PROCESS INTENSIFICATION NETWORK (PIN)

MINUTES OF THE 7TH MEETING,

NEWCASTLE UNIVERSITY, 23 MAY 2002.

The 7TH meeting of PIN was held in the Dept. Chemical & Process Engineering at Newcastle University on 23 May, 2002. On the previous afternoon a number of members visited Thermacore Europe, a company manufacturing heat pipes and located in Ashington, and several members met for dinner afterwards.

Introductory Talks

Colin Ramshaw (Colin.Ramshaw@ncl.ac.uk) welcomed members to the meeting, about 48 being present. Colin pointed out that the EPSRC, which funded PIN for its first three years, had given very high marks to the final report on the project. The PIN meeting at Newcastle was also an opportunity to view the outcome of the £1.6 million grant to the University from EPSRC and the Wellcome Foundation to upgrade the laboratory facilities.

David Reay (DAReay@aol.com) then updated members on what has been happening in PIN since the last meeting. *His overheads are available on the PIN web site* (www.ncl.ac.uk/pin/), but the highlight was the securing of funding for the current year from a number of industrial and Government sponsors, (see 'footer' on this page). Membership had reached 309, with more than 60% from industry. PIN was also planning to submit an expression of interest to the EU to be involved in a European Network of Excellence in the area. (Further details will be given in *PIN News*).

Technical Presentations

John Burns of Newcastle University (J.R.Burns@ncl.ac.uk) kicked off the Technical Presentations by telling us about his work on the measurement of liquid film behaviour on rotating discs, funded within the IMI programme. The requirement is for a rapid measurement method, and then the development of methods for controlling the flow. John described techniques for measuring the film thickness and its residence time, and this allowed calculation of the 'spin up' zone, which is the middle of three zones, preceded by the jetting zone and followed by the Nusselt zone where the velocity of the film over the disc passes its peak and decreases.

The theory of the electrical resistance measurement was described, followed by details of the disc construction. A Perspex disc of 30 cm diameter had embedded in it brass electrodes, spaced 1 cm apart. The sampling frequency of the signal was 6.25 kHz, and the sensors were calibrated to monitor film thicknesses of 10-1000 microns. Additionally, flow of a tracer liquid was monitored using the probes, and the relative conductivity of for example water/ethanol, can give the concentrations. The tracer response method and the film resistance method gave good agreement. Details investigated included the variation in radial velocity across the disc, (allowing the

The Process Intensification Network (PIN) is sponsored by the following organisations:
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spin-up radius to be calculated), and the impact of fluid viscosity on behaviour of the film.

It was concluded that electrical resistance could be used to monitor film thickness; tracer response could also be used, and the spin-up zone could be examined. In future John plans to carry out local measurements, such as examining the surface waves, put in metal discs, and measure other factors. *The overheads from John's talk can be fund on the PIN web site.*

Ruh Goh described work on the rotating fluidised bed incinerator at the Sheffield University Waste Incineration Centre (Y.R.Goh@sheffield.ac.uk). The Centre has 20 staff, including post-graduates, and this particular project was supported by an EPSRC ROPA grant with industrial input. Ruh said that in Japan there were 50 fluidised bed incinerators, while in the UK 10 units were used to burn sewage sludge. She showed us a conventional fluid bed unit, which included a separator unit. In the rotating bed a separator was not needed.

The characteristics of the conventional fluid bed unit were: low excess air needs, low NO_x, no bottom ash, low carbon-in-ash, but snags included much more fly ash and the fuel needs pre-treating to give a maximum length of 150 mm. (This is done by shredding, which is expensive and unreliable).

The impetus for new waste disposal methods comes from the UK problem of excess use of landfill, which accounts for 90% of disposal, but is now taxed. Biological waste can be anaerobically digested, but the rest can be treated by incineration. With regard to sludge, each year there are 35 million tonnes of sewage sludge, 67 mt of industrial sludge, and 11 mt from agriculture. The calorific value is typically 50-60% that of coal. There are few alternatives to incineration for sludge disposal, which reduces its volume by 80% and mass by 70%

The rotating fluid bed unit is suitable for sludge. 'G' forces are in the range 4-20 g and the output can be used for gasification. The bed can be horizontal or vertical. Ruh showed photos of a small unit and an industrial scale unit, which could service a population of 10,000. The horizontal arrangement gives even distribution of the bed depth. Rotating at 400 rpm, the throughput is 20-30 times that of a conventional fluid bed unit, based on volume. This is reflected in its compactness, and the unit also has a higher combustion efficiency and more stable combustion. Ruh illustrated the talk with a video of chicken litter combustion. The temperature range was 800-900^{o}C, and the silica sand used in the bed would not melt until 1500^{o}C was reached.

