

NECZA 2010 PROGRAM

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The 14th Meeting of the North-East Corridor Zeolite Association

Friday, December 10, 2010

Lynch Seminar Room, Department of Chemistry
University of Pennsylvania

8:20 - 9:20 Registration, Refreshment, Poster set-up

9:20 - 9:25 Meeting opening and Introduction

9:25 – 10:00 Douglas Ruthven (University of Maine)
Recent Progress in Studies of Intracrystalline Diffusion in Zeolites and MOFs

10:00 – 10:25 Charles G. Coe (Villanova University)
Structural Effects on the Adsorptive Properties of Carbon Molecular Sieves

10:25 - 11:00 Break, Posters

11:00 – 11:30 Laszlo Nemeth (UOP LLC/Honeywell)
*Synthesis, Characterization and Catalytic Application for Sn-Beta Zeolite
as a New Unique Single Site Heterogeneous Catalyst*

11:30 – 12:10 Edith Flanigen & Stephen T. Wilson (UOP LLC/Honeywell)
Aluminophosphate-Based Molecular Sieves; A Review of the History, Chemistry, and Applications

12:10 - 1:20 Lunch, Posters

1:20 - 1:50 Steven L. Suib (University of Connecticut)
Porous Metal Oxide Molecular Sieves: Synthesis and Novel Applications

1:50 - 2:20 Lajos Balogh (Elsevier Nanomedicine)
Size, Shape, and Dynamic Porosity of Dendrimers

2:20 – 2:45 Break, Posters

2:35 – 2:45 Business Meeting

2:45 - 3:00 Selected poster presentation 1

3:00 - 3:15 Selected poster presentation 2

3:15 – 3:45 Dionisos Vlachos (University of Delaware)
TBA

3:45 – 4:15 Anne Gaffney (Air Liquid & The Langmuir Research Institute)
Pulsed-TPR and Complex Microporous Diffusion Characterization Using TAP Reactor System

4:15 Close of meeting

Oral Presentations

Recent Progress in Studies of Intracrystalline Diffusion in Zeolites and MOFs

Douglas Ruthven*

Professor Emeritus, Department of Chem. Engineering, University of Maine, Orono, ME 04469

A detailed understanding of intracrystalline diffusion is essential for the rational design of adsorption separation and catalytic processes. A review of the extensive literature of this subject reveals large discrepancies between the diffusivities measured, for similar zeolite samples, by different experimental techniques. In general the longer the “length scale” of the measurement the smaller the apparent diffusivity. Over the past five years as a result of the introduction of two new experimental techniques (interference microscopy and fluorescence microscopy) these discrepancies are now reasonably well understood.

Interference microscopy allows the transient intracrystalline concentration profiles to be measured in real time during adsorption or desorption from a single large zeolite crystal. The results show clearly the pronounced effect of surface and intracrystalline (defect) transport barriers in many crystals. The magnitude of the surface barriers is found to vary significantly between different crystals from the same sample and in some cases even between different surfaces of the same crystal! Clearly the old ideas that zeolite crystals can be considered as having a regular intracrystalline pore network of uniform pore size and that sorption rates are controlled mainly by internal diffusion in such a structure are not correct. Of the many different crystals examined only a small fraction show such behavior. The effects of surface resistance and internal barriers are much more important than had been generally assumed.

Fluorescence microscopy allows the movement of a single molecule to be tracked in real time. Such studies (with large molecules that cannot penetrate the intracrystalline pores) have shown that reaction occurs at distinct points on the crystal surface and there is a wide variation in the activities of different sites. With a reactant that can penetrate the pores of a ZSM-5 crystal we see the build up of products at particular points within the crystal. The accepted idea that a catalytic reaction can be modeled as being controlled by a combination of diffusion and reaction in a uniform structure is clearly far from the truth, at least for some systems.

* Much of the work discussed here was carried out by Professor Jorg Kaerger and his students at the University of Leipzig. The fluorescence microscopy data are from a recent review by M.B.J. Roefsaers et al.

