8^{TH} PIN MEETING

BHR GROUP, CRANFIELD, 14 NOVEMBER 2002.

MEETING MINUTES

The 8th PIN meeting was hosted by BHR Group in the Library at Cranfield University on 14 November 2002. The attendance was 58. Colin Ramshaw welcomed members by referring to the Whittle and Rolls-Royce Trent gas turbines in a nearby hangar as illustrating PI.

Introductory Talks

Colin then introduced Andrew Green, our host, (Agreen@bhrgroup.co.uk) who gave us an interesting overview of BHR Group. The company was founded over 50 years ago and is an independent contract research & development organisation. It covers all aspects of fluid engineering and is a world authority on mixing processes. Other major areas of expertise include process intensification and asset and process management. BHRG works for the chemical industry, utilities, manufacturers etc., and has extensive CFD capabilities. The FMP (Fluid Mixing Processes) programme is its largest single project, with more than 35 industrial supporters world-wide.

Andrew then told us about the new alliance between his company and CSIRO, the premier Australian research organisation, which will explore joint activities in fluid dynamics and fluids engineering.

He then reminded us of the call for papers for the 5th PI for the Chemical Industry Conference, to be held in Maastricht on 13-15 October 2003. See www.bhrgroup.com or contact Catronia Pile on cpile@bhrgroup.com

David Reay (DAReay@aol.com) then updated members on PIN activities since the last meeting. His presentation is available on the PIN web site, but highlights included the fact that sponsors have provided adequate funding to operate PIN until April 2003, and we hope to sign up the DTI and CCFRA (Food Research Association) as sponsors soon. (New sponsors are invited, contact David). PIN membership has expanded to 320, of which more than 60% are from industry and over 25% outside the UK. A survey of PI views carried out by MSc students at Heriot-Watt University is now available on the PIN web site – www.ncl.ac.uk/pin/

The recognition of the importance of PI is evident from the selection of four experts in the field at Newcastle, Colin Ramshaw, Roshan Jachuck, Kamelia Boodhoo and David, to serve as members of the new EPSRC College of Referees. The UK is also active in taking PI to Europe via the EC 6th Framework Programme – both BHRG and Newcastle University submitted Expressions of Interest for Integrated Projects (BHRG) and Networks of Excellence (N/C Univ.). Newcastle will also host the 1st Int. Symposium on PI & Miniaturisation in August 2003 – contact Galip Akay on Galip.Akay@ncl.ac.uk (*See Pin Web Site for David's Powerpoint presentation*)

Technical Presentations

The FlexReactor: Appropriately, Andrew Green gave the first technical presentation, his subject being the Flex Reactor. Andrew saw PI as “a step change in product, process and business performance through an in-depth understanding of fluid dynamics and process chemistry." PI is a design philosophy. Matching for example heat transfer to exothermicity enables a process to proceed safely at its optimum rate. Current key issues in manufacturing are flexibility and responsiveness to address changing market demands.

The first example of PI quoted by Andrew was the static mixer, where 100s of W/kg in mixing energy could be delivered, compared to 1-2 W/kg in a stirred tank. The up to 100-fold higher mass transfer rates in static mixers (compared to stirred tanks) is characterised by uniform energy dissipation, plug flow and good radial mixing. Moving to reactors, Andrew cited the Marbond HEX-reactor, which demonstrates mixing + reactions + heat transfer in one unit. However, this unit was not flexible – once built it could not be changed. Therefore the idea was to introduce flexibility through 'reconfigurability'. This was done in the FlexReactor.

The FlexReactor uses simple static mixer technology in a tubular unit with removable U bends, allowing connections to be changed to vary the residence time. The unit can be constructed using a variety of materials (lack of choice of materials sometimes being a PI blocker). A Smart Award has been given to the invention.

Capabilities of The FlexReactor include pressures to 20 bar (50 in practice), temperature range –70 to +250^{o}C, flows 1 – 100 l/h, pressure drop to 15 bar (100 l/h, 10 barg pressure drop), and at this capacity the energy dissipation is 100 W/kg. Heat removal in the unit Andrew showed was 15 kW, (LMTD 50 between process fluid and coolant). Exothermicity ranges up to 750 kJ/mole, residence time 2 s to 30 min. A derivative is the FlexPlant – a flexible laboratory which will fit in to a large walk-in fume cupboard. This allows rapid screening of suitable reactions for PI. This can be purchased or rented.

