Hiden Isochema
Advancing Sorption Analysis

Newsletter • Autumn 2014

A SPOTLIGHT ON: IONIC LIQUIDS

Earlier this year we sponsored ILSEPT, a fantastic conference chaired by Dr Mark Shiflett of DuPont and Professor Edward Maginn of the University of Notre Dame in the US. The program focused on the use of ionic liquids (ILs) in separation and purification technology. Our primary US representative John Bullis attended, together with Dr Mark Roper from the UK, and it provided us with an ideal opportunity to showcase our new XEMIS high pressure sorption microbalance.

"IGA instruments are now firmly established as the tool of choice for this application"

In recent years, there has been an immense amount of interest in ILs, and their gas and vapor sorption properties have been widely investigated by researchers working in the field. Furthermore, our IGA instruments are now firmly established as the tool of choice for this application[1] and have been widely used for vapor-liquid equilibrium (VLE) studies over the last decade or so.[2-5]

What is less common, however, is the use of our other instruments for the study of these materials.

Our attention was therefore drawn to a recent article by a team of researchers from the Laboratoire Polymères, Biopolymères et Surfaces at the Université de Rouen in France and their collaborators in Algeria and Ukraine.[6] In this study, Professor Stéphane Marais and his co-workers examined the sorption and permeation properties of supported ionic liquid membranes (SILMs) for the separation of water and volatile organic compounds (VOCs).

Supported liquid membranes (SLMs) typically contain organic solvents but this can result in instability due to solvent loss. As an alternative, ILs show great promise for the production of more stable SLMs because of their unique properties, in comparison with other solvents, including their negligible vapor pressure, high chemical and thermal stability, and a high capillary force, which minimizes their removal from the porous membrane support. Of course, for separation applications it is essential to be able to characterize the behavior of a given SILM with respect to the relevant gases and vapors.

The particular SILMs in this study were produced by immobilizing [bmim][BF_4], [bmim][PF_6] and [hmim][PF_6] in

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porous membranes prepared from a commercially available polymer, Matrimid 5218.

In contrast to the majority of previous IL studies featuring Hiden Isochema gas and vapor sorption instrumentation, Professor Marais and his co-workers used an IGAsorp. This system operates at ambient pressure in a flowing stream of nitrogen, which is mixed with a wet stream, in varying quantities, to provide precise control of the partial vapor pressure of the active species in the measurement chamber.

In the study, the equilibrium water, ethanol and cyclohexane sorption isotherms were determined for both the isolated ILs and the host membrane at 25°C. [bmim][BF_4] was found to absorb the most water and ethanol, and also exhibited the highest selectivities. The membrane containing this ionic liquid was thus selected for further testing.

A moisture vapor transmission rate (MVTR) cell was then used to determine the permeation rates of the three vapors through the selected membrane. The results showed that water had the highest permeability, followed by ethanol and then cyclohexane. They also allowed the calculation of ideal selectivities between each pair of species, which were found to be particularly high for water/cyclohexane and ethanol/cyclohexane.

As well as being a great example of the use of our instruments, we also think this is probably the first example of the use of an IGAsorp to study the sorption of water and hydrocarbon vapors by ILs, but if you know differently, please let us know.

References

[1] K. N. Marsh, J. F. Brennecke, R. D. Chirico, M. Frenkel, A. Heintz, J. W. Magee, C. J. Peters, L. P. N. Rebelo and K. R. Seddon, Pure and Applied Chemistry 81(5) (2009) 781-790

[2] J. L. Anthony, E. J. Maginn and J. F. Brennecke, Journal of Physical Chemistry B 105 (2001) 10942-10949

[3] J. L. Anthony, E. J. Maginn and J. F. Brennecke, Journal of Physical Chemistry B 106 (2002) 7315-7320

[4] M. B. Shiflett, A. Yokozeki, Industrial & Engineering Chemistry Research 44(12) (2005) 4453-4464

[5] M. B. Shiflett, A. Yokozeki, AIChE Journal 52(3) (2006) 1205-1219

[6] A. Dahi, K. Fatyeyeva, D. Langevin, C. Chappey, S. P. Rogalsky, O. P. Tarasyuk, A. Benamor and S. Marais, Journal of Membrane Science 458 (2014) 164-178

14th International Symposium on Metal-Hydrogen Systems: Fundamentals and Applications

In July, over 400 delegates enjoyed unusually warm and sunny weather in Salford and participated in five days of fantastic oral and poster presentations at MH2014.

