
Photo 1: P. Mondal

INTEGRATE Newsletter

November 2013
Issue 2

Year 2 in Review

This is the second newsletter from our ORF project INTEGRATE — Innovative Combinations of Groundwater Remediation Technologies. In this issue you will find an overview of the activities in Year 2. The main highlights of this second year are advances achieved in remediation technologies such as biodegradation, nanoscale zerovalent iron (nZVI), electrokinetics (EK), and self-sustaining treatment for active remediation (STAR). We have also accomplished important milestones in computer modeling of biodegradation combined with nZVI, nZVI transport, STAR, screening models, and DNAPL dissolution models. In addition, four new additional partnerships have been established this year and the project's progress has been demonstrated in 18 journal articles and 15 conference participations in Year 2. So far, in total, 24 papers have been published in refereed journals and 64 conference presentations have been delivered in the first two years of the project.

Our Students

From this issue on, we would like to recognize students and researchers that dedicate their time and efforts to this ORF. Thanks to them, our projects are successful. Our featured students on this issue are Laura Kinsman and Ahmed Chowdhury (pages 4 and 5).

Biodegradation

Biodegradation and Low Cost Substrates. In partnership with Pinchin Environmental, biodegradation of TCE was tested using low cost substrates (sawdust and peat) at a field application of a bio-barrier at a site in the Great Toronto Area. Prior to the field scale application, a series of microcosm studies and column tests (Photo 1) were performed by David Zhang, research associate, and Dr. Pulin Mondal, PDF, under Professor Brent Sleep's supervision. The objective of the laboratory experiments was to determine which

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low cost substrate, sawdust or peat, would be more conducive to reductive dechlorination at the trichloroethene (TCE) contaminated site. Molecular analyses targeting reductive dechlorination activity were performed both in microcosms and columns by Line Lomheim, Research Technician, working with Professor Elizabeth Edwards in BioZone and by Dr. Pulin Mondal. Preliminary analyses of the experimental results indicate that peat is more conducive to reductive dechlorination activity, with less methane production and lower hydraulic conductivity loss compared to sawdust.

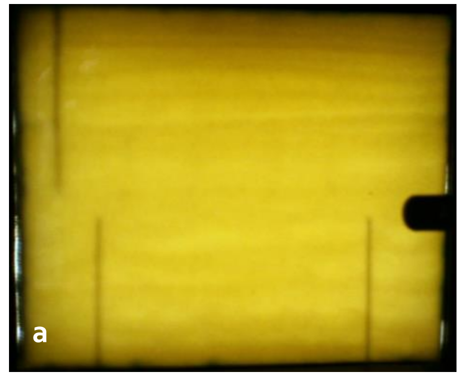
Advances in nZVI Applications

Bio-nZVI Microcosms. Microcosm studies were started last year to determine interactions of nZVI with biological activity associated with degradation of chlorinated ethenes. PDF Dr. Simone Larcher is responsible for this work and has found that excess sodium borohydride during nZVI synthesis generates a significant amount of hydrogen. This has important implications for the supply of hydrogen for biotic reductive dechlorination. Dr. Larcher was assisted in this work by Viviane Malveira Cavalcanti, an exchange student resident at the University Toronto through the Brazilian Science Without Borders Program.

nZVI Reactivity and Characterization. The objective of this research is to assess the efficiency of nZVI to remove heavy metals and cVOCs (chlorinated volatile organic compounds) when these contaminants are present, alone or as mixed waste, in synthetic or site groundwater samples. The remediation of mixed waste is challenging as some contaminants may significantly affect the removal of co-contaminants. For example, some heavy metals enhance the dechlorination of cVOCs while others do not have any significant effect. nZVI and Pd-nZVI are found to significantly degrade a wide range of cVOCs including chlorinated ethenes, ethanes and methanes when these are present in high concentrations in groundwater samples from actual contaminated sites. nZVI is successfully utilized to degrade cVOCs until Fe^0 gets completely oxidized to Fe^{3+}. Various studies have been conducted to better understand the reductive and adsorptive capacity differences between nano- and micro-scale zerovalent iron (mZVI). Detailed chemical and surface analyses (SEM/EDX, XPS, XRD) of nZVI and mZVI particles before and after contaminant removal have also been studied by PDF Dr. Hardiljeet Boparai.