As well as sludge combustion, this concept could be used for coal gasification, and co-gasification of coal and sludge. With regard to coal combustion alone, the heat output is too high, 100 MW/m^3, and even cooling would not benefit the system. Advantages of the rotating bed include high process intensification, high combustion intensity, easier start-up and shut-down, good turn-down and mixing, and the benefit of sulphur retention in the bed.

Several PIN members had already seen the heat pipe facilities of Thermacore the day before, and Song Lin of Thermacore Europe (song.lin@thermacore.com) gave us more information on the device and its uses, particularly in thermal control in micro-systems. Firstly Song described how heat pipes work – a two-phase liquid-vapour cycle in which liquid transport is carried out passively by capillary pumping action in a porous wick. (See www.thermacore-europe.com for further details). The heat transfer using the vapour ensures minimum temperature drop, giving a very high effective thermal conductivity. Other characteristics include silent operation, light weight, compact and design flexibility. Heat pipes are highly reliable and have a long life.

Design procedures such as working fluid selection (water is used between 5^{o}C and 200^{o}C), incorporation of bends, flat plate configurations and wick design (e.g. wire mesh, sintered metal powder) were described. Song showed pictures of different configurations, such as loop and rotating heat pipes (the latter using rotation to enhance heat fluxes as well as transporting the fluid), pulsating heat pipes and micro-heat pipes, of triangular cross-section with sides of 0.2 mm.

There are many uses for small heat pipes in micro-electronics thermal control – Moore's law states that chip power densities will double every 18 months, and this is necessitating ever more intense cooling methods, including heat pipes (e.g. for Pentium chips). Telecommunications base stations, a prime target for Thermacore, have seen internal heat generation densities rise from 2.5 kW/m^3 in 1995 to 8 kW/m^3 in 2002. Other applications/heat pipe configurations discussed included reactor temperature control and isothermalisation, waste heat recovery, heat pipe use in solar collectors, compact heat exchangers and spacecraft.

We returned to spinning discs with Kamelia Boodhoo (k.v.k.boodhoo@ncl.ac.uk), who told us about her research into photo-polymerisation on a spinning disc reactor (SDR). Firstly Kamelia described the chemistry involved. There are two types of polymerisation to be considered, chain polymerisation and step growth polymerisation. In turn, chain polymerisation can be sub-divided into free radical and cationic/ionic variants. Photo-initiation was examined with the free-radical variant, the mechanisms involving initiation, propagation and termination.

The conventional stirred tank reactor (STR) is limited in its acceptance of photo-polymerisation, as UV light can only penetrate a few mm, much of the mixture remaining unaffected, in addition to the other common limitations of STRs. With the SDR, the thin film generated on the disc is ideal for effective UV treatment, as is the good mixing and short, controllable residence times.

Kamelia reported on the experimental programme, which examined performance over a range of variables (e.g. disc speed 200-1000 rpm, monomer flow rate 1 and 5 ml/s). It was found that polymer conversion decreased from 90% at 200 rpm to 32% at 1000 rpm, with residence time decreasing as well, from 2.1 to 0.7s. UV intensity was varied from 10-130 mW/cm^2, the optimum being 25-45 mW/cm^2. Benchmarking was done with a static film, which gave 30% conversion in 10s.

The superior performance of the SDR was attributed to the important role mixing plays, (in addition to other well-know merits of SDRs). Distributed manufacturing of polymers was seen as a business opportunity for this concept. *The overheads forming Kamelia's talk are available for viewing on the PIN web site.*

Phillip Wright of Heriot-Watt University (p.c.wright@hw.ac.uk) discussed intensification in bio-processing. He stressed that one was looking at different time frames than for 'conventional' PI when one intensifies, for example, a bio-reaction. One can still achieve three orders of magnitude intensification using genetic techniques, but the reactor used is still much larger than a chemical reactor.

