One day Workshop on the Fundamentals of Sol-Gel Technology

September 1, 2007- ENSCM Montpellier- France

In conjunction with Sol-Gel 2007
September 2-7, 2007
Montpellier - France

Workshop Schedule

09:00-09:15	*Introduction/ opening*
09:15-10:15	**Sol-gel alkoxide precursors** by Prof. V. Kessler (SLU, Uppsala, Sweden)
10:15-10:30	*Coffee Break*
10:30-11:30	**Structure Development in Sol-Gel Processing** by Prof. C.J. Brinker (Sandia National Laboratories, USA)
11:30-12:30	**Organic-Inorganic Hybrid materials** by Dr. G. Kickelbick (Vienna University of Technology, Austria)
12:30-14:00	*Lunch*
14:00-15:00	**Films from sol-gel** by Prof. M. Aegerter (JSST , Switzerland)
15:00-16:00	**Colloids and Nanoparticles made by sol-gel process** by Dr. T. Gacoin (Ecole Polytechnique, Palaiseau, France)
16:00-16:15	*Coffee Break*
16:15-17:15	**Porous sol-gel solids** by Prof. K. Nakanishi (Kyoto University, Japan)
17:15-18:15	**Sol-gel and Bio-technology** by Dr. T. Coradin (UPMC, Paris, France)

How to get to there

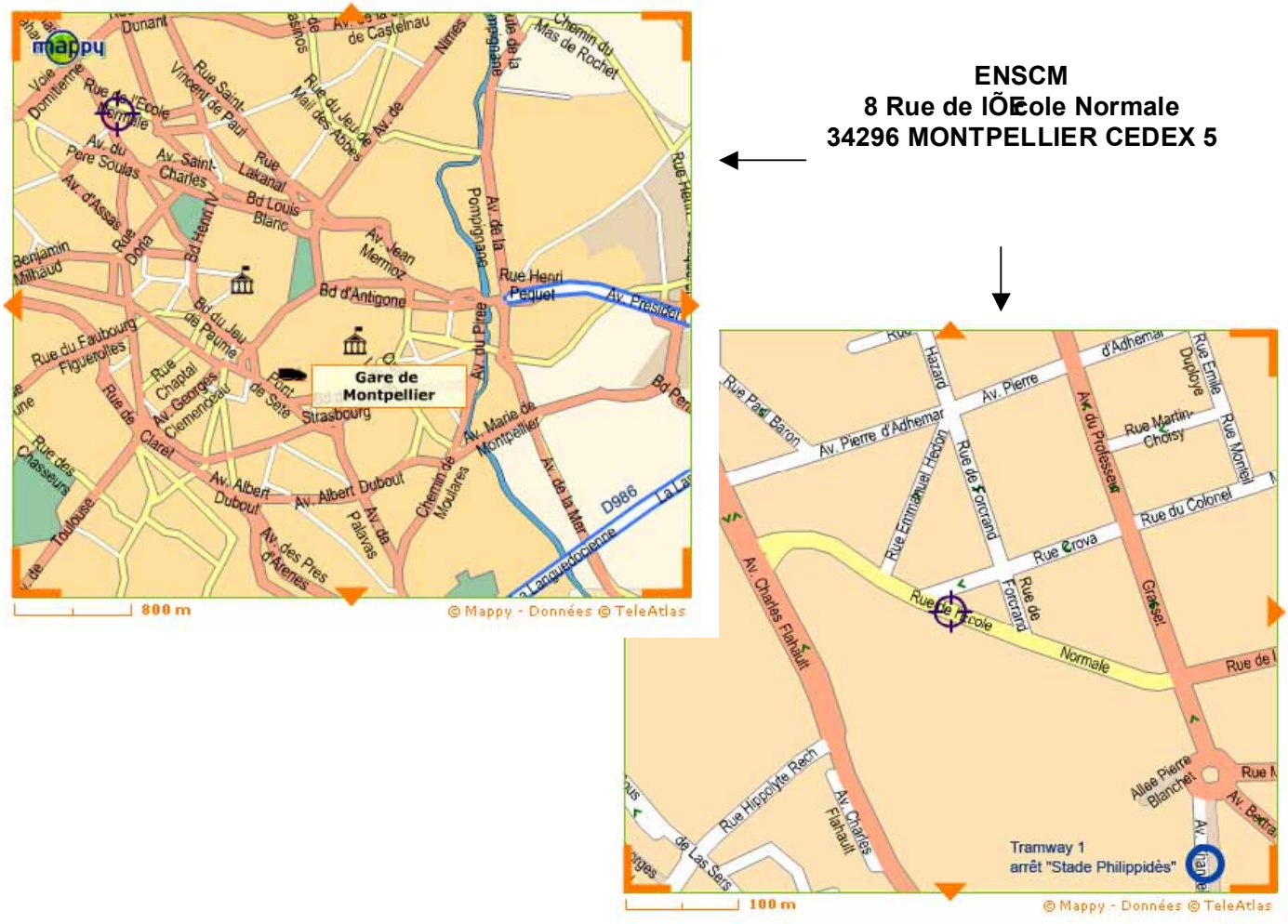
- Montpellier is accessible by air form Paris, London and other European airports. Montpellier is also well connected by rail to any major destination in Europe through the fast speed train (TGV). Paris-Montpellier journey typically takes 3 hours and 30 minutes and costs about 120 Euros for a 2nd class return ticket.