Polymer Flooding. A two-dimensional bench scale carboxy methyl cellulose (CMC) polymer transport experiment is underway to investigate the importance of viscosity in nZVI transport, as indicated by a modeling paper by Dr. Magdalena Krol (ES&T). This work was initiated by a summer student Ivan Damnjanovic, and is being completed by Dr. Pulin Mondal with supervision from Professor Sleep. To date, dye tracer experiments and hydraulic tests have been completed while CMC transport tests are underway (Photos 2a and 2b). A student from the Brazil Science Without Borders, Mateus Lima, and a Master's student, Ertiana Rrokaj, are assisting with these experiments. The 2-D experiment will help determine interactions between nZVI and CMC, a topic previously investigated in the laboratory by former PhD student, Dr. Erica Pensini. Dr. Pensini used atomic force microscopy (AFM) and a quartz microbalance to investigate the interactions of nZVI, polymers, and various substrates (see publication list for Dr. Pensini's papers).

nZVI Stabilized by Microemulssions. Ziheng (Harry) Wang's PhD research topic, supervised by Professor Edgar Acosta in Chemical Engineering at UofT. After investigating a large number of surfactant combinations Harry identified promising surfactant formulations for nZVI stabilization. He also evaluated the impact of surfactants on nZVI reactivity with



Photos 2a – 2-D CMC transport experiment before injection of CMC solution, 2b – after injection started (Courtesy P. Mondal)

carbon tetrachloride (CT) and reactive black 5 (RB5) using batch experiments. Harry found that surfactants with strong binding to iron surfaces significantly inhibited the surface reaction with water-soluble contaminants (e.g. RB5), while promoting the reaction with oil-soluble contaminants (e.g. CT). Harry is currently working on the stabilization of nZVI using microemulsions. The emulsified-nZVI particles demonstrate not only long-term stability but also reactivity with different contaminants (Photo 3).

Other nZVI related studies. Optimization of nZVI particle size with respect to colloidal stability, mobility and reactivity has been investigated via bench scale experiments by Dr. Cjestmir de Boer (PDF). Dr. de Boer has completed construction of columns for this work and will be using techniques he developed for monitoring nZVI in the columns.

The secondary impacts of technologies

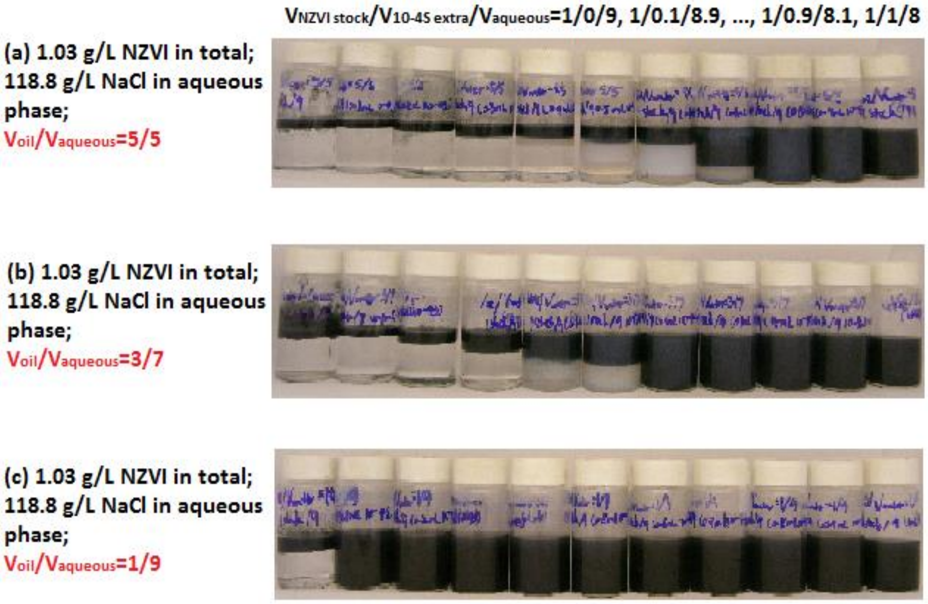


Photo 3. The effect of pH on nZVI suspension stabilized with surfactants after 24 hours of storage. The vials were prepared with nZVI (1.03g/L), surfactants and oil (ethyl caprate) in 118.8g/L of NaCl. Completely black vials indicate stable nZVI suspensions (Courtesy of Z. Wang).

associated with gas generation during nZVI applications is the thesis topic of Obai Mohammed, who is working with Professor Mumford. Obai started his PhD in September 2012 and is conducting experiments on the effect of entrapped gas bubbles on relative permeability. This will be followed by gas exsolution experiments examining gas production associated with nZVI reactions.

