Institution: Imperial College London

Unit of Assessment: 12 Aeronautical, Mechanical, Chemical and Manufacturing Engineering

Title of case study: 11. Organic Solvent Nanofiltration – A New Paradigm for Molecular Separations in Organic Liquids

1. Summary of the impact (indicative maximum 100 words)

Organic solvent nanofiltration (OSN) is a membrane separation technology used for separating molecules present in organic solvents. Research in the Livingston group has resulted in the creation of membranes with exceptional stability in organic solvents, coupled to high flux and excellent rejection performance. These membranes have been developed through to commercial products, and are manufactured by Evonik MET Ltd in the UK in a purpose-built facility in West London.

For many separations OSN uses ten times less energy than thermal methods, and can process molecules at low temperature. Through Evonik MET, OSN membranes and test equipment derived from the Imperial research have been supplied to over 100 customers including many of the major global chemical and pharmaceutical companies. For his work on OSN, Andrew Livingston received the 2009 Silver Medal of the Royal Academy of Engineering awarded "…to recognize an outstanding and demonstrated personal contribution to British engineering, which has resulted in successful market exploitation…" [7]

2. Underpinning research (indicative maximum 500 words)

Organic liquids are ubiquitous in chemical science based industries, from refining to pharmaceutical production. It is generally accepted that 40-70% of capital and operating costs in these industries are dedicated to separations; and a substantial fraction of this cost is related to the processing of organic liquids, both as product streams and solvents. Membranes have the potential to provide game changing alternatives to conventional concentration and purification technologies such as distillation, liquid extraction, adsorption and chromatography. In Organic Solvent Nanofiltration (OSN), a pressure is applied across a nanoporous film, so that solvents and small solutes in contact with the film permeate across, while larger molecules are unable to enter the membrane. Thus large and small molecules are separated, with no liquid-vapour phase change. The key features are that: (a) the energy requirement is an order of magnitude lower than for thermal techniques, and; (b) the molecules in the solution do not have to be heated. In spite of these clear advantages, prior to 2008 OSN had had a limited impact in industry.

Research at Imperial College into OSN has addressed three broad challenges: (i) developing applications of OSN to organic processes; (ii) understanding transport phenomena in OSN systems so as to enable process design; and (iii) most demanding, creating new OSN membranes to overcome poor stability in solvents. Addressing all three challenges has been essential to underpinning the impact that OSN technology has made over the last 5 years. This research has been carried out between 1999-present by postgraduate students and post-docs led by Professor Andrew Livingston FREng, who has been in post from 1990–present.

Research into applications of OSN membranes in organic processes began at Imperial in 1999 using commercial membranes which had been (then recently) developed for a specific lube oil dewaxing process by WR Grace. This research showed how OSN membranes could be used in homogeneous catalyst recycling, impurity removal, solvent exchange and solute concentration [1,2]. Livingston was invited to give various keynote/plenary lectures on these applications, including at Euromembrane 2004 (Hamburg) and ICOM 2005 (Seoul). The work on applications also highlighted that the limited number of available membranes were inadequate for the requirements of most potential applications, due to very limited stability in organic solvents.

Research into transport phenomena considered how the solvents, solutes and membranes interact and how these interactions determine process performance. The thermodynamic properties of the solutions from which molecules are to be separated were shown to be important in determining flux and rejection [3]. Research into concentration polarisation in both flat sheet and spiral wound modules [4] was used to provide underpinning data for process scale up and implementation, providing confidence that the performance of OSN technology could be reliably estimated, and this work has been presented at the prestigious Gordon Research Conference on Membranes: Materials and Processes (London NH, 2006) and Euromembrane 2009 (keynote).

Research into membrane fabrication and the influence of membrane fabrication parameters on membrane performance started in 2003. A key discovery was that membranes fabricated from polyimide could be cross-linked post-fabrication by contacting them with a solution containing diamines to give polyamide-imides, which provide outstanding resistance to organic solvents. Surprisingly, this stability was gained without any substantial effect on the permeation rates of molecules through the membranes [5]. A second key finding was that the permeation pathways in the membranes could be manipulated through directed control of the formulation and fabrication conditions, and specifically through the addition of a co-solvent to polymer dope solutions [6]. This research has led to presentations at the North American Membrane Society (NAMS) in 2012 (plenary) and ICOM 2011 (keynote) and the Gordon Research Conference on Membranes: Materials and Processes (London NH, 2012).