Speaker's Bio

Professor Ruthven was born in Ernakulum, India. He attended Sidney Sussex College, Cambridge University from 1957 to 1961 and from 1963 to 1966. After several student awards he earned Ph. D. degree in 1966. He started to teach at the Department of Chemical Engineering at University of New Brunswick, Fredericton, N.B. Canada in 1966, raised to the rank of full professor by 1975, and also served as department Chair from 1993 till 1995. Following this he became Chair of the Department of Chemical Engineering at University of Maine, Orono ME in the period 1995-2003 and he is still professor of this university.

He authored and edited four seminal books in the area of adsorption and separation sciences and published approximately 300 papers. Dr. Ruthven's research has been acknowledged with numerous awards beginning with DSc degree at University of Cambridge in 1988. He was elected to the Royal Society of Canada in 1989 and earned UNB merit awards in 1986-87 and 89-90. The Max Planck Institute and Humboldt Foundation awarded him the Max Planck Research Prize jointly with Professor Jorg Kroger in 1993, the Humboldt Forschungpreis in 2002, and a Senior Research Fellowship from 2009. He also served as President of the International Adsorption Society in the period of 1998-2001 and earned the Century of Achievement Award from the Canadian Society for Chemical Engineering in 1999. This latter society organized a Symposium in Professor Ruthven's Honor in Ottawa in 2009.

Structural Effects on the Adsorptive Properties of Carbon Molecular Sieves

Charles G. Coe

Air Products and Chemicals, Inc., 7201 Hamilton Blvd, Allentown, PA 18195 (presently at Chemical Engineering Department, Villanova University, PA)

The structural variability of microporous adsorbents provides a means for controlling the equilibrium and kinetic adsorption parameters over a wide range. Specific structural features can be combined to provide materials having adsorption properties best suited for a particular gas separation. After giving an introduction to adsorptive separations, examples from our past research at Air Products on the development of carbon molecular sieves for producing nitrogen from air will be reviewed to illustrate the remarkable importance of tailoring porosity for kinetic based separations. A multidisciplinary approach involving experimental synthesis, modeling, and extensive analytical characterization elucidated some new insights leading to improved air separation processes as well as other new applications.

Carbon molecular sieves (CMS) are a subgroup of activated carbon which allow separation of nitrogen from oxygen on a kinetic basis. The effective micropore size of the CMS can be controlled by selective hydrocarbon cracking to narrow only the pore mouth preserving the usable O₂ uptake rate. Remarkably, deposition of only 0.1% (by weight) of carbon can convert an unselective CMS to a CMS with O₂/N₂ selectivity of >20. In addition to the pore mouth structure, the overall pellet density and porosity are important. Based on our understanding we developed a model that explained the complete loss of O₂ selectivity when certain CMS materials were crushed. This model aided in developing superior carbons for use in N₂ PSA. Using microporous carbons having higher densities we were able to prepare N₂PSA adsorbents with 20% higher O₂ capacity using high temperature pyrolysis followed by controlled oxidation. This review will hopefully reinforce the critical importance of using fundamental approaches to optimize adsorbent properties for a given application.

Speaker's Bio

After obtaining his PhD in inorganic chemistry from Carnegie Mellon University, Dr. Coe joined the Chemical Additives division of Air Products and Chemicals. Early in his career he transferred to the corporate research group at the company headquarters where he developed an extensive expertise in molecular sieve science. For many years he teamed with project leaders across business units to enable the development of improved adsorbents and catalysts. Dr. Coe was also instrumental in developing new analytical methods and instrumentation to obtain fundamental information on small experimental samples. Before retiring from Air Products, he was named a strategic technologist for the Corporation and provided internal consultation on a broad range of materials characterization issues involving catalysts and adsorbents. His accomplishments have led to 35 issued US patents, 38 peer-reviewed publications, and numerous invited lectures. He recently received the 2010 Catalysis Club of Philadelphia Award.

Since retiring, Dr. Coe has joined the Chemical Engineering faculty at Villanova University and is sharing his knowledge with the next generation of engineers and scientists.

