

From maximum to most efficient production using a continuous oscillatory baffled reactor

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Nowadays we talk less of manufacturing technologies in chemical engineering as traditional batch stirred tanks have proved to work for a variety of chemical and biochemical engineering applications in both commodity and speciality sectors. In financial terms, the capital costs for manufacturing technologies are marginal to the overall operational profits, but the key question is always how much extra profit would any new manufacturing technology bring, in comparison with the existing one? The answer has to be “substantial” if such a new manufacturing technology would be adopted by industrial producers.

Current Manufacturing Processes

The next question is where any additional profits would come from if the new manufacturing technology is to be implemented? There are a variety of sources for additional profits, but the main one relies on consistent product quality, which entails uniform composition, size distribution and morphological properties. The consistent size distribution also means that there are less under and oversize particles in polymer applications (Ni et al. 1998, 1999), thereby a reduction in waste.

From the chemical engineering viewpoint, consistent product quality should automatically be associated with consistent fluid mechanical conditions in reactors, such as plug flow. We have learnt from the standard textbooks of

Chemical Reaction Engineering that plug flow can be achieved by either running a number of continuous stirred tank reactor (CSTR) in series, or by operating high net flow Reynolds numbers in tubular reactors. To incorporate a number of CSTRs in series in a chemical factory would physically require a set-up of N CSTRs, each with an identical control unit. Ultimately this would significantly increase the capital costs and bring down the overall profits of the operation. For the tubular type of reactors, on the other hand, the main bulk phase would flow through the reactor with other reactants added in at different stages. Without going into too much detail, such an arrangement would certainly have many advantages over the former in terms of the inventory required and capital costs. The vital drawback for this type of reactor is, however, that significantly high net flow Reynolds numbers are needed in order for plug flow to be achieved, this translates as a very long reactor length for even low to moderate residence times. Consequently there have been very few continuous operations in relation to the production of fine and speciality chemicals. There are however examples of continuous operation in commodity chemicals, such as sulphonation and nitration on a scale of 100-1000s tonnes per annum.

Are there any other continuous reactors that allow both plug flow and much better performance than the two cases above? The answer is certainly yes. The oscillatory baffled reactor (OBR) would be one such reactor. The OBR generally consists of a column or tube containing periodically spaced orifice baffles with an oscillatory motion superimposed onto the flow. The mixing in an OBR is completely different from that in a stirred tank, and is provided by the generation and cessation of eddies (Ni et al. 2002). Such mixing is enhanced and much more uniform than traditional stirred tank vessels, as the intensity of the radial mixing is similar to that of the axial one (Mackely and Ni, 1991, 1993). Each baffled cell effectively acts as a continuous stirred tank vessel (Ni 1995, Ni

et al. 2000). The use of an OBR to achieve plug flow condition coupled with a small steady flow component to obtain a long residence time allows production to be operated in a continuous manner. But the critical question is, has such reactors been proved to work in industries? Well, there were a few batch and continuous OBR units sold to various industrial companies worldwide during the 1990s, who attempted the OBR technology for manufacturing of different types of products and failed to make it into a commercial development! This article is to address this specific issue of continuous manufacturing utilising the OBR with a real industrial and commercial development.

Process conversion to OBR currently in hand

James Robinson Limited (JRL) is an autonomous subsidiary of Yule Catto plc based in Huddersfield. The company has a world-wide reputation as a manufacturer of hair dyes and intermediates, photographic chemicals, photochromic dyes and fuel marking dyes. An intermediate for a photographic chemical is currently produced on a several hundred-kilogram scale by a batch operation. The current process involves three stages: wetting, diazotisation and cyclisation, and two batch stirred tanks are employed and operated in a cascaded format, occupying a floor space of 12 m by 10 m by 10 m. The wetting and diazotisation are done in the 1st stirred tank and the content of which is then discharged into the 2nd one for the cyclisation. The batch volume throughput after the diazotisation is 3000 litres and after cyclisation is 13000 litres. Each batch operation takes about 18 hours to complete, including 12 hours for both the wetting and preparation of the buffer in the two stirred tanks; 2 hours for diazotisation; <1 hour for cyclisation and 3 hours for isolation. The current operational parameters permit the manufacture of a single batch per day.

Converting the above batch process into a continuous operation requires one glass OBR unit of two sections: Section A consists of a number of 40 mm diameter tubes and Section B of 80 mm diameter tubes. The wetting and diazotisation take place in Section A, while the cyclisation in Section B. Figure 1 is the schematic diagram showing the set up of the continuous OBR. Parts of the glass tubes are jacketed for the purpose of cooling and heating. These tubes, each 2 m long, are connected by U-bends and are arranged in a snake formation, starting from the top to bottom and then from the bottom back up to top and once more, see in Figure 1. The total flow path is of about 70 m in length, providing a residence time of about 2 hours. There are various ports fitted along the flow path for inputting components, sampling and measurements of temperature, pressure and pH.

