characteristics of the OBR enable reactions to proceed in a greatly reduced volume of carrier solvent, axiomatically reducing waste and disposal costs, concomitant with savings in energy for heating and cooling, .

As previously stated the OBR is essentially a pipe wrapped in another pipe (for exotherm control) giving rise to an estimated savings on Capex of 50%. With the ease of operation Opex savings are in the region of 70% .

THE OBR PROJECT

OBJECTIVES

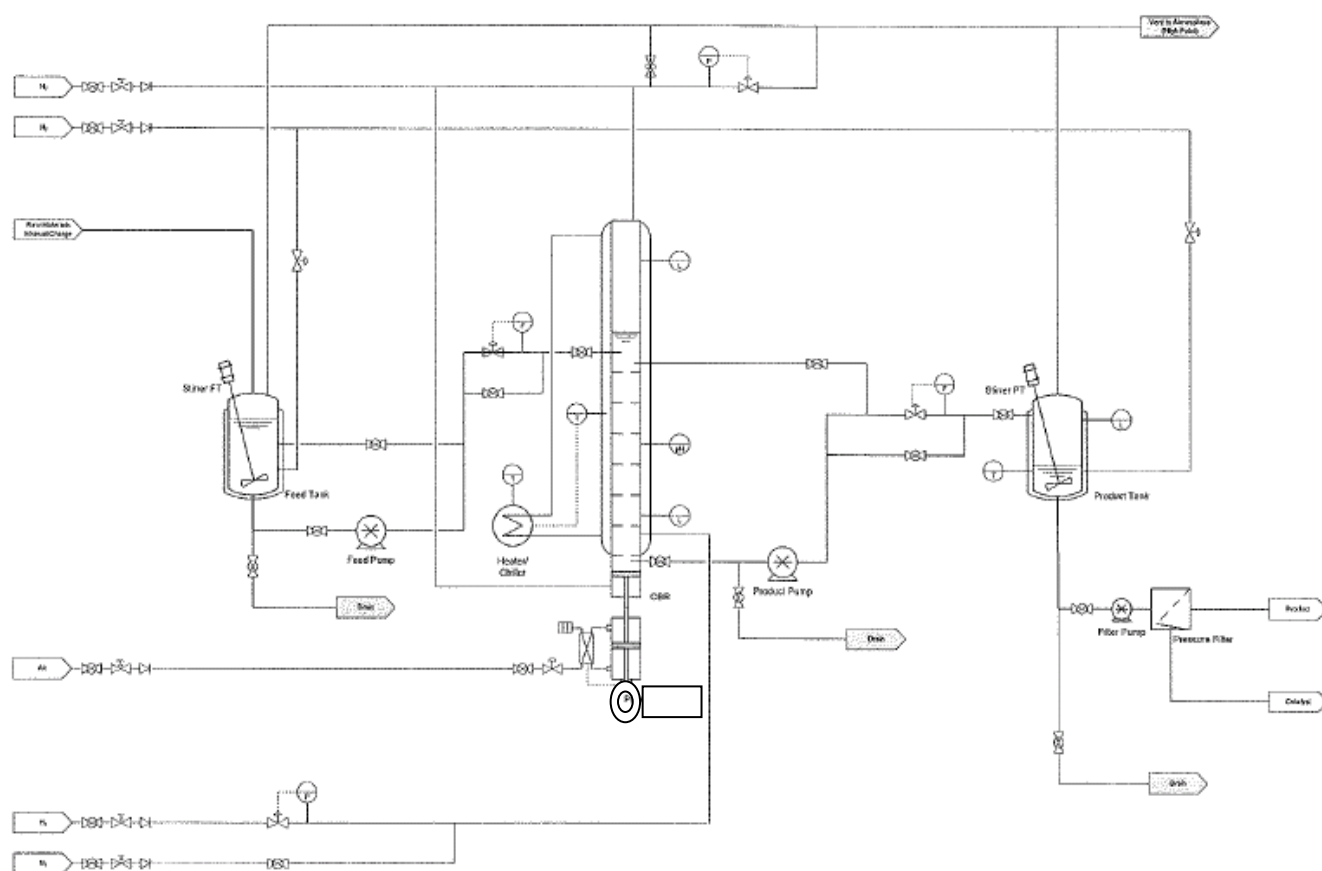
- To design, build and operate an OBR capable of producing production quantities of product.
- To perform three phase hydrogenation and oxidation reactions.
- To incorporate an intrinsically safe design.

To achieve these objectives a consortium was formed between; NiTech solutions Ltd, James Robinson Ltd and Oxford Chemicals Ltd. NiTech Solutions Ltd, a spin off company from Heriot-Watt University, are the owners of the process IP and provided the license and design package for the reactor. James Robinson Ltd of Huddersfield have been involved in the production of photo chemicals and dyestuffs for over a century, and Oxford Chemicals Ltd, have been manufacturing aroma chemicals at their Hartlepool site for the last fifteen years.