Synthesis, Characterization and Catalytic Application for Sn-Beta Zeolite as a New Unique Single Site Heterogeneous Catalyst

Laszlo T. Nemeth

UOP LLC, Des Plaines, IL 60016, USA

Sn-Beta zeolite (Beta structure with framework incorporated tin (Sn)) has been synthesized, characterized and tested as a heterogeneous catalyst. In order to achieve the high selectivity with heterogeneous catalysts, isolated well-defined active sites with the appropriate orbital orientation and geometrical structure have to be prepared. We show that the introduction of paired framework Sn(IV) sites, exclusively in T5/T6 positions of a crystalline silica nanoporous material (Beta zeolite), produces the specific electronic orbital orientations and specific spatial constraints for coordinating carbonyl compounds, and performing the industrially relevant oxidation of aldehydes and ketones into esters using H₂O₂ as oxidant. The selectivity observed was only previously achievable using enzymes. This result provides a major step forward in the design, control, and characterization of uniform active sites in heterogeneous catalysis, *a priori* required for the ultimate goal of 100 percent selectivity resulting in cost-effective and environmentally sound chemistry.

Specifically this Sn-Beta heterogeneous catalyst was used for the Baeyer-Villiger reaction with hydrogen peroxide. Cyclic ketones are transformed into the corresponding lactones with very high selectivity. The new oxidation system can also be used for unsaturated ketones which are oxidized to the corresponding unsaturated lactones with very high chemoselectivity. The oxidation mechanism has been elucidated by in-situ IR spectroscopy and ¹⁸O labeling experiments. It is proposed that the carbonyl group is activated selectively by polarization by the Sn Lewis acid, followed by the attack of hydrogen peroxide. The reaction goes through an intermediate of the Criegee type.

Converting sugars in aqueous media and other potential applications of Sn-Beta zeolite will also be discussed.

Speaker's Bio

Laszlo Nemeth is Senior R&D Associate in Corporate Research at UOP (Des Plaines IL) and holds a joint appointment at University of Illinois at Chicago as adjunct professor. He joined UOP in 1992 where he invented a wide range of new materials, including oxidation catalysts, solid superacids, and modified UOP Zeolytic Materials. He also intensively studied the related chemistry of liquid phase oxidations, carbonylation reactions, and hydrocarbon transformations. Based on this work he has published 32 UOP patents, 17 foreign patents, and 43 peer reviewed scientific papers.

In 1999 and 2001 Dr. Nemeth spent his UOP sponsored sabbatical leaves with George Olah (Nobel Prize in Chemistry) at University of Southern California and Avelino Corma at University of Valencia and studied the chemistry of various superacids with their research groups. He completed the Dale Carnegie management courses and obtained Six Sigma Black Belt certification. Dr. Nemeth earned B. S. Chemistry and Doctor of Science degrees in Hungary.

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ALUMINOPHOSPHATE-BASED MOLECULAR SIEVES A REVIEW OF THE HISTORY, CHEMISTRY, AND APPLICATIONS

Edith M. Flanigen and Stephen T. Wilson

UOP LLC, a Honeywell Company

A review will be presented of the story of the discovery of the aluminophosphate-based molecular sieves at Union Carbide Corporation in the late 70's and early 80's, in response to a management challenge to discover a new generation of molecular sieve materials. The methodology of a small team of researchers to devise a strategy based on new framework compositions and a periodic table concept, along with its implementation, will be detailed.

The first success resulted in the aluminophosphate family of materials comprising 15 novel structures and an AlPO_4 framework composition. The successful synthesis was based on a novel reactive gel "recipe" unlike that typically used in zeolite synthesis. Subsequent implementation of a periodic table strategy and multinary frameworks resulted in the SAPO, MeAPO and EIAPPO families of materials, denominated the AlPO_4 -based molecular sieve generation. Thirteen elements in the periodic table were incorporated into the aluminophosphate-based framework. The new generation comprises over two dozen structures and over two hundred compositions.

The synthesis, chemistry and properties of this new generation will be compared to those of the conventional zeolites. The properties delineated include pore size, surface selectivity, acidity and stability.

Emphasis will be on the SAPO materials which have received most interest in commercial or near-commercial applications, including catalytic dewaxing and methanol to light olefins (MTO). Some insights into the difficulty of achieving successful commercialization of a new material will be highlighted.

Speakers' Bio

Edith M. Flanigen joined the research staff of Union Carbide Corporation in Tonawanda, NY in 1952 and served as a research scientist with Union Carbide until 1988, when she transferred to UOP, a joint venture of Union Carbide and Allied Signal. She retired from UOP in 1994, and is a consultant for UOP LLC, now a Honeywell Company.