Orifice baffles with an optimal restriction ratio are equally placed in both the straight sections and bends of the reactor. The baffles are made of PVDF and are designed to fit closely to the wall of the tubes. Each set of baffles is supported by three 3 mm diameter longitudinal PVDF rods. Two out-of-phase stainless steel bellows at the starting ends of the glass tubes provide fluid oscillation and are driven by a helical geared motor through a frequency inverter. A frequency range of 0-6 Hz can be achieved, and oscillation amplitude of 0 to 20 mm can be obtained by adjusting the off-centre positions of the crank in a flywheel.

For both sections, the net flow of the bulk aqueous phase is taken from the water mains and the flow rates are monitored by flow meters. Non-return valves are used in the flow inlets in order to reduce any propagation of oscillation upstream. In Section A, the limiting reactant is premixed with water and wetting agent and the mixture diazotised along the length of the OBR. At the same time, the preparation of a buffer for cyclisation is taking place in Section B. The stages

of wetting and diazotisation are completed independently. Once the two streams meet, the cyclisation stage commences and the product is continuously formed. Table 1 summarises the key operational indicators for both the existing batch and the continuous OBR processes.

Table 1 Comparison of operations

	The existing batch stirred tank production	The converted continuous OBR manufacturing
Reactor volume (litre)	16000	270
Factory space (m ³)	1200	60
Wetting stage (hr)	12	0.3
Diazotisation (hr)	2	0.3
Cyclisation stage (hr)	0.3	0.3
Yield (%)	83	83
Product purity (%)	99.5	99.5
Output/kg@100%/day	180	150

Note that the yield and quality data are from batch OBR assessments. It is very clear from Table 1 that by converting the existing batch production into continuous operation, we achieve product quality and yield consistent with the batch process; an increased productivity through the removal of down-time and more compact reactor set-up so that reduced capital cost, energy and space usage, improved intrinsic safety and reduced environmental impact become feasible. This has significant impact on the profit margins for such an operation. The project is scheduled to completion in 9 months, and by the end of that, the OBR unit will be dismantled, transported and reassembled at James Robinson Ltd and ready for continuous production on an industrial scale. The continuous manufacturing will replace the existing batch operation.

Conclusion

This article shows that the continuous OBR manufacturing technology is real and viable in the production of a sophisticated and multistage organic chemical product. The product is made using hazardous and corrosive chemicals. We hope that this article would clear up the apprehension and hesitation on taking up this technology and would bring in a new outlook to your operation.

The continuous OBR technology is best suited to reactions with multi inputs of a multiphase nature, and can find applications in a range of industrial operations including phase separated synthesis, inorganic and organic crystals and particles, flocs, dyes, paints, pigments, surface coatings, adhesives, organic and pharmaceutical intermediates. Professor Ni's group at Heriot-Watt University offers various services on converting batch operations to continuous productions for any of the above products. The duration of each of those projects would take usually from 6 to 12 months depending on the specific requirement of the work. We look forward to hearing from you.

It is worth clarifying that the aim of this article is not to suggest that the OBR technology should replace or supersede conventional technologies, such as the stirred tank, but to highlight that the OBR is a real and viable alternative where appropriate, is easily accessible and can successfully be applied to niche processes or products, especially given that the modern trend of Chemical Engineering is to move away from large commodity manufacturing to speciality and high added value products. Continuous manufacturing is the key switching from the maximum to the most efficient production of those products.

Open Workshop

We are holding four demonstrations on Wednesdays 30th July, 6th August, 13th August and 20th August 2003 at Heriot-Watt University campus in Edinburgh. In the workshops you will be able to witness the OBR reactor technology in operation. You and your colleagues are welcome to come along. Please email either Professor Xiong-Wei Ni (x.ni@hw.ac.uk) or Dr Andrew Fitch (a.w.fitch@hw.ac.uk) for your preferred dates. We look forward to meeting you.

References

Mackley, M.R. and Ni, X., 1991, Mixing and dispersion in a baffled tube for steady laminar and pulsatile flow. *Chem. Eng. Sci.* 46: 3139-3151.

Mackley, M.R. and Ni, X., 1993, Experimental fluid dispersion measurements in periodic baffled tube arrays. *Chem. Eng. Sci.* 48: 3293-3305.

Ni, X., 1995, A study of fluid dispersion in oscillatory flow through a baffled tube. *J. of Chemical Tech. & Biotechnology*, 64: 165-174.

Ni, X., Cosgrove, J.A., Arnott, A.D., Greated, C.A. and Cumming, R.H., 2000, On the measurement of strain rate in an oscillatory baffled column using particle image velocimetry. *Chem. Eng. Sci.* 55(16): 3195-3208.