The plant was constructed by Haycock and Hague of Selby, who delivered a mechanical oscillating unit compatible with the use of Hydrogen. The consortium was driven by the team at The Centre for Process Innovation resulting in the design, build and installation of a bespoke OBR. The rig was specifically designed to perform both two and three phase catalytic reactions from atmospheric pressure up to 18bar. This process involved taking the technology

from a university concept to designing a plant capable of producing up to 500Kg of product per week. The rig also included several facilities to accommodate flow rates up to 6 litres per hour of feedstock. Once commissioned, the first consortium member to use the reactor was James Robinson Ltd (JRL), who trialled a reaction which entailed the three phase catalytic reduction of an aromatic nitroso compound to an amine in continuous mode. Below is an outline of the operation and initial trials carried out by JRL;

SCHEMATIC OF OBR



OPERATION

Before commencing processing, the feed vessel, reactor and receiver were purged with Nitrogen. The solvent, aromatic nitroso and catalyst was charged to

the feed vessel. This feedstock was then homogenised using a high shear mixer then pumped into the reactor until the high level indicator was reached, this represented the “batch”. The oscillator was started and the reactor was then pressured upto 2.5 bar using Nitrogen. The batch was then circulated around the reactor, the batch heated to 50C and the heating/ cooling switched to automatic mode. Hydrogen was then introduced to the reactor, and the reaction monitored until the batch was fully hydrogenated, the recirculation pump was then stopped. A typical hydrogenation time was 75 minutes; results are shown in Table 1 below. These results are represented graphically in Figure 3.

Once batch hydrogenation was complete the reactor was switched to continuous mode, the reactor was allowed to elute to the low level control via a screening filter to remove catalyst, into the product receiver. Fresh feedstock was then pumped into the reactor to the high level, allowed to hydrogenate, (8 minutes) then eluted via the filter to the product receiver, thus completing one cycle in continuous mode.

Table 2 below Shows typical results obtained in continuous mode at 50C and a Hydrogen flow rate of 1 ltr/minute, producing high quality product consistent in both assay and purity.

OBR PROJECT RESULTS

HPLC Data for batch hydrogenation of aromatic nitroso compound



Table 1: Kinetic Study showing batch conversion of Nitroso to Amine vs time

Time/minutes	ArNH ₂ (%)	ArNO/ (%)	
15	35.2	64.8	
30	59.8	40.2	
45	77.8	22.2	
60	91.7	8.3	
75 (batch complete)	99.6	Not detected	

Data for continuous hydrogenation

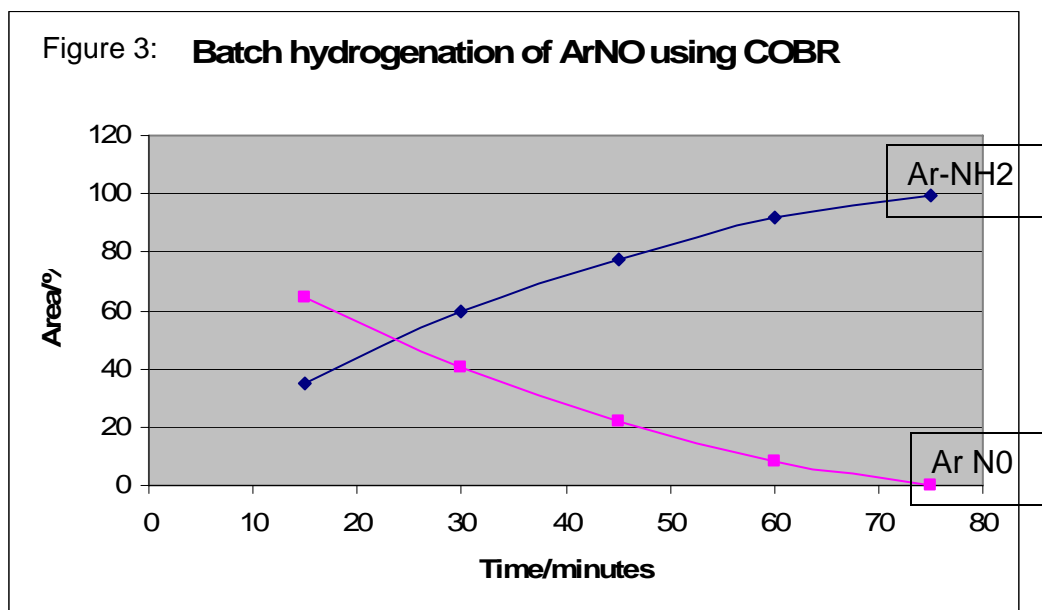
Table 2: Typical results for continuous hydrogenation of an aromatic nitroso to amine

Run Number	1	2	3	4	5	6	7
Time in continuous mode/minutes	-	360	240	180	240	160	120
Number of elutions	-	12	12	12	12	8	12

Weight solution/g	screened	-	5288	4783	5120	4817	3129	3532
Weight returned/g	product	-	1308	1221	1339	1232	803	854
Weight yield/%		-	73	76	78	76	76	72
Assay/%		-	100.1	99.1	98.5	100.4	99.8	99.0
Purity%		-	>99.9	>99.9	>99.9	>99.9	>99.9	>99.9
Indicated product yield/day		-	5.2	7.3	10.7	7.4	7.1	10.2

The table above shows that when operating in continuous mode the reactor produced consistent yields and quality in both assay and purity.