Dr. Flanigen has attained international prominence for her work in the fields of silicate chemistry and the chemistry of zeolites and molecular sieve materials. Her accomplishments include synthesizing new molecular sieve materials for application as adsorbents and catalysts; inventing the hydrothermal emerald synthesis process; and pioneering the use of mid-range infrared spectroscopy for interpreting zeolite structures. She led the Union Carbide Research Group that discovered new generations of molecular sieve materials in the late 70's and 80's, including families of aluminophosphates, silicoaluminophosphates, and other novel aluminophosphate-based molecular sieves. This discovery laid the groundwork for an explosion in the discovery of new compositions and structures of molecular sieves from the 1980's through the 2000's. Over two dozen elements have been incorporated into microporous oxide frameworks and over 100 unique structures reported.

Dr. Flanigen has authored or co-authored over 36 publications and has been granted 109 U.S. patents. She received Doctor of Science, *Honoris Causa*, degrees from D'Youville College in 1983, and Syracuse University in 2008. Her other awards and honors include: the Francis P. Garvan - John M. Olin Medal of the American Chemical Society, 1993; the Perkin Medal of the Society of Chemical Industry, American Section, 1992; the Chemical Pioneer Award of the American Institute of Chemists, 1991; and Election to the National Academy of Engineering of the United States, 1991. She shared the inaugural Donald W. Breck Award of the IZA in 1983, and received the inaugural IZA Award for long-term contributions to zeolite science in 1994.



Steve Wilson joined the research staff of Union Carbide Corporation in Tarrytown, NY in 1976. He has worked for 32 years in the area of molecular sieve research, first with Union Carbide, then with UOP, following the joint venture between Union Carbide and Allied Signal. His research interests include synthesis, characterization, and application development. He was part of a small team that discovered a new family of microporous aluminophosphates, or AlPOs, which lead to the discovery of the related SAPOs and metal substituted AlPOs. These materials fundamentally changed the scientific basis of world thinking on the possible compositions of microporous structures. One of these AlPO-family materials, SAPO-34, is now the active component of the catalyst in the methanol-to-olefin process. In 1983 Dr. Wilson shared the inaugural Donald W. Breck Award of the IZA, and in 2006 Steve received the ACS Award in the Chemistry of Materials. Dr. Wilson is co-inventor on 73 US patents and co-author of 36 publications. He earned a B.S. in Chemistry from Wake Forest University (1968) and a Ph.D. in Inorganic Chemistry from Harvard University (1975).

Porous Metal Oxide Molecular Sieves: Synthesis and Novel Applications

Steven L. Suib

University of Connecticut

This presentation will focus on synthesis and applications of porous metal oxide molecular sieves and layered materials. Pulsed laser deposition methods will be discussed in the preparation of molecular sieves. Applications of microwaves in synthesis and catalysis will also be covered. Some of the catalytic reactions being studied are selective oxidations, remediation of environmentally hazardous substrates, and potential use in biomass conversion.

Speaker's Bio

Steven L. Suib was born in western New York state where he obtained a B. S. degree in chemistry and geology at the State University College of New York at Fredonia. His graduate work was done at the University of Illinois at Champaign Urbana with Galen D. Stucky. His postdoctoral work was done at Illinois with Larry R. Faulkner. He joined the faculty of the Department of Chemistry at the University of Connecticut in 1980 where he is currently Board of Trustees Distinguished Professor. His research interests include solid state inorganic chemistry including studies of zeolites and microporous materials; physical chemistry; environmental chemistry including green syntheses, heterogeneous catalysis; plasma chemistry and catalysis; semiconductors; inorganic photochemistry; photocatalysis; ceramics, composites, preparation and characterization of these systems using structural, crystallographic, surface, electrochemical, luminescence, microscopic and EPR techniques. He is a member of the American Chemical Society (ACS), the American Ceramic Society, Phi Lambda Upsilon, Phi Kappa Phi, Sigma Xi, and the Materials Research Society. He teaches general, inorganic and environmental chemistry. Recent awards include the Chemical Pioneer Award, AIC Fellow, CT Academy of Arts and Sciences, NASA Fellowship, and the Northeast Region Award of ACS.