The next stage was OSPRI – scaling up to a pilot unit which could be driven into a chemical plant. Scaling down is possible – the FlexPlant is laboratory-sized but pilot scale in volume (10's of litres). Andrew said it was difficult to construct at a smaller scale – here one enters the laminar flow regime. For small throughputs/volumes (10's to 100's of millilitres) one could go from continuous to batch processing.

During discussion, Andrew stated that although the FlexReactor was of shell & tube configuration, there were static mixers on the tube-side and an enhanced shell-side. It was not as good as the Marbond in this respect, and although the latter had a higher energy dissipation it was less efficient than the FlexReactor in turning pressure drop energy into mixing energy. Concerning the handling of viscous fluids, Andrew pointed out that although at the small scale the mixers were welded in, with 12 mm or high tube diameters they could be removed and cleaned. Cleanliness can be checked by removing the U bends. One can also use an aggressive cleaning regime using resistant metal alloys.

Potential Benefits of PI to Rhodia: Sylvaine Neveu of Rhodia (Sylvaine.Neveu@eu.rhodia.com) described how PI could benefit her company. After detailing the varied activities of Rhodia, which lies in size between ICI and Clariant in terms of size in the speciality chemicals area, she described the main processes – polymerisation and organic/inorganic synthesis – which might be subjected to PI. PI, she stated, significantly enhanced transport rates and gave every molecule the same processing experience. Sylvaine showed an interesting graphical representation of PI, plotting mass transfer vs. heat transfer. The plate heat exchanger won on heat transfer at the expense of low mass transfer, while the converse was true for the rotating packed bed. Micro-reactors were good at both!

Rhodia saw the multiple reactions in organic synthesis as being potential beneficiaries of PI. Impinging jet mixer technology was one opportunity and in the Bourne reaction one could improve selectivity and diminish secondary products, thus removing the need for separation (or at least making it easier). In exothermic reactions, the introduction rates of reactants are limited by the speed of heat removal – PI can improve this.

For inorganic processes, a typical product would undergo precipitation, separation/washing, drying, calcination and then presentation to the customer. All steps must be intensified to improve productivity, but it is not easy. The precipitation process is a quality-critical step, and nucleation, growth, aggregation and agglomeration all need controlling. Mixing has an effect on these, as does hydrodynamics. Using agitated batch or semi-batch reactors, one gets a large particle size distribution and a problem of scale-up, due to limited heat & mass transfer. Scale-up criteria are also difficult to assess, for example the relationship between hydrodynamics and nucleation rate.

Using PI would accelerate heat and mass transfer, give better between reactants, better hydrodynamic control, and make the system more easily scaleable.

In 1994 Rhodia was granted a patent for the production of an Al/OH/xCly polyaluminiumchloride compound for water treatment. The production was however associated with the formation of an undesirable insoluble secondary product. Use of a 14 cu.m semi-batch reactor did not allow separation, and the solution was to install a 'quick mixer' which was Y-shaped and mixed the reactants under good stoichiometric conditions. The final composition was as needed with no by-products.

For polymerisation, Rhodia is studying going from batch/semi-batch to continuous processing. But there is a need to change the product each day in the same plant (change in demand), and in continuous configurations daily changeover can give rise to problems with intermediate products. However, PI decreases the plant size, raises production and decreases intermediates. In devolatisation, thin films also assist mass transfer.

In summary, Rhodia sees PI as giving greater production, lower investment cost, greater speed of product development and high product quality.

During discussion, Sylvaine confirmed that Rhodia was using PI for heat & mass transfer intensification. She believed the best way to adopt a proactive approach to PI was to use it from the beginning of a product development. One can show the advantages at the laboratory scale, then one has the choice in production between, for example, a PI or a standard reactor. With regard to investment, one can add PI equipment without changing everything (the questioner was adopting this approach rather than implementing full changes).

Asked how far Rhodia had gone in developing criteria for testing processes for PI relevance, Sylvaine said that they have a systematic approach, starting with precipitation and extending to other fields. They are studying carefully the efficiency of a range of PI devices, heat & mass transfer, hydrodynamics etc., and to obtain laboratory scale devices. Only then will a study be made of process parameters for the larger scale.

One member observed that one difficulty of PI is the range of modules and lack of hydrodynamic data – therefore one goes for the 'safe bet', the batch reactor. Sylvaine said that one needs a good knowledge of the plant to convince the plant people that PI is worth investing in. (*See PIN Web Site for Sylvaine's Powerpoint presentation*).