- The workshop will take place at the National School of Chemistry of Montpellier (Ecole Nationale Superieure de Chimie de Montpellier) located 8 Rue de l'ecole Normale (see Map Below). From the train station (Gare Saint-Roch) or the Corum take the Tram number 1 (Blue line) towards "Mosson" and stop at the tram stop "Stade Philipides". Follow the Avenue du professeur Grasset for 100 m and turn left into the ecole normale. Walk another 100 m and the Ecole de Chimie will be on your right. Once in the school of chemistry follow the ISGS signage to the auditorium.

Location

Ecole Nationale Supérieur de Chimie de Montpellier (ENSCM)
8 Rue de l'Ecole Normale
34296 Montpellier

Access Map for ENSCM (Ecole Nationale Supˇrie ure de Chimie de Montpellier)



How to register

Pre-registration form: http://www.isgs.org/fr2007.asp

Registration fees:

Full rate:	**€400 or $535 ***
ISGS member's:	**€300 or $400**
Student rate:	**€100 or $134**

***Average exchange rate 1€ = 1.338$**

Registration fees do include: Coffee Breaks, Lunch and Workshop Notes

Detailed program

Sol-gel alkoxide precursors and their solution transformations on the way to oxide materials

V G. Kessler, Swedish University of Agricultural Sciences

Silicon alkoxides and metal alkoxides are broadly recognized as promising precursors of oxide materials in the sol-gel technique. The alkoxides of silicon are moderately reactive inorganic esters, while the alkoxides of metals are highly reactive salts of common alcohols. The poor reactivity of silicon alkoxides in hydrolysis can be improved either by basic catalysis (S_N2 mechanism), acidic catalysis (proton-assisted S_N1 mechanism) or by modification with chelating ligands. Formation of silica-based materials can be directed towards polymer colloids with porosity and mechanic properties determined by kinetics of hydrolysis-polycondensation, or, at extreme pH or via colloid templation with surfactants, towards micellar self-organized systems. Metal alkoxides are ionic salts with kinetics unhindered in homogeneous solutions. They are strong Lewis and Brönsted bases. The structure and composition of metal alkoxide complexes can rather efficiently be predicted, applying the Molecular Structure Design Concept – an application for molecular species of the Goldschmidt's ionic self-assembly principles. High reactivity in both hydrolysis and ligand transfer processes excludes formation of polymer colloids starting from metal alkoxides. The solution transformations of these precursors proceed via thermodynamically driven self-assembly processes, leading to Micelles Templated by Self-Assembly of Ligands (MTSALs) and can be controlled by a proper choice of solvents and ligands . The exploitation of the properties of precursors, tailored via introduction of heteroligands, such as oxo-ligands (through controlled microhydrolysis or micropyrolysis), or chelating ligands, such as carboxylates, β-diketonates, β-ketoesters, or even chelating and bridging ligands, such as alkanolamines, permit to selectively influence porosity and mechanic properties of the final materials.