EK-ISCO in Fine Grained Media

Electrokinetic Enhanced Oxidant Delivery for In-Situ Remediation of Fine-grain Porous Media. In-Situ Chemical Oxidation (ISCO) has been used for contaminated site remediation at numerous large scale sites in different geologic settings. However, poor oxidant delivery through low permeability soils poses a limitation on the success of this technology. Limited oxidant delivery causes back diffusion of contaminants from the less permeable zones. It is proposed that Electrokinetics (EK) can be used to enhance oxidant delivery in soils with low hydraulic conductivity. Therefore, the main objective of this study is to investigate the use of EK to deliver oxidants through fine grained porous media and to quantify the remediation in a 2D laboratory experiment. Experiments were conducted by Ahmed Chowdhury under different conditions including a control (without oxidant and EK), ISCO only (with oxidant only, no EK) and EK-ISCO (with oxidant and EK) and samples were analyzed to quantify contaminant (TCE) degradation. Visual observations reveal that permanganate penetrated only approximately 1-2 cm into the fine soil regions in the ISCO only experiment, thus, leaving most of the fine soil inaccessible to ISCO treatment. EK enhanced transport experiments showed that permanganate migrated through the entire 8 cm wide fine soil zones. These experiments suggest that EK has the potential to effectively deliver permanganate into fine grained porous media where delivery would be otherwise impractical. Further investigation of contaminant degradation resulting from oxidant delivery in the fine grained porous matrix is currently in progress.

STAR (Self-sustained Treatment for Active Remediation)

STAR can also be defined as 'self-sustaining smoldering', a highly controlled burning reaction – similar to charcoal in a BBQ. Self-sustained smouldering destroys organic contaminants embedded in porous media while simultaneously generates enough energy to propagate itself through the subsurface. STAR has proven to be highly effective at remediating soil contaminated with heavy hydrocarbons and coal tar, for which few remedial options exist. With a rapid treatment time and little external energy required, STAR may provide a highly cost effective in situ or ex situ treatment approach.

Multi-Dimensional STAR. Influence of NAPL Remobilization Tarek Rashwan, who completed a final year undergraduate thesis on the mobility of heated liquids under STAR, started his Master's degree on the STAR project at Western in September 2013. Also starting a Master's degree at the same time is new student Cody Murray, whose research will focus on the development of a continuous STAR treatment system, in which a fixed bed reactor is fed a continuous input of NAPL which is destroyed as it percolates to the self-sustaining reaction maintained within the bed. This research, requiring a balance of reaction and NAPL mobilization rates, will improve our understanding of NAPL mobility in the heated zone above the STAR reaction front.

Process Sensitivity of STAR. Laura Kinsman (Master's student) is setting up tall column experiments to examine the scaling of STAR. Laura returned in June from a four month research visit to the University of Queensland supported by her award from the NSERC Michael Smith Foreign Study Supplement. At UQ, Laura learned about the fundamentals of smouldering combustion and examined scaling of existing data sets with Professor Jose Torero, widely recognized as a world leader in smouldering science and fire engineering. Laura has built 3 new STAR columns of increasing size and has planned a suite of experiments that will look at STAR propagation dynamics and timescales across several orders of magnitude, from the bench to the pilot reactor scale.

By-products in soil and groundwater, gaseous emissions. Experiments conducted at the bench scale during the last quarter by Dr. Rory Hadden (former PDF) are complete and results are being prepared for a journal manuscript. This work focused on the proportion of VOCs in the gaseous emissions and their sensitivity to key variables (air flow rate, NAPL content). Rory has been recruited to a faculty position at the University of Edinburgh, but he will stay affiliated with the project as a collaborator. In addition, Geosyntec is planning a detailed emissions study at the pilot scale STAR reactor to examine whether or not the high destructive efficiency observed in the lab will correspond to the ex situ pilot scale; this is critical for designing the off-gas treatment system for the full scale system, currently being considered for a large oil company. Also, in collaboration with our colleagues at the University of Strathclyde, they will examine the fate of metals as a result of the STAR process in their new STAR laboratory.