Intensified Precipitators for Ultrafine Particles: Matt Scalley of Newcastle University (M.J.Scalley@ncl.ac.uk) is working with Protensive Ltd. under a Teaching Company Scheme on a cheap and simple way to produce fine particles.

Firstly, Matt explained the process of reactive crystallisation, the reaction of two soluble species to form an insoluble product. The driving force is supersaturation, and as supersaturation increases, three regimes of nucleation are passed through, these being heterogeneous, homogeneous (the best for micro-particles) and agglomeration. The research is directed in part at examining the influence of hydrodynamics on particle formation. There is a need for micro-mixing and one also needs uniform supersaturation throughout the reactor (uniform mixing and plug flow).

The conventional process is a stirred pot, which is non-uniform and has other drawbacks. Normally the cost of getting micro-mixing therein is high in terms of energy use.

There are two solutions – the micro-reactor using narrow channels or the spinning disc reactor (SDR). The micro-reactor has 0.5 mm diameter channels, and laminar flow. A short diffusion path is given together with plug flow. One can easily add another stream to for example coat the particles. For higher outputs, one would employ many channels in parallel. The SDR would use its intense mixing in thin films. This could possibly lead to nano-composites made via the disc. Gas-liquid or liquid-liquid reactions could be employed for production of TiO_2. The SDR gives much smaller particles (50-60 nanometres, or 0.5 microns) with a narrower size distribution than the 1-2 microns with some of 5-7 microns using stirred pots.

The SDR speed is in the range 2000-8000 rpm. Residence times are < 1 second. Also now being studied are the effects of ultrasound on performance, and ways for controlling the particle shape. Matt summarised the advantages and predicted that one might eventually ‘dial up’ particle properties and degree of scale-up.

Matt said that applications included pharmaceuticals, chocolate, paints/pigments and magnetic recording media.

During questions, Matt said that results had been achieved using micro-channel reactors, but the SDR outperforms these to date. (*Matt’s Powerpoint presentation can be seen on the PIN Web site*).

Laboratory Protocols for PI: Ian Reynolds of BHR Group (IReynolds@bhrgroup.com) introduced his talk by summarising the aims and benefits of PI, but said that there were constraints – e.g. clogging, the ‘biggest is best’ attitude, and the fact that some reactions cannot be speeded up. The PI methodology allows one to see whether a process can be intensified. It includes laboratory protocols.

This involves designing a small high intensity stream to try to model the heat exchanger-reactor in the protocol vessel. One needs simple, safe, high intensity mixing, homogenous mixing, heat transfer capabilities, and a well-understood process behaviour. The BHR unit has a volume of 733 ml and has a 400 rpm stirrer with highly effective impellers.

Why use laboratory protocols? Less fluid is needed, they are quick and simple, use known equipment and there is no risk of fouling or blocking.

The characteristics were tested using the Bourne mixing-sensitive reaction scheme. The protocol vessel was characterised: e.g. 95% mixing time determination, local point energy dissipation rates, critical feed time (when it changes to micro-mixing-controlled state). Static mixers of various types were also simulated, on the basis of energy dissipation rates, Reynolds number and meso- and micro-mixing timescales.

BHR Group achieved a good match of by-products between static mixers and stirred tank reactor. In future they hope to look at the simulation of HEX-reactors and spinning disc reactors.

During discussion, it was pointed out that heat transfer was also important, so how does one make sense of heat transfer in a glass vessel? Ian replied that a stainless steel one would be used in the future.

Free Radical Polymerisation on an SDR: Phil Leverson of Newcastle University has been investigating styrene polymerisation on a spinning disc. Using a 36 cm diameter disc, the polymerisation rate is over 100 times faster than any known batch system. However, it is noted that conversion is a function of feed viscosity and rotating speed. Phil is investigating why this happens.

The spinning disc test facility was discussed by Phil, a probe at the periphery of the disc being used to measure the concentration of product at that location.

One suggestion is that rate enhancement is due to a reduction in bimolecular termination reactions.

Model validation was attempted. A comparison of experimental and model results showed that the model slightly underestimates the experimental conversion. The reason for this is not known yet, but is under investigation. Good comparison was achieved on the prediction of the polymer molecular weight distribution. The predicted value was 23356 g/mole, while experimentally a value of 25000 g/mole was measured.