Structure Development in Sol-Gel Processing

C. J Brinker, the University of New Mexico

In sol-gel processing the structure of the inorganic gel reflects the sequence of reversible hydrolysis and condensation reactions of alkoxide precursors. For bulk silicate systems prepared under acidic conditions, where the rate of siloxane hydrolysis is minimized, the structure develops according to a reaction limited condensation pathway resulting in highly ramified networks paralleling those in organic polymer systems. Under basic conditions where, siloxane hydrolysis rates are maximized, ripening leads to more highly condensed particulate systems that reduce the solid-liquid interfacial area. Recently sol-gel chemistry has been combined with surfactant or block-copolymer self-assembly leading to so-called ordered mesoporous silica. In these systems uniform confinement of sol-gel chemistry within nanoscale domains results in novel framework architectures that alter the siloxane ring distribution. This lecture will discuss structure development during 'classical' sol-gel processing and contrast that with nano-confined sol-gel chemistry.

Organic-Inorganic Hybrid materials
G. Kickelbick, Vienna University of Technology

Inorganic-organic hybrid materials are one of the most expanding material classes based on the sol-gel process. The synergistic combination of the various components in one material leads to properties and processing possibilities that cannot be reached by other materials. Particular advantages of hybrid materials are that they can be produced under mild reaction conditions in usual organic solvents.

This introductory presentation will give an overview of basic principles that have to be followed in the synthesis of these materials with respect to the desired functionality of the final material. Examples of different combinations of building blocks from the molecular to the nanoscale are given. Furthermore methods for materials processing are presented and finally potential applications are summarized.

Films from sol-gel
M. Aegerter, Journal of Sol-Gel Science and Technology

Wet chemical technology processing, the so-called Sol-Gel process, paves the way to the versatility and ease of liquid film deposition on many types of substrates for a variety of inorganic and hybrid coatings materials. Many techniques have been developed and are in practical use for laboratory and industrial applications.
This lecture will describe the most important conventional techniques such as spin coating, dip coating, spraying, meniscus coating, all well known to coat glasses, but also some recent developments to coat thin glasses and plastic foils at low temperature using printing techniques and which allow to get flat and patterned coatings.
Processing-substrate pre treatment, coating procedure, curing, etc.-will be presented and the properties of the obtained coatings will be discussed showing the advantages and disadvantages of the sol-gel process in comparison to other deposition processes. To illustrate the versatility of the processes, several examples, mostly taken from industrial products in different fields, will be shown.

Colloids and Nanoparticle
T. Gacoin, Ecole Polytechnique

Nanoparticles as constituents of materials with novel properties are currently studied intensively in the frame of various approaches (physics, chemistry, biology) to materials science. In the last decade, tremendous improvements have been achieved in the elaboration of nanoparticles through colloid chemistry routes and many compounds are now accessible as colloidal dispersions. The main interest for nanoparticles arise from size effects that are observed when considering materials with dimensions that are close to the characteristic lengths of some physical properties. Beyond the optical transparency of well dispersed systems, this has been shown in many compounds to lead to drastic modifications of their physical properties as compared to the relative bulk materials, modulated by the enhanced importance of surface states.
This lecture will provide an overview of the latest development of nanoparticles research toward applications. The different strategies of elaboration and the main physical properties of nanoparticles will be first reviewed. Then, the different strategies of surface modification will be discussed, either for the optimization of the particles physical properties, or for the further chemical manipulation of the particles for the elaboration of nanostructured materials. Starting from applications that aim to exploit the properties of nanoparticles as individual objects, the last part of the talk

will concern the latest development on coupled systems such as self organized nanostructures exhibiting cooperative effects or multifunctional nanoparticles.