In Situ STAR. Shallow (8 ft) and deep (25 ft) ignition tests were conducted in collaboration with Geosyntec Consultants and Dupont, at the New Jersey DuPont site which is contaminated with coal tar. The shallow soil is a heterogeneous fill material, while the deep soil is a heterogeneous, alluvial deposit. The first test was reported in the previous issue of this newsletter; the second test was very successful with the reaction spreading across a 2 ft thick contaminated layer and remediating a cylindrical region with a radius of influence of approximately 12 ft. These were the final 2 field pilot tests. A manuscript is in preparation for detailing the field test results. The full source zone treatment is now being designed. This will be the first full scale application of STAR technology *in situ*.

Photo 4. STAR *Ex Situ* Reactor (Courtesy of RESTORE).

Grant Scholes has finished his master's degree which focused on a novel method for igniting STAR for in situ applications. This method was successful at the lab scale and also in the in situ pilot trials. This is a critical step for the commercialization of STAR, since lab-scale STAR ignition is not robust for rapid and repeated field deployment. This new approach is both a scientific and technological advance, and the work is currently being written up for journal submission.

Plans are under way to apply STAR across the entire DuPont New Jersey site (pending final approval by the site owner). This would involve multiple ignitions of subsurface smouldering and would be the first commercial scale application of STAR. Treatment would be for coal tar both at shallow (3 m) and deep (7m) locations across the site. A new master's student, Mehrnoosh Ebrahimzadeh, is investigating the potential application of geophysics for real-time tracking of the propagation of smouldering fronts associated with STAR in the field. If laboratory experiments demonstrate the technique to be promising, it is anticipated that field testing will occur at a future pilot test.

Modeling

Combined Remedy of Bioremediation

Laura Kinsman, MESc Student

Laura graduated from the Western University in 2012 with Bachelor's degree in Civil and Environmental Engineering and Environmental Science. Her Bachelor's fourth-year thesis was on "Characterizing groundwater Flows in the Alberta Oil Sands using Major Ions" with Dr. Jason Gerhard and Dr. Clare Robinson. Currently she is enrolled in a MESc program at Western, supervised by Prof Gerhard. Laura held the NSERC Alexander Graham Bell Canada Graduate Scholarship last year, and was awarded the Ontario Graduate Scholarship for this year. She was also awarded the NSERC Michael Smith Foreign Study Supplement, which allowed her to visit Professor Jose Torero at the University of Queensland in Brisbane, Australia from February to June 2013.

The objective of Laura's research is to conduct an experimental and analytical investigation into how liquid smouldering combustion phenomena scale in relation to STAR. She worked with Professor Torero at the University of Queensland to develop an understanding of the key processes involved in liquid smouldering combustion at small column and large ex-situ reactor scales. A suite of intermediate scale experiments is currently in progress to provide important information for fully understanding the scaling relationships of STAR.

and NZVI, model development. The paper on modelling the results from a field trial (Bennett et al., *Journal of Contaminant Hydrology*, 2010) has been published in *Environmental Science and Technology* (Krol at al. 2013). PhD student Ahmed Chowdhury and Dr. Magdalena Krol (PDF) are now modelling an nZVI field trial performed at the DND site in London, ON. At the site, a pilot scale nZVI field application was performed to understand nZVI transport and reactivity in the saturated and unsaturated zones. In this study, one of the injection wells was partially screened in the saturated layer leading to nZVI also being delivered to the vadose zone. This led to an investigation of NZVI fate and transport in both saturated and unsaturated zones using COMPSIM (numerical simulator developed by Professor Sleep). The simulations showed that significant amounts of nZVI were attached to the soil in the unsaturated zone, which affected the delivery of nZVI to the contaminated subsurface (Figure 1). However, TCE concentrations in the study area were still reduced by more than 99% of the historical TCE concentration. It is hypothesized that injection of nZVI resulted in short-term TCE degradation (~30 days) while long-term TCE degradation is due to enhanced biodegradation from the injected CMC which serves as an electron donor. This work is being written up as a journal article to be submitted later this year. Once microcosm experiments are complete the model will be further developed for simulating the combined effects of biological activity and nZVI reactions. Salman Sabahi (PhD student at UofT) will continue this modelling work.