The production of hydrogen was one area where Phillip said PI could be used. The hydrogenase enzyme can do this. When genetic engineering is used to intensify the reaction rate, giving the orders of magnitude improvement mentioned above, mass transfer and reaction become important factors. One aspect is the study of the effect of shear on organisms – the aim being to introduce oxygen into them without rupturing the cell. So very high turbulence cannot be used, but employing oscillatory baffle reactors is possible. Other reactor design considerations include the need to get light into them. Bubble columns can be so arranged, but one has to avoid high agitation and velocities.

Some bio-processes can be carried out in an integrated plant, part chemical reactor and part bio-reactor, although the latter unit is the largest physically. The most recent project, funded under the earlier Low Carbon Technology Programme, involves integration of units to produce a multi-functional plant for hydrogen production, making a pharmaceutical and fixing CO_2. The aim is to reduce the number of discrete unit operations and to scale up the plant to 350 litres. One aspect of the work involves photo-bio-reactor design and associated scale-up. The tubular reactor was found to be best for this.

Funding/Collaborative Opportunities

David Reay then introduced two collaborative opportunities – a proposal to the EPSRC and a new call for proposals from the Carbon Trust.

The EPSRC proposal currently being put together involves eight universities, micro-engineering laboratories and industry. Entitled 'Multi-phase transport phenomena in micro-systems', the project will aim to address a number of challenges to flow and heat transfer at the micro-scale, relevant to thermal control in many areas, including micro-reactors and heat exchangers. Specific areas to be studied include condensation at the micro-scale, flow boiling, and surface tension and interface curvature considerations. 'Micro' covers: 1-3 mm, 1 mm to 50 microns, and <50 microns characteristic dimensions.

Prior to submitting the proposal in the autumn of this year, further interest in participation from industry is invited. Please contact David on DAReay@aol.com. *The overheads accompanying the talk can be viewed on the PIN web site.*

Responsibility for R&D under the EEBPP was transferred on 1 April to the Low Carbon Innovation Programme (LCIP), under the Carbon Trust. A new LCIP scheme, the Foundation Programme, was launched on 15 May. Details can be viewed on the CT web site: www.thecarbontrust.co.uk and calls for proposals will be issued at intervals during the year. A call for R&D projects is currently open, with a deadline for outline submissions of 5 July 2002[1]. This call will accept feasibility studies, with funding of up to £50K, and R&D projects, with funding to £150K. The main criterion is a contribution to reducing greenhouse gas emissions. Demonstration projects to bridge the gap between R&D and commercialisation will also be supported. There is only £1.5 million available to co-fund projects in this first call, but there will be other calls. It may be worth waiting until 'teething troubles' are ironed out – inevitable with new programmes.

***Impromptu* Presentations**

The afternoon session comprised several short presentations, ranging from those discussing technical innovation to reports on other group activities and opportunities to participate in the new EU calls for 'Expressions of Interest'[2].

Asterios Gavriilidis of University College London (UCL) reported on the design and performance of micro-engineered and intensified catalytic reactors, (a.gavriilidis@ucl.ac.uk). UCL was looking at their fabrication in silicon/glass for oxy-dehydrogenation reactions etc. Channel width was 600 microns, length 20 mm. Tests showed a linear increase in conversion with temperature to 540^{o}C. Reducing the channel depth was found to increase conversion and reduce selectivity.

Asterios also described a falling film reactor with a micro-structure. 50-100 micron films were formed with a residence time of a few seconds. It was calculated that a 1600 m^3 methane reformer could be reduced to 6 m^3 using a 400 m^2/m^3compact heat exchanger structure. A further development was a zeolite reactor of serpentine structure for epoxidation.

Willem de Vries of NOVEM (w.de.vries@novem.nl) updated us on the activities of the Dutch PI group and the PI Guide planned in the Netherlands. A group meeting on 2 May heard from a medium-sized company making floor coverings (lino) which had moved from batch to semi-continuous processing. ABB Lummus described how they were implementing PI in some process routes to be introduced in 2003. DSM Research Laboratories were offering laboratory services to other organisations, and students from TU Eindhoven had been on a successful study tour of the USA looking at PI[3]. Willem said that the Dutch PI Guide - 'How to do PI' – will start being put together towards the end of this year. It will be a compilation of the experience of companies in PI.