Figure 3 is a typical plot for the formation of amine by nitroso reduction.



Additionally as a result of the enhanced heat and mass transfer characteristics, combined with plug flow, the OBR is particularly adept for crystallisation and polymerisation. By controlling the mixing, dispersion, heat transfer and residence time of feedstock in the reactor, the particle size and morphology can be both predicted and controlled, this coupled with the plug flow leads to a very narrow size distribution of particulates eluting from the OBR compared to a stirred tank reactor.

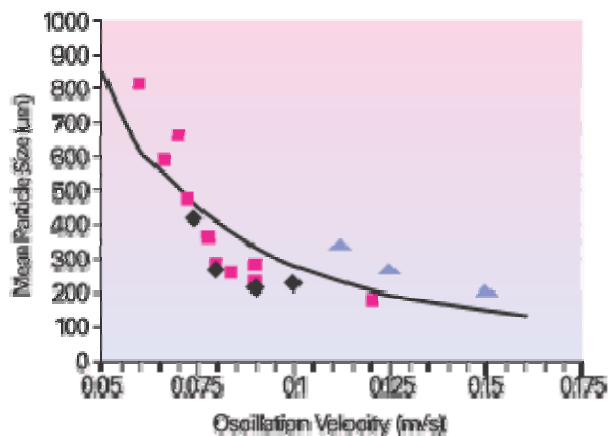
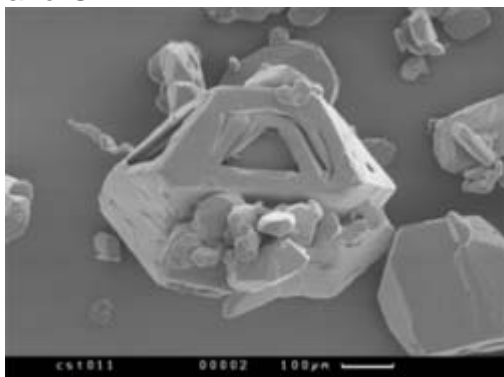


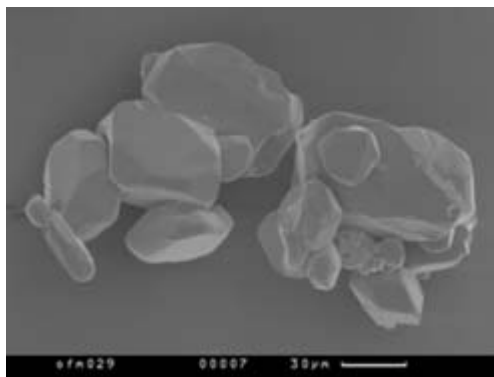
Figure 4 shows mean particle size vs oscillation frequency

Figure 4 above shows how oscillation velocities, i.e. the product of oscillation amplitude and frequency, within the OBR can influence particle size.

While Figure 5 below shows a comparison of product morphology between STR and OBR.

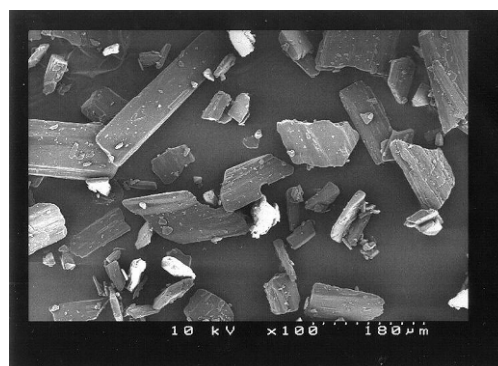
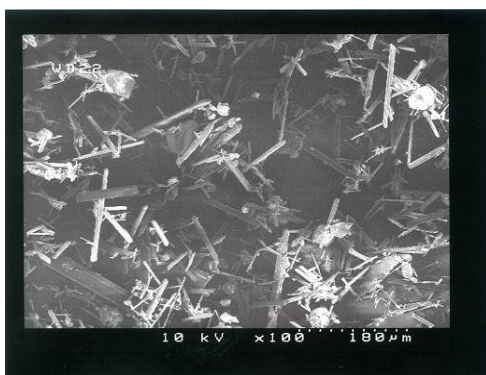


Traditional Stirred Tank



OBR

Denis – the above images indicate tighter size distribution rather than morphology. If you would prefer to show morphology, you may prefer the ones below:



CONCLUSION

When operating the OBR at 50C and 3Bar pressure we were able to reduce an aromatic nitroso to the amine in continuous mode, producing product of consistent purity and assay over extended periods of time (20 hours) with yields comparable to batch processing. Throughout the trials there was no evidence of either fouling or catalyst retention within the reactor. The catalyst was successfully recycled throughout the trials. After confirmation that the “batch “ could be cooled, held in situ overnight re heated and oscillation reapplied, one month of continuous operation was recorded during these trials.

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