SIZE, SHAPE AND DYNAMIC POROSITY OF DENDRIMERS

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The size and shape of organic macromolecules in solution are important for their motion and reactivity both *in vivo* and *in vitro*. Dendrimers are symmetrical organic molecules with a fractal structure that contain a large number of equivalent and regularly spaced internal and external functional groups with a high degree of branching, and at higher generations, with nearly spherical geometry. They are built around a core from connectors and branching units, which together form repetitive shell structures, called generations. Dendrimer materials are a mixture of these nearly identical soluble and stable (macro)molecules. Interior and/or exterior of dendrimers can be synthesized to be cationic, anionic, or non-ionic. Polyionic dendrimers can carry other organic molecules, inorganic ions or nanoclusters and are often subject of experimental and theoretical investigations. In solutions, depending on their family and generation, dendrimer molecules may behave as “small” branching molecules, “soft” colloids (draining networks, etc.), or “hard” organic nanoparticles. Size and shape are especially important for dendrimers, whose interaction with water and degree of hydration, is necessary knowledge to correctly interpret their capability to transport and release physiologically active small molecules for targeted drug delivery or imaging agents. Their size and shape depend on their end-groups and also on external parameters, such as pH and temperature.

Solvent molecules are able to penetrate into these molecules and bind to them forming a pseudo-biphase system different from both emulsions and micelles. Controlling key properties of dendrimers permits fabrication of a wide repertoire of higher order (2D, 3D) complex architectures that can be synthesized with pre-designed properties. These fundamental building blocks can further be organized and fabricated into higher order hierarchical structures, such as 0D (quantum dots), 1D (nanofibers) 2D (ultrathin multilayers) and 3D (bulk) nanomaterials.

In this talk porosity of dendrimers and its role in solubility and reactivity will be described and discussed in this talk based on theoretical calculations and experimental measurements.

Speaker's Bio

Lajos P. Balogh is the Principal of AA Nanotechnology Consulting, a business in Nanotechnology and Nanomedicine. He is a former Co-Director of the NanoBiotechnology Center and Director of Nanotechnology Research in the Department of Radiation Medicine at the Roswell Park Cancer Institute, and Professor of the University at Buffalo, SUNY.

Dr. Balogh received his Ph.D. from the Kossuth L. University (KLTE) in Hungary in Chemical Technology and was invited to the University of Massachusetts Lowell in 1991 as a Visiting Professor. Later he worked at the Michigan Molecular Institute as senior scientist, and had faculty appointments in the Departments of Internal Medicine and Biomedical Engineering at the University of Michigan Ann Arbor. He has authored or coauthored over 150 scientific publications and 12 patents in various disciplines.

Dr. Balogh is the Editor-in-Chief of the journal *Nanomedicine: Nanotechnology, Biology and Medicine* (*Elsevier*) and member of numerous USA Government, NGO, and International expert committees, including NIH NANO and EPA Nanotechnology study sections. He is also a member of the Steering Committee of the ANSI Nanotechnology Panel and serves on the US Technical Advisory Committee to the International Standard Organization on Nanotechnology (TC-229). He is one of the five Founders of the American Society for Nanomedicine. (<http://www.amsocnanomed.org>)

His present research interests involve the design, synthesis, and characterization of multifunctional dendrimers and hybrid nanodevices for the targeted delivery of smart contrast agents and anticancer drugs. He is interested in creating new nanoscience tools to understand fundamental biologic processes, and interactions of nanodevices with cells and tissues including toxicity, biodistribution, and pharmacokinetics.

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Pulsed-TPR and Complex Microporous Diffusion Characterization Using TAP Reactor System

**Rebecca Fushimi¹, Sergiy Shekhtman¹, Gregory Yablonsky², John Gleaves³,
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In this presentation two unique characterization experiments, pulsed-TPR and microporous diffusion modeling, are described using the Temporal Analysis of Products (TAP) reactor system. A new type of TPR (Temperature Programmed Reaction) study is presented where the surface concentration of reactants is held constant during the temperature ramp using a pulsed input from the TAP system. The native oxides from a simple Pd/SiO₂ catalyst were removed by pulsing CO and ramping the temperature. The reactant input was held constant yet the production of CO₂ showed a damped oscillation attributed to an oxygen supply mechanism from the solid phase.