Sylvaine Neveu of Rhodia in France introduced a proposed project for the EU called IMPULSE (sylvaine.neveu@eu.rhodia.com). This also at the time involved the University of Nancy and Siemens Axiva. The proposed project is entitled: 'Integrated

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[1] Be first to find out the background to these calls, and gather hints on how to improve your chances of success, by attending the PIN meetings. (Date extended by one week from 28 June)

[2] PIN is now associated with several expressions of interest for networks and integrated projects, submitted to the EC by the deadline of 7 June.

[3] A book describing the study tour and containing other lectures on PI – Process Industry in Progress – has been published by TU Eindhoven, reference ISBN 90-386-2933-8.

multi-scale process units with locally structured elements'. Essentially this means, explained Sylvaine, that production units are created by integration and interconnection of a wide range of small scale units, leading to a 'macro' production plant/device. She said that a meeting on the subject would be held at CEFIC on 5 June (attended by Roshan Jachuck of Newcastle University – r.j.j.jachuck@ncl.ac.uk).

John Hare (john.hare@hsl.gov.uk) discussed work at the Health & Safety Laboratory, Buxton, on laboratory scale sulphonation of arenes. John stated that runaway reactions could occur in novel reactors, and the HSL research project was directed at understanding what could happen. The work used standard calorimeters to obtain design data on mass transfer in liquid-liquid reactions, and the data could be used for HEX-reactors. With regard to the sulphonation process, reaction rates were measured and the dependence on flow, temperature etc. was investigated. The safety implications were being studied.

John invited partners to join this work. He was particularly looking for mass transfer specialists, industrial companies who carry out arene sulphonation, and intensified reactor manufacturers. *The overheads from John's presentation are available on the PIN web site.*

Retaining the safety interest, David Edwards of Loughborough University (d.w.edwards@lboro.ac.uk) discussed an approach to safety involving identifying hazards at an early stage and then changing the design to remove them. A comparison could be made with existing conventional plant, and data from plants was needed to help to establish the methodology. David invited industry to provide plant data. For more information, contact David or Roshan Jachuck (R.J.J.Jachuck@ncl.ac.uk).

Yuying Yan of Nottingham Trent University (yuying.yan@ntu.ac.uk) then introduced his work on transport phenomena at a bubble-liquid interface. He suggested that the simulation procedures developed could have a variety of applications relevant to PI, including bubble-related chemical rectors, micro-bubble formation, sliding bubbles in flow boiling and drops and particles in liquids. Yuying described the novel aspects of the approach and gave results. The numerical method used was validated and illustrated on two examples – natural convection and deformed bubbles.

The next three *impromptus* related to the EC call for Expressions of Interest (EoI's). Michel Demissy of INERIS in France (michel.demissy@ineris.fr) invited participation in an EoI on hazard reduction in chemical processes. One aspect of research at INERIS relevant to this was an investigation with Corning SA into whether some batch reactions could be made continuous in small glass reactors. The aim of the EoI is to identify reactions and candidate processes, do kinetic evaluations, study the options for continuous processes, design a micro-reactor, do experiments and then carry out a hazard reduction analysis. Andrew Green of BHR Group (agreen@bhrgroup.co.uk) then outlined the BHRG EoI on integrated projects in the area of PI. The aim is to look for big initiatives with a big impact. Roshan Jachuck (r.j.j.jachuck@ncl.ac.uk) then introduced the Network of Excellence EoI, of which PIN is a part.[4]

Finally, Ming Tham, (Ming.Tham@ncl.ac.uk) who organises and maintains our web site, introduced the new format. He said that the web was being made the central point of focus of PIN for the outside world. He stressed the need for feedback, articles, links etc. to be added to the site, so that members can extract maximum benefit. Please contact Ming.

Most members then visited the refurbished laboratories, seeing in particular the many rotating reactor plants in purpose-built cells.

Colin Ramshaw thanked all present and closed the meeting.

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Minutes prepared by David Reay from his notes, 10 June 2002.

[4] All three proposals have been submitted to the EC. There will be opportunities to join these later in the year when the full competitive proposal stage is reached. Contact those involved if you are interested.