STAR – Modelling. Master's student Tanzeer Hasan, who successfully defended his thesis, extended the heuristic STAR front propagation model to two-dimensions and validated it against laboratory experiments. That work has been submitted to a journal. PhD student, Marco Zanoni, is developing a more complete thermodynamic model of STAR using the COMSOL Multiphysics Software to better understand the interacting processes (heat transfer, mass transfer, relative permeability to air, etc) occurring during the propagation of a self-sustaining smouldering front.

Screening Models. PhD student Salman Sabahi submitted a manuscript on using the practical finite analysis method for simulating advective - dispersive - reactive transport to the Hydrological Sciences Journal. Dr. Amalia Kokkinaki has two papers on modelling DNAPL dissolution.

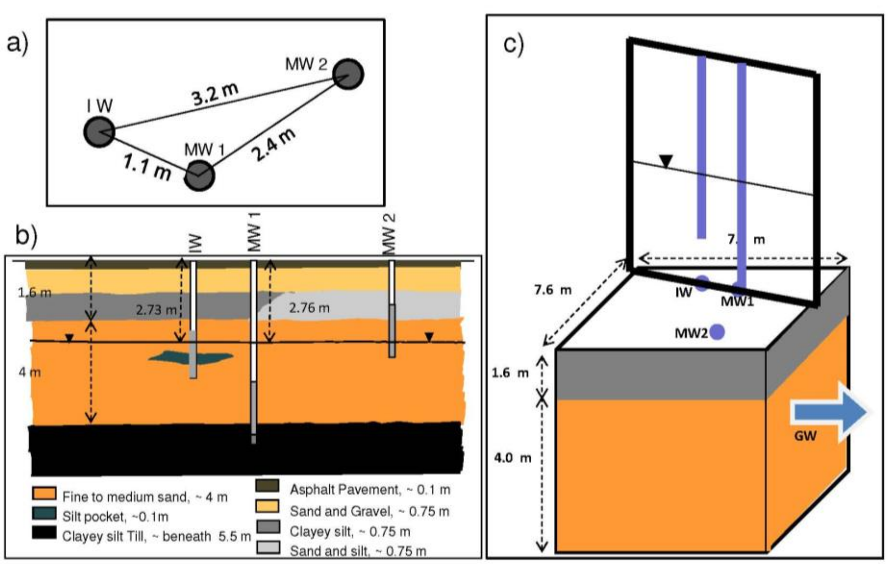


Figure 1. nZVI Transport - Model Development: (a) relative well locations at the site; (b) cross-section of study area; (c) model domain with volumetric section or plane passing through the IW-

Ahmed Chowdhury, PhD Candidate

Ahmed has a Bachelor's degree from Bangladesh University of Engineering and Technology (BUET). He worked as a lecturer at the Presidency University, Dhaka, and at the Military Institute of Science and Technology (MIST), both in Bangladesh. After graduating with a MESc in Environmental Engineering with RESTORE at the Western University, Ahmed joined the PhD program at Western, again with RESTORE, under the supervision of Prof. Denis O'Carroll.

For his PhD, Ahmed is investigating the mobility of nZVI in the subsurface under the Influence of electrokinetics. His research objective is to determine ways of delivering nZVI particles to source zone while they are reactive. He is focusing on soils with low hydraulic conductivity (i.e. clay) where mobility is a big challenge. Electrokinetics (EK), the application of direct electric current, is expected to enhance the transport of nZVI to the target zone, even through clayey soils. Ahmed has successfully built his own laboratory bench top prototype and has completed his experiments using fine sands. He is now focusing on an *in situ* application of this technology and expanding the application of EK to the delivery of oxidants for *in situ* chemical oxidation (ISCO) to fine soils.