Porous sol-gel solids
K. Nakanishi, Kyoto University

Gels are crosslinked system often formed in the co-presence of diluting additives called "solvent". Upon removal of the solvents, spaces occupied them are left as "pores". The crosslinking backbone may deform during the solvent removal, and influences the volume, size and shape of the resultant pores. By controlling the structural and mechanical properties of crosslinking backbone, materials with desired final pore structures can be prepared. The lecture overviews basic principles of synthesis and characterization of porous materials. Examples of monolithic materials with hierarchical well-defined macropores and controlled mesopores in various crosslinking systems containing metal oxides, organo-siloxane polymers and pure hydrocarbons follow; Synthetic progress in alkoxy-derived macroporous silica lead to the preparation of long-range ordered mesoporous skeletons in well-defined macroporous framework. Alkylene-bridged silicon alkoxides can also be prepared into similarly hierarchical porous structures with broadened variations in framework morphology. Macro-mesoporous alkoxy-derived pure titania and zirconia have been prepared using hydrochloric acid – mediated processes. Titania monolith is a promising candidate as a separation medium to discriminate phosphorylated compounds in a liquid chromatography mode. Pure alumina macroporous monolith has been first synthesized from aluminum salt using propylene glycol as a proton scavenger to thrust the solution pH from acidic into neutral conditions. Alumina-based complex oxides such as garnets and spinels can also be prepared in pure phases. Polymerization and phase separation in organic crosslinker system was also controlled to obtain well-defined co-continuous macro-frameworks instead of those composed of aggregated particles. A special case of controlling mesopores in randomly crosslinked silsesquioxane system to fabricate low-density, aerogel-like monolithic materials will also be introduced.

Sol-gel and Bio-technology
T Coradin, Universite Pierre et Marie Curie

Biotechnological processes make use of the properties of biological systems to design functional devices such as sensors or reactors. The contribution of Material Science to this field mainly consists in providing suitable environments that stabilize biologicals against chemical and physical denaturation and favor their processing as films, particles or fibers.

These features remarkably resemble the key advantages of sol-gel approaches to inorganic materials synthesis when compared to traditional high temperature routes, that led to the development of hybrid organic-inorganic materials. However, biological systems present certain limitations in term of chemical and physical stability that had to be overcome before their encapsulation in sol-gel matrices could be successfully performed.

In this lecture, we will present the different strategies that have emerged along these last 25 years to immobilize biomolecules (proteins, enzymes, immune agents, nucleic acids and phospholipids) and whole cells within sol-gel hosts. The influence of encapsulation on biologicals will be discussed, emphasizing that the presence of a bio-organic additive is often needed to optimize the host properties on a long term.

Relevant analytical methods for “in silica” monitoring of biological activities will be examined. Selected examples will help to demonstrate the actual possibilities of sol-gel biotechnology.

As perspectives, most recent advances in this field will be presented, demonstrating that a still-expanding number of encapsulation strategies and encapsulated biologicals are currently under study. We will also suggest that the limitations in industrial developments of sol-gel materials in biotechnology may not differ significantly from other fields of applications.

The Speakers

Vadim Kessler

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Vadim G. Kessler graduated with honours from the Moscow State University (MSU) in 1987 and obtained there his Ph.D. degree in 1990 with Dr. N.Ya.Turova, being appointed directly after this an Assistant Professor at the Department of Inorganic Chemistry MSU. He has spent his first sabbatical year (1992/93) as an associate Researcher of CNRS at the University of Nice with Prof. L.G.Hubert-Pfalzgraf and became an Associate Professor at MSU in 1995. The second sabbatical year, 1995/96, he worked as visiting scientist at the University of Stockholm with Prof. M.Nygren and moved finally to Sweden, being appointed an Associate Professor at the Department of Chemistry, SLU in Uppsala in 1997. He received there his Habilitation Degree in 2000 and was promoted to Full Professor at the same University in 2004. The main research fields are the synthesis and characterization of structure and reactivity of metal alkoxides, β-diketonates and carboxyaltes and their application in the synthesis of inorganic and hybrid materials using Soft Chemistry techniques. His received in 2003 the International Donald Ulrich Award “For Excellence in Sol-Gel Science” and became a member of the Advisory Board of the Journal of Sol-Gel Science and Technology in 2006. Vadim is the author of 130 publications and 4 patents. He is a member of the American Chemical Society, the International Sol-gel Society, International Union of Crystallography and Struchkov Prize Society for promotion of crystallography.