3. References to the research (indicative maximum of six references)
* References that best indicate quality of underpinning research.

*[1] J.T. Scarpello, D. Nair, L.M. Freitas dos Santos, L.S. White, A.G. Livingston, "The separation of homogeneous organometallic catalysts using solvent resistant nanofiltration", Journal of Membrane Science, Vol 203, pp. 71-85, (2002) DOI: 10.1016/S0376-7388(01)00751-7

[2] C. Pink, H-T. Wong, F. Ferreira, A.G. Livingston, "Recovery and Reuse of Ionic Liquids and Palladium Catalyst for Suzuki Reactions Using Organic Solvent Nanofiltration", Green Chemistry, Vol 8, pp. 373 – 379, (2006) DOI: 10.1039/B516778G

[3] L.G. Peeva, E. Gibbins, S.S. Luthra, L.S. White, R.P. Stateva, A.G. Livingston, "Effect of concentration polarization and osmotic pressure on flux in organic solvent nanofiltration", J.Mem Sci., Vol 236, pp.121-136, (2004) DOI: 10.1016/j.memsci.2004.03.004

[4] P. Silva, L.G. Peeva, A.G. Livingston, "Organic solvent nanofiltration (OSN) with spiral-wound membrane elements-Highly rejected solute system", Journal of Membrane Science, Vol 349, pp. 167-174, (2010) DOI: 10.1016/j.memsci.2009.11.038

*[5] Y.H. See-Toh, F.W. Lim, A.G. Livingston, "Polymeric Membranes for Nanofiltration in Polar Aprotic Solvents" Journal of Membrane Science, Vol 301, pp. 3-10, (2007) DOI: 10.1016/j.memsci.2007.06.034

*[6] Y.H. See-Toh, M. Silva, A.G. Livingston, "Controlling molecular weight cut-off curves for highly solvent stable organic solvent nanofiltration (OSN) membranes", Journal of Membrane Science, Vol 324, pp. 220-232, (2008) DOI: 10.1016/j.memsci.2008.07.023

4. Details of the impact (indicative maximum 750 words)

The research at Imperial College produced membranes at a small pilot scale, and has resulted in patents and patent applications covering the membranes and their means of fabrication. Key among these is UK Patent GB2437519 [8] and the resulting international patent family, which protects the cross linking of polyimide membranes to make them stable in a wide range of organic solvents. Intellectual property on membrane fabrication and applications of the membranes was assigned from Imperial College to Imperial Innovations plc, the technology transfer company set up by Imperial College. Innovations then licensed the findings of the OSN research to an Imperial College spin-out company, Membrane Extraction Technology (MET) Limited. MET's business goal was to develop and commercialise the OSN technology. To do this MET developed a manufacturing process for fabricating the first generation of OSN membranes based on cross linked polyimide, including the stages of membrane formation and post formation processing to crosslink the membrane and impregnate it with conditioning agents. Techniques for wrapping spiral wound membrane modules were developed by MET, together with highly solvent stable adhesives for these modules. This resulted in the creation of the first spiral wound membrane products in 2009, which have been commercialised under the registered trademark Duramem^{TM} [9]. This development activity was supported by five PhD students from the Livingston laboratory being employed by MET.

In parallel with these product development activities, MET undertook a dissemination campaign in which the Imperial OSN technology was promoted at membrane and chemical conferences and trade shows (eg ACHEMA (DE), ChemShow (USA), Informex (US), CPHi (Europe), as well as through individual visits to customers. MET developed a lab bench test cell (the METcell) in which the OSN membranes could be evaluated and tested by interested end users. This was followed by an extension to a cross flow cell system, and a bench top testing unit capable of handling small spiral modules. This activity generated an enormous interest in the technology from process development and separations professionals in the chemical and pharmaceutical industries and more than 100 MET cell units have been sold as at June 2013. The customers for these cells include many major pharmaceuticals and chemical companies, and companies with commercial interests in solvent processing, for example in the electronics industry.

