UND NORTH DAKOTA

DIVISION OF RESEARCH & ECONOMIC DEVELOPMENT

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Grants & Contracts Administration Twamley Hall, Room 100 264 Centennial Dr Stop 7306 Grand Forks, ND 58202-7306 Phone: 701.777.4151 UND.grantcontracts@research.UND.edu Website: UND.edu/research November 1, 2016

Ms. Karlene Fine, Executive Director North Dakota Industrial Commission ATTN: Oil and Gas Research Program State Capitol 14th Floor 600 East Boulevard Avenue, Department 405 Bismarck, ND 58505-0840

Dear Ms. Fine:

Subject: UND Proposal Entitled "Functional Nanoparticle-Augmented Surfactant Fluid for Enhanced Oil Recovery in Williston Basin" for a North Dakota Industrial Commission Oil and Gas Research Program Funding Request.

A UND Research Team lead by Dr. Hui Pu and Dr. Julia Zhao is proposing a project to develop a novel nanoparticle enriched surfactant fluid with tailored properties for enhanced oil recovery (EOR). The proposed EOR process has a potential to significantly impact oil recovery in the Bakken Formation.

Enclosed please find an original and one copy of the subject proposal along with a check of the \$100 application fee.

If you have any questions, please contact me.

Sincerely,

Hui Pu, Ph.D. Assistant Professor Department of Petroleum Engineering University of North Dakota 701-777-6861 hui.pu@engr.und.edu.

Julia Xiaojun Zhao, Ph.D. Professor Department of Chemistry University of North Dakota 701-777-3610 Julia.zhao@und.edu

Burt

Dr. Barry Milavetz, Associate Vice President of Research Economic Development University of North Dakota Barry.milavetz@und.edu Oil and Gas Research Program

North Dakota

Industrial Commission

Application

Project Title: Functional Nanoparticle-Augmented Surfactant Fluid for Enhanced Oil Recovery in Williston Basin

Applicant: Department of Petroleum Engineering, Department of Chemistry, Institute of Energy Studies, University of North Dakota

Principal Investigator: Hui Pu, Julia Zhao

Date of Application: November 1, 2016

Amount of Request: \$769,134

Total Amount of Proposed Project:

\$1,724,006

Duration of Project: 36 months

Point of Contact (POC): Hui Pu

POC Telephone: 701.777.6861

POC E-Mail Address:

hui.pu@engr.und.edu

POC Address: Department of Petroleum Engineering 243 Centennial Drive Stop 8154 Grand Forks ND 58202-8154

TABLE OF CONTENTS

Please use this table to fill in the correct corresponding page number.

	1
Abstract	4
Project Description	5
Standards of Success	13
Background/Qualifications	14
Management	14
Timetable	15
Budget	15
Confidential Information	15
Patents/Rights to Technical Data	15
References	Appendix A
List of Preliminarily Selected Surfactants	Appendix B
Resumes of Key Personnel	Appendix C
Relevant UND Laboratory Facilities and	Appendix D
Capabilities	
Letter of Support	Appendix E
Budget Justification	Appendix F

Transmittal and Commitment Letter

Affidavit of Tax Liability

Statement of status on Other Project Funding

ABSTRACT

Objective:

The objective of this project is to develop a novel nanoparticle enriched surfactant fluid for enhanced oil recovery (EOR) in Williston basin. In this fluid, the nanoparticles will carry surfactant to deeply penetrate rock matrix, then effectively displace oil locked in micro- and nano-pores of tight rocks, and finally carry the oil out of the rocks. The features of the designed nanoparticles will be: 1) controllable delivery of surfactant and alter the wettability of interfaces of oil with the fluid; 2) high mobility, water solubility, stability, and uniform dispersion in the reservoir fluids; 3) tunable chemical composition, shape, size, porosity and functionality; 4) environmentally friendly; and 5) low cost. The commercialization of this technology will lead to higher oil recovery, prolonged reservoir life, reduced operation cost and further minimizing the environmental complications.

Expected Results:

The results will provide conceptual validation of nanoparticle loaded surfactant fluid for EOR in Bakken tight formation. Vital data will be collected based on the fluid performance regarding their wettability, mobility and the effects of EOR in the Bakken play from macro- to nanoscale levels. Positive recovery rates are expected. Accumulated data, mechanisms and optimization outcomes will lay a solid foundation to the technology's commercialization in the near future.

Duration:

The duration of the proposed project will be 36 months (05/01/2017 to 04/30/2020).

Total Project Cost:

The total cost of the project is \$1,724,006. The amount requested from the Oil and Gas Research Council (OGRC) is \$769,134 and combination of cash and in-kind co-funding totaling at least \$954,872 from InPetro Technologies Inc. and the University of North Dakota (UND). EOG Resources Inc. is providing Bakken crude oil samples from its North Dakota wells as in-kind cost share support.

Participants:

A UND research team of three faculty members, one postdoc, and three graduate students from three departments, Petroleum Engineering, Chemistry, and Institute of Energy Studies, will participate in the

project. Two industrial partners, InPetro Technologies Inc. and EOG Resources Inc. will support the project by providing samples and software.