Richard Meftah, Creative Imaging

The technical program featured a number of different sessions within three tracks – Fundamentals, Materials and Applications – covering contributions ranging from the fundamental behavior of hydrogen in binary hydride thin films to the development of practical hydrogen storage units. This packed schedule was supplemented with a varied social program; delegates enjoyed a typically English cream tea at one of three local stately homes and a conference dinner held at Old Trafford, home of Manchester United. The conference proceedings are due to be published soon in a special issue of the *Journal of Alloys and Compounds*.

Hiden Isochema was proud to be a Silver Sponsor of the event and was pleased to be able to provide further organizational support, with Dr Darren Broom being an active member of the local organizing committee. Furthermore, the organizers were delighted with the success of the conference as a whole and have now handed over the baton to Professor Andreas Züttel who will chair the next MH conference in Switzerland in 2016.

An impressive set of photos from the event can be found at: mh2014.salford.ac.uk/photos

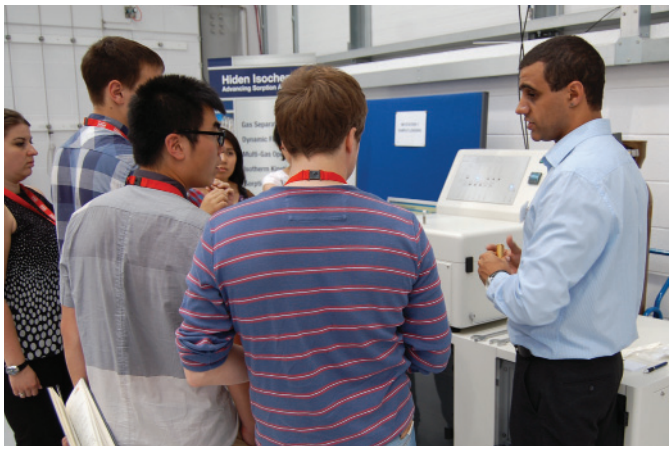
MH2014 Summer School a Great Success

The summer school on the characterization of hydrogen-material interactions that we co-hosted with the University of Salford, prior to the main MH2014 conference, was a great success and was attended by 39 students, from institutions in 15 different countries.

Day one of proceedings began with introductory lectures at the University of Salford buildings at MediaCity UK in Manchester, chaired by Dr Martin Owen Jones (ISIS neutron source, UK), one of the event's co-organizers.

The day featured a series of talks covering the behavior of hydrogen in materials. The invited speakers included Professor Andreas Züttel (EPFL and EMPA, Switzerland), Dr Paul Anderson (University of Birmingham, UK), Dr Theodore Steriotis (NCSR "*Demokritos*", Greece), and Dr Nuno Bimbo (University of Bath, UK).

The second day took place at our headquarters in Warrington. Following a welcome and introduction, Drs Darren Broom and Mark Roper of Hiden Isochema gave



talks entitled "*Manometric Measurement of Hydrogen Sorption by Materials*" and "*Gravimetric Measurement of Hydrogen Sorption by Materials*".

These complementary talks addressed the two main macroscopic methods for the determination of hydrogen uptake by materials from a practical perspective and covered many of the issues that can affect these measurements.

The rest of the day was then spent on practical demonstrations of the measurement of hydrogen sorption using a number of different Hiden Isochema instruments. This allowed a further, thorough discussion of the important considerations for the performance of accurate hydrogen sorption measurements.