The TAP pulse response technique also offers the ability to characterize multiple steps of the diffusion process in micro- and mesoporous materials. In the experiments described here, the energetics of the adsorption/desorption into/from of the pore mouth are distinguished from the adsorption on the external (plane) of the catalyst surface. Samples of SAPO and 13X zeolites along with a mesoporous alumina were tested using six different probe molecules: argon, butane, propane, ethane, propylene and ethylene. From this data complex models of surface and gas phase intracrystalline diffusion are described.

Speaker's Bio

Anne Gaffney received her Ph.D. in Physical Organic Chemistry in 1981 from the University of Delaware and her BA in Chemistry and Mathematics in 1976 from Mount Holyoke College in South Hadley, MA. She has been working in the Chemical Industry for 29 years for various companies, including ARCO Chemical Company, DuPont, Rohm and Haas, Lummus Technology and Air Liquide. She is the inventor or co-inventor of over 100 patents and author or co-author of over 80 publications. This year Dr. Gaffney was selected as an ACS Fellow and was also awarded the ACS Distinguished Service Award from the Petroleum Chemistry Division in 2010. Dr. Gaffney serves as the National Representative to the North American Catalysis Society, representing the Philadelphia Section. She is one of the co-founders of the non-profit organization, The Langmuir Research Institute, and will be presenting for LRI today. She recently joined Air Liquid as Director of Academic & Governmental R&D Partnerships North America.

Modern catalytic technologies for converting biomass to renewable fuels and chemicals

Dion G. Vlachos

*Department of Chemical Engineering, Center for Catalytic Science and Technology, and Catalysis
Center for Energy Innovation, University of Delaware, Newark, DE 19716-3110*

In this talk, modern catalytic-based technologies will be presented that may overcome challenges of traditional methods in converting biomass to renewable fuels and chemicals. The first is based on catalytic fast pyrolysis whereby biomass is heated at extremely fast rates in the presence of a zeolite catalyst, such as ZSM5, to impart shape selectivity in the product distribution. The technology leads to reduced char formation and a product distribution containing mainly aromatics. The second is based on biomass hydrolysis to simple derivatives, such as sugars, followed by new methods of deoxygenation using zeolites to value-added intermediates for chemicals or fuels.

Speaker's Bio

Dion Vlachos is the Elizabeth Inez Kelley Professor of Chemical Engineering at the University of Delaware. He is the Director of Center for Catalytic Science and Technology (CCST) and the Director of the Catalysis Center for Energy Innovation (CCEI), an Energy Frontier Research Center (EFRC) funded by the Department of Energy (DOE).

Dr. Vlachos obtained a five years diploma in Chemical Engineering from the National Technical Univ. of Athens, in Greece, in 1987. He obtained his MS and Ph.D. from the University of Minnesota in 1990 and 1992, respectively, and spent a postdoctoral year at the Army High Performance Computing Research Center, MN, after which he joined UMass as an Assistant Professor. He was promoted to an associate professor at UMass in 1998. He joined the Univ. of Delaware in 2000. He was a Visiting Fellow at Princeton University in the spring of 2000, a visiting faculty at Thomas Jefferson Univ. and Hospital in spring of 2007 and the George Pierce Distinguished Prof. of Chemical Engineering and Materials Science at the Univ. of Minnesota in the fall of 2007.

Dr. Vlachos is the recipient of an ONR Young Investigator Award and a NSF Career Award. He was selected as one of the top 100 engineers of 2009 and as a member of the [American Association for the Advancement of Science](#) (AAAS) in 2009. He is a member of the American Institute of Chemical Engineers, the American Chemical Society, the Combustion Institute, the Catalysis Society, and SIAM.

Dr. Vlachos' main research thrust is multiscale modeling and experiments along with their application to renewable energy, catalysis and portable microchemical devices for power generation, biofuels, growth of nanomaterials, and microporous thin films. He is the corresponding author of more than 190 refereed publications and has given more than 140 plenary lectures, keynote lectures, and other invited talks. He serves on the editorial advisory board of several journals (e.g., Industrial and Engineering Chemistry Research (I&ECR), Applied Catalysis A: General, The Combustion Institute, The Open Energy and Fuels Journal).