List of Publications

Alexandra, R., J.I. Gerhard, and B.H. Kueper. 2012. Hydraulic displacement of dense non-aqueous phase liquids for source zone stabilization, *Journal of Ground Water*, 50(5), pp. 765-774.

Boparai, H.K., M. Joseph, D.M. O'Carroll. 2011. Kinetics and thermodynamics of cadmium ion removal by adsorption onto nano zerovalent iron particles. *Journal of Hazardous Materials*, 186 (1) 458-465.

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Brovelli, A., D.A. Barry, C. Robinson, and J.I. Gerhard. 2012. Analysis of acidity production during enhanced reductive dechlorination using a simplified reactive transport model. *Advances in Water Resources*, 43, 14 – 27.

Chowdhury, A. I. A., O'Carroll, D. M., Xua, Y., Sleep, B. E. 2012. Electrophoresis enhanced transport of nano-scale zero valent iron. *Advances in Water Resources*, 40: 71-82.

Drake, S. S., O'Carroll, D. M. and Gerhard, J. I. 2013. Wettability contrasts between fresh and weathered diesel fuels. *Journal of Contaminant Hydrology*, 144, pp. 46-57.

Kocur, C.M., O'Carroll, D.M., Sleep, B.E. 2013. Impact of nZVI stability on mobility in porous media. *Journal of Contaminant Hydrology*. 145, 17–25.

Kokkinaki, A., O'Carroll, D. M., Werth, C. J. and Sleep, B. E. 2013. An evaluation of Sherwood-Gilland models for NAPL dissolution and relation their relationship to soil properties, *Journal of Contaminant Hydrology*.

Kokkinaki, A., O'Carroll, D. M., Werth, C. J. and Sleep, B. E. 2013. Coupled simulation of DNAPL infiltration and dissolution in three dimensional heterogeneous domains: process model validation. *Water Resources Research*, DOI: 10.1002/wrcr.20503.

Krol, M.M., Oleniuk, A.J., Kocur, C.M., Sleep, B.E., Bennett, P., Xiong, Z., O'Carroll, D.M. 2013. A Field-Validated Model for *In Situ* Transport of Polymer-Stabilized nZVI and Implications for Subsurface Injection. *Environmental Science and Technology*, 47 (13) 7332–7340.

MacPhee, S.L., J.I. Gerhard, and G. Rein. 2012. A novel method for simulating smoldering propagation and its application to STAR (Self-sustaining treatment for active remediation), *Environmental Modelling and Software*, 31: 84-98.

O'Carroll, D.M., B. Sleep, M. Krol, H. Boparai, C. Kocur. 2013. Nanoscale zerovalent iron and bimetallic particles for contaminated site remediation. *Advances in Water Resources*, 51, 104-122.

Pensini, E., Yip, C.M., O'Carroll, D. M., and Sleep, B.E. 2012. Effect of Water Chemistry and Aging on Iron—Mica Interaction Forces: Implications for Iron Particle Transport. *Langmuir*, 28 (28) 10453–10463.

Pensini, E., Sleep, B.E., Yip, C.M., O'Carroll, D. 2012. Forces of interactions between bare and polymer coated iron and silica: Effect of pH, ionic strength and humic acids. *Environmental Science and Technology*. 46 (24) 13401–13408.

Pensini, E., Sleep, B.E., Yip, C.M., O'Carroll, D. 2013. CMC binding to mineral substrates: Characterization by atomic force microscopy based -force spectroscopy and quartz-crystal microbalance with dissipation monitoring. *Journal of Colloid and Interface Science*. , 402, 58-67.

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Pensini, E., Sleep, B. E., Yip, C. M. 2013 Forces of interactions between iron and aluminum silicates: effect of water chemistry and polymer coatings,. 411(1), 8 – 15, *Journal of Colloid and Interface Science*.

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Wang, Z., Lam, A, Acosta, E. 2013. Suspensions of Iron Oxide Nanoparticles Stabilized by Anionic Surfactants. *Journal of Surfactants and Detergents*. DOI: 10.1007/s11743-012-1425-1.

Wang, Z., Acosta, E. Formulation Design for Targeting Delivery of Iron Oxide Nanoparticles to TCE Zone. 2013. *Journal of Contaminant Hydrology*. 155: 9-19.