A key milestone in the impact of OSN technology was the installation of Duramem^{TM} spiral wound membrane modules in a plant at GSK Ulverston in 2009, where they were installed in a facility regulated under GMP (Good Manufacturing Practice) [10]. In this application, the membrane modules were applied to the concentration and recovery of a valuable active pharmaceutical ingredient. In several cases customers who initially began with test cell products have constructed pilot and production scale processing units for OSN, including Johnson and Johnson [11] and Merck AG [12]. The OSN membranes have also proven highly effective in the purification of solvents for re-use, for example in the electronics industry.

The growing success of the OSN technology attracted a business unit of Evonik who manufacture polymers, Evonik Fibres, to acquire MET for several million euros. The acquisition completed in March 2010 to form Evonik MET [13]. This was a positive outcome with the shares being acquired at a substantial multiple of the price MET investors had paid for them. Evonik have so far invested in a new premises and a new membrane manufacturing facility in Greenford, West London, operational in June 2011, and have tripled the number of employees globally from 10 to 30 since the acquisition. The OSN membranes invented at Imperial College are now produced commercially in this new facility as large process scale 8” x 40” spiral wound elements. The compounded annual growth rate of Evonik MET in 2013 is in excess of 40%, with nearly all going to export. To support these exports, Evonik MET has appointed Business Development Managers in the USA, India, China, and Brazil, and has applications development labs in the USA and India. Evonik Vice President for Fibres and Membranes, writes [14]:

> *“As one of the world leading specialty chemical companies, and also the owner of Evonik MET, we have unique insight into the value and impact of OSN technology in chemistry-based businesses. This is because we see both internal applications within Evonik’s own businesses, and at the same time are involved in providing the technology to customers in other chemicals businesses. The value proposition for the industry and its applications is high. As an example,£1M of sales is creating either process optimization potential or new product sales of at least £10M and beyond, which makes the OSN technology a significant enabling technology and game changer in the chemical and adjacent industries. This is a truly motivating figure and is why, with the marketing and technical power of Evonik behind it, this technology is making a major impact across the spectrum of organic chemical processes”.*

Relating this to MET/Evonik MET OSN turnover of £4M from 1 Jan 2008-31 Dec 2012 [15] indicates that the technology is already providing in excess of £40M-£80M of value to chemical and related industries. The beneficiaries of the Imperial OSN research therefore include the global chemical sciences based companies that purchase the products derived from the research, and the UK economy which gains from a newly established UK based manufacturing industry, in which the UK is a clear world leader.

5. Sources to corroborate the impact (indicative maximum of 10 references.)

[7] http://www.raeng.org.uk/news/releases/shownews.htm?NewsID=509 Archived at https://www.imperial.ac.uk/ref/webarchive/psf on 17/09/2013

[8] UK Patent GB2437519 Integrally skinned asymmetric polyimide membrane United Kingdom Patent 2437519-A (priority date 28 April 2006)

[9] Evonik Website: http://duramem.evonik.com (Archived at https://www.imperial.ac.uk/ref/webarchive/mrf on 5th September, 2013)

[10] Technical Operations Manager, GlaxoSmithKline to confirm the use of OSN technology by GSK

[11] Vice President, Johnson and Johnson Pharmaceutical Research & Development to confirm the use of OSN technology by Johnson and Johnson.

[12] Conference Presentation by Development Engineer, Merck KGaA which confirms the use of OSN technology by Merck

[13] Evonik Acquisition Press Release 2010 http://corporate.evonik.com/en/media/archive/pages/news-details.aspx?newsid=13191 Archived on 29/10/2013 at https://www.imperial.ac.uk/ref/webarchive/v1f

[14] Vice President, Fibres and Membranes, Performance Polymer, Evonik Industries to confirm the impact and value of OSN across the spectrum of organic chemical processes

[15] Evonik Membrane Extraction Technology Limited Directors Report and Financial Statements for the Year Ended 31 December 2012 - filed at Companies House, and reports for previous years available at Companies House.