Phil concluded by saying that further data were being collected to complete the model validation, in particular by examining the effects of varying feed composition and reaction temperatures. *(Phil's Powerpoint presentation can be viewed on the PIN Website*).

Launch of EC 6th Framework Programme: John Sillwood (John.Sillwood@npl.co.uk) of NPL, which hosts the UK contact point for industrial technologies in FP6, arrived hot foot from Brussels, where the new Framework programme was launched to an audience of 8000 earlier in the week. The interest to PIN in this is strong, as members have submitted major expressions of interest in 'Integrated Projects' (BHR Group) and 'Networks of Excellence' (Newcastle University).

John presented essential information from Brussels on priority area 3, which covers nanotechnology and related topics (the closest area to PI). A call for proposals is expected on 17 December 2002. Data on expressions of interest are given on the Cordis web site – see http://eoi.cordis.lu/search_form.cfm

The expressions of interest were designed to inform the EC of areas of interest, and to alert researchers about FP6 and new implementation methods. One important point is that research should 'respond to societal needs'. There will be a 'European Research Area', and the addressing of competition problems is planned.

The funding for FP6 is 17.5 Billion EURO. The programme features include:

Concentration of effort in certain areas
Creation of real EU added value
Improving links between policies & schemes
New instruments
Simplification of management & implementation
15% of priority themes budget for SMEs in addition to special measures elsewhere

Most of the budget goes to integrating European research. Nanotechnologies & related areas get 1.3 BEURO and 430 MEURO goes to CRAFT.

New activities include Integrated Projects and Networks of Excellence (see above). There are traditional targeted R&D projects (STREPs) and co-ordination actions (were called Thematic Networks). Specific support actions cover what were accompanying measures.

New partners can be taken in to projects during their operation, although this has to be allowed for in the original budget. There is increased management autonomy and more freedom on IPR, although accounts have to be audited.

Integrated Projects involve research to provide information and technology to develop products and process technologies. Can cover R, D & D, dissemination, training, mobility etc. Networks of Excellence address fragmentation – their prime deliverable, and should create integration and advanced know-how.

Priority area 3 is aiming at breakthrough long term research. Integrated projects in this area should follow a multi-disciplinary approach. NoEs may address a single topic (e.g. PI). STREPs should be aimed at leading edge research, or fundamentally at the development of knowledge.

Two areas John highlighted as relevant to PI in Area 3 were radical changes in 'basic materials', including cleaner, safer and more eco-efficient production. (IPs and STREPs) and sustainable waste management and hazard reduction (NoEs, Co-ordinated Actions and Specific Support Actions).

(**The PIN webs site contains John's overheads, as well as <u>preliminary versions</u> of appropriate parts of the call for proposals)**

How to get Novel Technology Accepted: was the subject of Ian Henderson' s talk. Ian (Ian.Henderson@processtech.freeserve.co.uk) works for Protensive Ltd., and he started by playing Devil's advocate with regard to PI. For instance, Ian said that the cost of installation (of a PI plant compared to a conventional one) does not change; pipework may be more complex; land is often available (e.g. Wilton on Teesside); storage tanks may dominate the plant layout, and existing technology will, unlike PI, be well down the learning curve. Other points included the observation that saving 10-50% in capital cost was not currently a dominant factor. (Margin, output volume and timing can be more important). Preferably one needs orders of magnitude reduction in capital costs.

Looking at some important criteria in turn, Ian observed that:

- Energy costs – PI does not give inherently lower energy costs
- Chemistry yield – Good justification – lower raw materials, less waste, less energy etc.

- Safety – existing plant is safe, therefore why change?
- Improved manufacturing – One can do 'Just in Time (JIT) via PI. But failure would be dramatic unless PI is used as 'add on' stream, for example as a side-stream.
- Novel/much improved products – this is a real money-making opportunity for PI if one can only do it with the new technology. Also existing products should continue to be made so that failure does not damage the core business.

Ian said that the best chances for PI are where one can:

Develop novel or enhanced products (including JIT)
Obtain increased yield
Develop opportunities through the company strategy, where once established the risks of the PI strategy are reduced and other benefits can be considered.

During the lunch break, Bernd Werner of the Institut fur Mikrotechnik Mainz, Germany (Werner@imm-mainz.de) showed a poster and examples of the micro-engineered PI systems developed at his institute.