Zhi, D., Zhou, Z., and Sleep, B. E., Influence of wettability on interfacial area during immiscible liquid invasion into a 3D self-affine rough fracture: Lattice Boltzmann simulations. *Advances in Water Resources*, August 2013.

FAST FACTS

24

students involved in this ORF

22 and 56

papers and citations, respectively,

Conference Presentations in Year 2

- 1 modelling presentation at the Thermal Remediation Workshop, Austin TX, Jan 9, 2013
- 2 modelling presentations at the AGU Fall Meeting, CA, Dec 2012
- 1 nZVI presentation by Sleep and O'Carroll at the Groundwater Association Symposium in Brazil, Sao Paulo, Oct 4, 2012
- 2 nZVI presentations by Z. Wang at the 104th AOCS Ann Meeting & Expo. Montreal, QC. Apr28-May1 2013
- 5 STAR presentations at the AquaConSoil, Barcelona, Apr 2013
- 1 STAR presentation at the 2013 MGP Symposium, Georgia, Nov 2012

Important Milestones and Numbers

- Congratulations to Tanzeer Hasan and Grant Scholes for graduating
- Congratulations to Tanzeer Hasan for his new position as a lecturer at Dhaka University, Bangladesh
- Congratulations to Rory Hadden for his new position as Assistant Professor at the University of Edinburgh
- We have had 9 Post-Docs (5 at present)
- Current students: 9 PhD, 9 Master`s
- 22 papers have been published so far, totaling 56 citations on Scopus
- We had 61 presentations—platform, poster and guest lecture—over the first two years of the project

2nd Annual Meeting

A very successful second annual progress meeting, held on September 20, 2013, was attended by 81 people, including the members of the Science Advisory Board, the Technical Implementation Committee, private sector partners, representatives of the Ontario Ministry of Energy and Environment, and 63 students and post-doctoral fellows. An invited keynote address was delivered by Dr. Kent Novakowski from Queen's University.

The following presentations were made by several students and PDFs involved in the project.

- Numerical Modeling of Stabilized nZVI Field Application - Magdalena Krol (UofT)
- Enhanced Reductive Dechlorination of cVOCs by nZVI - Ariel Garcia and Jorge Dominguez (WU)
- Influence of Surfactant Coatings on nZVI Reactivity with Water-soluble and Oil-soluble Contaminants - Ziheng Wang (UofT)
- Low Cost Carbon Substrates to Stimulate In-Situ Bioremediation of Chlorinated Solvents - Pulin Mondal, UofT)
- Degradation of a Multi-component Chlorinated Solvent at a nZVI Field Injection Site - Chris Kocur (WU)
- Effects of nZVI Injection on Microbial Communities at a Field Site - Elizabeth Edwards (UofT)
- Upscaling STAR: Experimental Study of Scaling Relationships for Smouldering Combustion to Remediate Soil - Laura Kinsman (WU)
- Kinetic Parameter Estimation for Coal Tar Pyrolysis and Oxidation in Smouldering Combustion Applied to STAR - Marco Zanoni (WU)
- Novel Method for CSIA of Contaminated Groundwater Across the Sediment-Water Interface - Elodie Passeport (UofT)
- Evaluating Time-lapse Electrical Resistivity Tomography for Monitoring DNAPL Source Zone Remediation - Chris Power (WU)

Impact

Since the start of the project, interest in STAR and nZVI research results by the industry has grown. STAR has being considered a real world solution for subsurface contamination and its application has been translated to other fields. For example, the UofT/UWO team proposed STAR as a novel sanitation technology for the developing world in a competition promoted by the Bill and Melinda Gates Foundation. They were chosen among the three finalists. As for nZVI, trials performed in Australia under supervision of the UWO team have stimulated application of that technology by local consulting companies. The impact of this research project for Ontario, in terms of scientific advancement and economic development, is growing and has enormous potential.


Attendees at the 2013 Annual meeting at Western University

Acknowledgments

Private Sector Partners:

Partner Institutions:

This is project is funded by the **Ontario Research Fund.**

We would like to thank Hardiljeet Boparai, Ahmed Chowdhury, Laura Kinsman, Magdalena Krol, Simone Larcher, and Pulin Mondal for their contributions to this Newsletter.

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Issue 2 November 2013

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