Ni, X., Jian H. and Fitch, A.W., "CFD modelling of flow patterns in an oscillatory baffled column", *Chemical Engineering Science*, 57, No. 14, 2002, 2849-2862.

Ni, X., Zhang, Y. and Mustafa, I., 1998, An investigation of droplet size and size distribution in methylmethacrylate suspensions in a batch oscillatory baffled reactor. *Chem. Eng. Sci.*, 53(No. 16): 2903-2919.

Ni, X., Zhang, Y. and Mustafa, I., 1999, Correction of polymer particle size with droplet size in suspension polymerisation of methylmethacrylate in a batch oscillatory baffled reactor. *Chem. Eng. Sci.*, 54: 841-850.

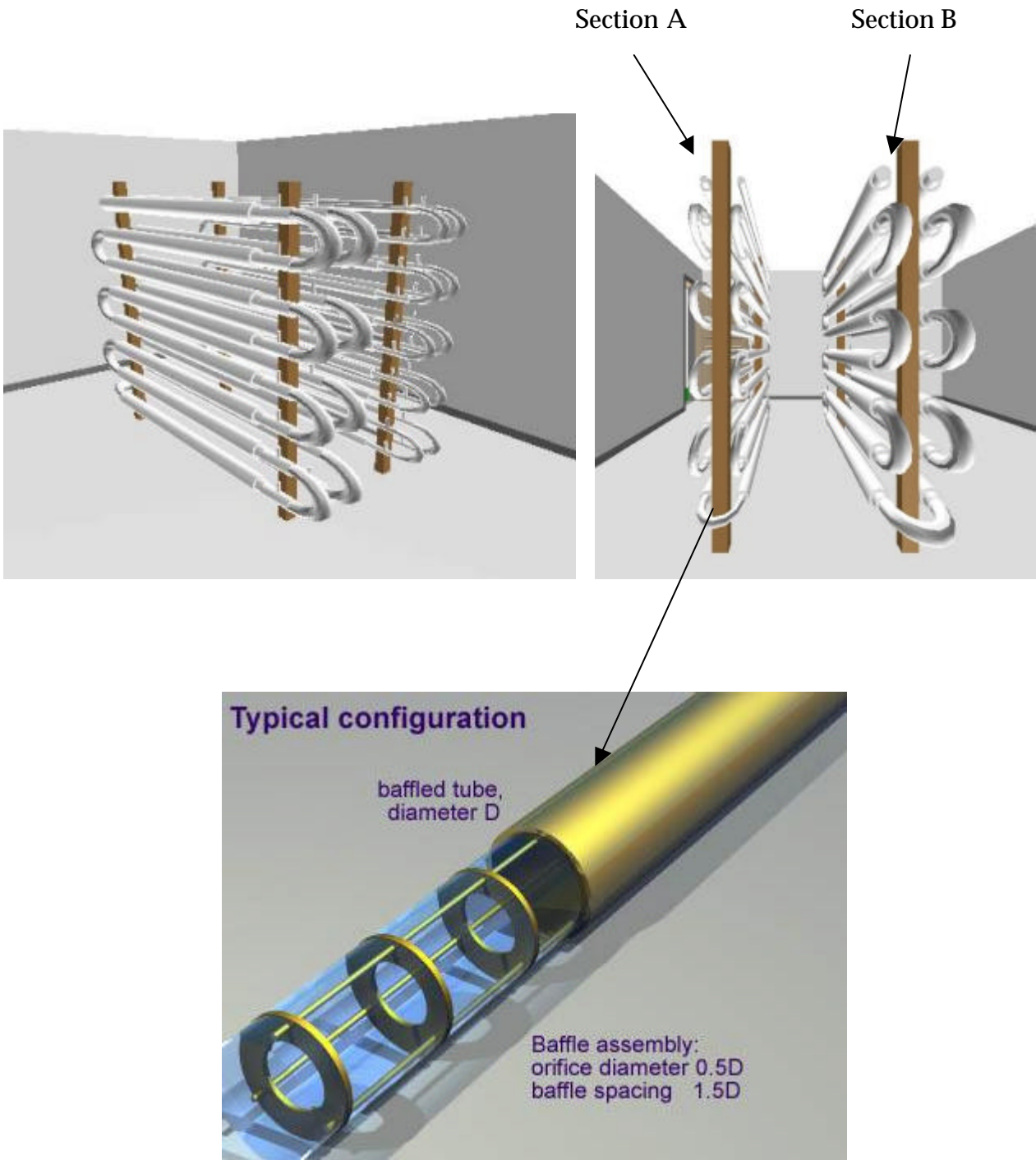


Figure 1 Layout of the OBR set up