***Impromptu* Presentations**

Five impromptu presentations were given in the afternoon session, which was chaired by Roshan Jachuck of Newcastle University.

Controlling & Monitoring Heat Exchangers: Robert Ashe of Ashe Morris Ltd. (Robert.Ashe@ashemorris.com) a design company, introduced us to an interesting new concept, the variable area heat exchanger. He described this as a different way of building and using heat exchangers, and illustrated it with an example for controlling the temperature of a chemical reactor, with a 10 kW heat load. If one measures the heat balance across the coil used to heat the (endothermic) reaction, one often gets + or – 20% error on the balance, based upon in part the use of a valve to control the flow thought the fixed heat exchanger surfaces. If one replaces this valve with a tiny heat exchanger, so that one has in effect two coils, one can get a heat balance of + or – 1.7%. In fact it can be as accurate as one wants, giving perfect control.

The unit selected was a 10 litre volume Hastalloy batch reactor. By measuring the enthalpy and controlling the temperature in a narrow band, for a batch reactor one can get a smaller unit with better stability, faster temperature control and accurate enthalpy data can be collected for on-line monitoring. In answer to questions, Robert confirmed that the system could be retrofitted.

RAM – Rotated Arc Mixer: Dilip Manuel, (Dilip.Manuel@csiro.au) working at BHRG as part of the above mentioned links between CSIRO and BHRG, showed a video of the operation of his RAM, a new mixer for highly viscous materials. The mixer comprises an external rotating tube, and an internal static tube with slots in it. It is more effective than static mixers, uses 20% of the energy, has no stagnant regions, no internal surfaces, employs chaotic mixing and can be used in batch or continuous mode. The performance is a function of length, tube diameter and slot size.

Dilip is currently designing an industrial scale RAM – see www.dbce.csiro.au or www.cmit.csiro.au

Drinks Dispenser Challenge: Hoi Yeung of Cranfield University (h.yeung@cranfield.ac.uk) put to us an interesting fluid flow problem associated with beer! Two phase flow in small annular gaps is a characteristic of the nozzle systems used to dispense drinks. For drinks where a 'head' is required or gas has to be introduced for other reasons, the nozzle development has involved measurements of pressure drop vs. flow, and the pressure drop decreases as the gas is introduced into the liquid. Hoi is interested in explanations for this and opportunities for other applications of such nozzles in PI plant. (*Graphs shown by Hoi are on a Powerpoint file on the PIN Website*)

The National Measurement System (NMS): Jeff Howarth of NEL (Jhowarth@nel.uk) introduced us to the NMS programmes, which maintain and develop a national infrastructure to ensure that measurement in the UK is valid, consistent, fit for purpose and internationally recognised. The programme budget is £2.5 million p.a. Although not many people have heard of the NMS, it is of considerable importance to industry, and Jeff is currently contacting PIN members with a questionnaire to assist in the preparation of the future NMS activities. Themes in 2001-4 relevant to PI include industrial & combustion thermometry, and thermal modelling, as well as complex methods for complex and biomaterials. Also included are transient methods for thermophysical properties of liquids, and knowledge & technology transfer.

Jeff is looking for inputs to help plan the 2004-7 programme

Dutch Update: Henk van den Berg (h.vandenberg@ct.utwente.nl) told us of the latest activities of the Dutch PI Taskforce. At a meeting in October 2002 it was reported that 10 projects (supported by NOVEM) had been completed. Two save 30% on energy, pollution and capital cost. It was evident at the meeting that many chemists and consultants had still not heard of PI.

The Dutch PI Guide is to show data and the tools available to help implementation. However, Henk has run into a roadblock because of confidentiality of data. The Dutch 'How to do PI' Guide will also cover criteria for success. The 'How to do' aspect would set it apart from the UK Guide.

Henk is reviewing with his Group the applications of PI, and proposes to present these at the Maastricht conference next year (see above). Henk asks for assistance from PIN members in identifying applications. Please contact him on the above email. Additionally, the Dutch group is looking at training courses in PI. (*Henk's presentation is available on the PIN web site*).

Laboratory Visit

We were then shown round the BHRG laboratories – an interesting visit by all accounts.

Our hosts provided us with excellent hospitality and facilities, and the feedback to date has been most appreciative. Thank you, Andrew, and your colleagues.

These minutes were written by David Reay on the basis of notes prepared by him.
23 November 2002.