C. Jeffrey BRINKER

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C. Jeffrey Brinker received his B.S., M.S., and Ph.D. degrees in ceramic science and engineering from Rutgers University. Jeff joined Sandia National Laboratories as a Member of the Technical Staff in 1979. He was promoted to Distinguished Member of the Technical Staff at SNL and appointed Distinguished National Laboratory Professor of Chemistry and Chemical Engineering at the University of New Mexico in 1991. Since 1999, he has been jointly employed at SNL where he is Sandia Fellow and at UNM where he is Regent’s Professor of Chemical and Nuclear Engineering with co-appointments in the Departments of Molecular Genetics and Microbiology and Chemistry. Brinker has been recognized nationally and internationally for his work in sol-gel processing and its extension to self-assembly of porous and composite nanostructures. His awards include an R&D100 Award, the American Chemical Society’s Ralph K. Iler Award in the Chemistry of Colloidal Materials (sponsored by DuPont), five Department of Energy Basic Energy Sciences Awards, the DOE Ernest O. Lawrence Memorial Award in Materials Science, and the Materials Research Society 2003 MRS Medal. In February 2002 he was elected into the National Academy of Engineering.

Guido KICKELBICK

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Guido Kickelbick received his PhD in 1993 in the field of Inorganic Chemistry from the University in Würzburg (Germany) where he worked under the supervision of Professor Ulrich Schubert on the synthesis and characterization of surface-functionalized metal oxo clusters. After receiving his PhD degree he was awarded a post-doctoral fellowship with Krzysztof Matyjaszewski at the Center for Macromolecular Engineering at the Carnegie Mellon University in Pittsburgh (USA) on the application of controlled radical polymerization in the formation of hybrid materials. In 1998 he returned back to the Vienna University of Technology where he worked since then in the field of hybrid materials and nanocomposites as well as surface-functionalized nanoparticles with a particular focus on the combination of organic polymers and inorganic components. Guido Kickelbick has published more than 150 scientific papers on different aspects of inorganic, polymer and materials chemistry. He is in the editorial board of several scientific journals and a member of the Austrian Chemical Society, American Chemical Society, German Chemical Society and Materials Research Society.

Michel AEGERTER

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SWITZERLAND

Prof. Dr. Michel A. Aegerter graduated as Engineer and Physicist in 1962 at the Swiss Institute of Technology- EPFL, Switzerland and received his Ph.D degree (Dr. ès Sciences) in 1966 at the University of Neuchâtel, Switzerland. After post-doctoral studies at the University of Utah, Salt Lake City, USA, he joined again the University of Neuchâtel as assistant professor and then the University of Sao Paulo, Sao Carlos, Brazil as a full Professor till 1995. There, starting in 1984, he built the first South-American group developing fundamental research in the sol-gel field. In 1995 he accepted an invitation to be the Director of the Department of Coating Technology at the Leibniz-Institute for New Materials gem. GmbH- INM in Saarbruecken, Germany, a position that he hold till his retirement in October 2006. During this period his fields of activity covered the research and industrial development of many functional coatings deposited on glasses and plastics, such as electrically conducting coatings, photoelectrochemical and electrochromic coatings, as well as the development of new sol-gel and nanocomposite coating technology. He was also Chairman of the Technical Committee TC-16 (Sol-Gel Glasses) of the International Commission on Glass (ICG) and member of many International Societies and Advisory Committees of Journals and Conferences., as well as referee for several international scientific journals. Presently Prof. M. Aegerter is a consultant and lives in Switzerland. He is Honorary Professor of the University of Saarland, Saarbruecken (Germany), editorial Chairman of the International Conferences on Coatings on Glass-ICCG and Editor-in-Chief of the J. Sol-Gel Science and Technology. Prof. Dr. Aegerter is the author and co-author of more than 460 scientific publications, 8 patents and co-editor of 12 books.