In addition to the practical demonstrations, the students also visited Hiden Analytical, a well-known manufacturer of quadrupole mass spectrometers and Hiden Isochema's parent company.

Following the intensive day at Hiden, the students were then able to take a break from the scientific program and enjoyed a one day excursion to the picturesque village of

Grasmere in the Lake District, which is famous for being the home (from 1799 to 1808) of William Wordsworth, one of the best known of England's Romantic Poets.

The summer school then returned to MediaCity UK for the final day, which was chaired by Dr Dan Bull (University of Salford, UK), another of the co-organizers. This featured a series of lectures by invited speakers who each specialize in different microscopic characterization techniques, including Inelastic Neutron Scattering (INS), powder diffraction, and Nuclear Magnetic Resonance (NMR), Raman and Infrared (IR) spectroscopies.

The speakers on the Sunday included Dr Sabrina Sartori (University of Oslo/UNIK and IFE, Norway), Dr Timmy Ramirez-Cuesta (Oak Ridge National Laboratory, USA), Dr Andreas Borgschulte (EMPA, Switzerland) and Professor Mark Conradi (Washington University, USA).

The feedback that we have received has been very positive. We also hope that the students both enjoyed and benefited from the varied program on offer.

For more details, please see our blog: isochema.com/summerschool

News From Our Blog

- IGA installed at prestigious Imperial College London
- University of Bath present latest Isotope Exchange Apparatus data
- Hiden Group launch new landing page

To find out more about any of these stories, or to read our other blog items, please visit:
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NEW Applications Articles:

"Separation of rare gases and chiral molecules by selective binding in porous organic cages"
L. Chen et al. Nature Materials 13 (2014) 954-960

An impressive study featuring both a Hiden Isochema automated breakthrough reactor and an IGA to investigate the removal of low concentrations of Kr and Xe from air using the organic cage molecule CC3.

"Polymeric molecular sieve membranes via *in situ* cross-linking of non-porous polymer membrane templates"
Z.-A. Qiao et al. Nature Communications 5 (2014) 3705

A team from Oak Ridge National Laboratory and the University of Tennessee in the US use an IGA to characterize the equilibrium uptake of CO_2 and N_2 by porous hypercrosslinked polymer membranes for gas separation.

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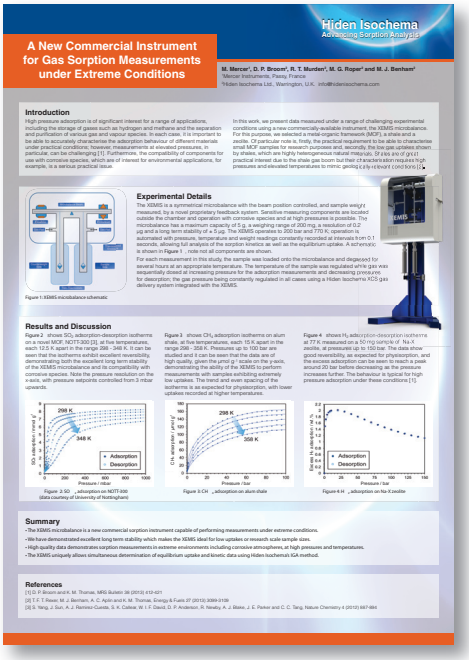
XEMIS Conference Posters



We have been busy this year promoting our new XEMIS microbalance and it continues to attract great interest from both new and existing customers. Talks have been given and posters presented at several international conferences including the French Carbon Group meeting held in Orleans, France; at Characterization of Porous Solids (COPS-X), which was held in Granada, Spain and the Federation of European Zeolite Associations (FEZA) 2014 meeting held in Leipzig, Germany. In November Dr Darren Broom will be heading off to AIChE 2014 in Atlanta, USA, to present a poster entitled "A New Commercial Instrument for Gas Sorption Measurements Under Extreme Conditions", which features the latest XEMIS data produced in our in-house applications laboratory.

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