Thierry GACOIN

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Thierry Gacoin received his PhD in 1994 in the field of Materials Science from the Université Pierre et Marie Curie (Paris VI) where he worked under the supervision of Prof. Jean-Pierre Boilot on the elaboration of silica /nanoparticles composite materials for optical applications. Since 1995, he works as an associate scientist at the CNRS (French National Research Council), in the Solid State Chemistry Group of the Condensed Matter Physics Laboratory (Ecole Polytechnique, Palaiseau). His research is mostly devoted to soft chemistry routes to nanostructured materials, mainly for optical applications. His fields of competence concern the elaboration of hybrid sol-gel materials and coatings, and the synthesis and functionnalization of colloidal nanoparticles. His current systems of interest are luminescent nanoparticles (mostly rare earth doped oxides) for applications as biological probes or for the development of transparent and luminescent coatings, and nanostructured mesoporous films for optical applications such as low refractive index or photo-catalytic coatings.

Kazuki NAKANISHI

Dept. of Chemistry, Graduate School of Science, Kyoto University
Kitashirakawa, Sakyo-ku, Kyoto, 606-8502 JAPAN

Kazuki Nakanishi majored in physical chemistry of polymeric solids in Department of Industrial Chemistry, Graduate School of Engineering, Kyoto University. He joined the research group of Professor Naohiro Soga in 1986 as a research associate, where he started his first works on sol-gel synthesis of inorganic materials. He encountered the phase separation of gelling silica system in August 1987, and has been enchanted by her for twenty years. He received his Doctor of Engineering degree from Kyoto University in 1991. He started collaborating with Prof. Nobuo Tanaka, Kyoto Institute of Technology and Dr. Hiroyoshi Minakuchi (President of Kyoto Monotech Corp.) on the development of novel HPLC columns using his monolithic silica with controlled macro- and mesopores. The industrialization of the columns was achieved in collaboration with Merck KGaA, Germany. The first HPLC column was released worldwide in October 2000, and it received the Gold Award of Pittsburgh Conference in 2001. During the above period, he was promoted to associate professor in 1995, received D.R. Ulrich Prize in 1997 and ICG Prize in Memory of Prof. Vittorio Gottardi in 1999. He stayed in Johannes Gutenberg University, Mainz, Germany as a visiting professor invited by Prof. Klaus Unger in autumn 2000. He moved to Department of Chemistry, Graduate School of Science, Kyoto University in 2005 where he continues extending his activity on sol-gel derived porous materials. He was awarded Academic Prize of the Ceramic Society of Japan in 2006. Kazuki Nakanishi is the author of >150 publications and >40 patents, and the co-author of 15 books. He is the member of the American Ceramic Society, Materials Research Society, International and Japanese Sol-Gel Societies, and several other Japanese societies related to materials and analytical chemistry.

Thibaud CORADIN

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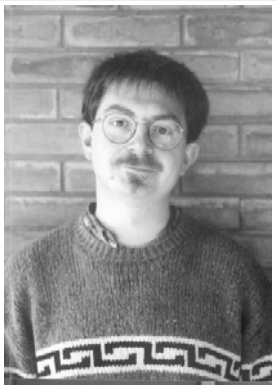

Thibaud Coradin received his PhD in 1997 in the field of Intercalation Chemistry from the Université Paris XI under the supervision of Pr R. Clément. He was then a post-doctoral fellow at the Royal Institution of Great Britain in London, in the group of Pr Peter Day, working on molecular magnets. He was then a temporary lecturer in the Ecole Nationale Supérieure de Chimie de Paris, studying organometallics and spin-trapping systems via EPR and ENDOR spectroscopy in the group of Pr D. Gourier. In 1999, he was appointed as a CNRS Research Fellow in the Laboratoire de Chimie de la Matière Condensée de Paris (LCMCP), working with Pr Jacques Livage on silica biomineralization and sol-gel bioencapsulation. Since 2005, he is leading the “Biogel” group of the LCMCP and has extended his research interests to the development of “green” sol-gel chemistry, based on the use of biological systems, including biopolymers and whole cells, to design bio-functional nanohybrids and nanocomposites. He co-authored over 60 publications and 8 book chapters.

For more information :

http://www.isgs.org/FR2007.asp

or contact the workshop coordinator

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