PROJECT DESCRIPTION

Objectives:

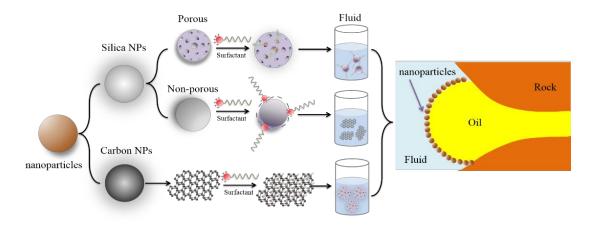
The Bakken Formation in Williston basin is one of the largest contiguous deposits of oil and natural gas in the United States. While the horizontal drilling and multi-stage hydraulic fracturing technologies contribute to the current high oil production rate, primary recovery is low, ranging from 5 to 10% of OOIP.¹ Our project addresses this critical need in the context of: *How can we develop an ideal EOR fluid to successfully penetrate rocks, then effectively displace oil locked in micro- and nano-pores of tight rocks, and finally carry the oil out of the rocks?* To meet this need, at extreme temperature, pressure and salinity of the harsh reservoir environment, the challenges are (1) limited effective fluid for displacing oil from tight formations, (2) limited mobility of surfactant-based fluid for leaving tight formations. In order to address these challenges, we propose to develop a novel nanoparticle-surfactant based fluid. The combination of nanoparticles and surfactant will increase the oil recovery in Bakken play.² Additionally, the silica and carbon materials used for making nanoparticles will be environmentally friendly and economically advantageous. It is expected the proposed EOR will continue to transform the Bakken Formation into a prolific oil producer and promote energy independence in the US.

Methodology: To achieve the proposed objectives, four major tasks will be conducted using different well-designed methodologies. First, functional silica and carbon based nanoparticles will be synthesized and integrated with surfactant. In parallel, a number of surfactants will be screened to select the most effective one for oil displacing. Before applying the nanoparticle-surfactant fluid into the oil field, the Bakken samples from North Dakota will be thoroughly characterized. Finally, the nanoparticle-surfactant fluid will interact with the Bakken to evaluate the EOR rate.

Task 1. Development of Nanoparticle Enriched Surfactant for EOR (*Lead: Dr. Zhao, Team members: Dr. Zhang, and Ms. Karen Liu***).** Among several EOR options, surfactant imbibition and gas injection (CO₂ flooding and huff-n-puff) receives most of attentions.³ However, the low mobility of surfactant results in low EOR response and notable amount of surfactants remained inside rocks.

5

Additionally, the waste of surfactants increases the cost of oil production in addtion to the low oil recovery rate and potential enviormental impact. Recent applications of nanomaterials in EOR have demonstrated promising results. Mondini *et al* proposed oleic acid coated nanoparticles and PEG-based surfactant.⁴ Haroun *et. al* tested several nanoparticles included Fe(III)O, CuO and NiO in carbonate reservoirs.⁵ However, these research are still in the initial stage and silica and carbon nanoparticles are not applied. The features of silica and carbon, 1) low cost, 2) environmentally friendly, 3) well studied surface chemistry for easy linking surfactants, 4) good water solubility and stability, would make them effective surfactant carriers to compose fluid for EOR. Our design is shown in Figure 1.





Porous and non-porous silica nanoparticles and carbon nanoparticles will be developed. Surfactant can be doped into the porous nanoparticles or adhered to the nonporous nanoparticle surface. It is expected that the porous nanoparticles would carry more surfactants. By tuning nanoparticle size and porosity, efficiency of carrying surfactant will be optimized. The nanoparticle-surfactant hybrid will be a key element in the fluid. Their small size and specially designed surface chemistry alter the water and oil interfacial wettability, reduce interfacial energy barrier, and increase movability, which would facilitate easy penetration of rocks as well as departure of rocks after displacing oil. Furthermore, the nature of the silica and carbon materials will remain stable at high temperature, pressure and salinity (194-248°F, TDS 150,000-300,000 ppm) at a relatively low cost.

The synthesis and optimization of nanoparticle-surfactant hybrid is a key step in this project. The following experimental design will be performed to achieve the proposed goals.

<u>1.1 Synthesis of Porous Silica Nanoparticles.</u> The porous silica nanoparticle will be synthesized using reverse microemulsion method based on our previous work.^{6,7,8} The synthesis will focus on optimization of three factors, a) porosity; b) surface chemical groups; and c) sizes, through changing microemulsion composition, reaction time, and post-coating chemicals.

<u>1.2 Synthesis of Non-porous Silica Nanoparticles.</u> The Sőber method followed heating treatment will be utilized to make non-porous silica nanoparticles.⁹ Varying heating temperature will be used. The tensile strength of nanoparticles will be tested for selection of the hardest nanoparticles.

<u>1.3 Synthesis of carbon nanoparticles.</u> Generally, synthesis of carbon nanoparticles can be divided into two categories, namely top down and bottom up. In this project, we will use bottom-up chemical method to synthesize carbon nanoparticles based on our previous experience.^{10,11} This experiments will focus on the following aspects: a) Carbon containing molecules and oxidative agent stoichiometry; b) Different fabrication approaches including hydrothermal reaction, microwave reaction, and combustion; c) Reaction temperature; and d) Reaction time.

<u>1.4 Integration of Surfactants to Nanoparticles.</u> Though selection of surfactants will be part of Task 2, the integration work will occur in parallel, surfactants will be integrated onto nanoparticles through two approaches: doping surfactants inside porous silica nanoparticles and adhering surfactant onto the

particle surface. Negatively charged porous silica nanoparticles can dope positively charged surfactants. Surface chemical groups on carbon and non-porous silica nanoparticles will be varied based on the selected surfactant chemical structures. The hydrophilic groups of surfactant will form chemical bonds with nanoparticle surface groups, reserving hydrophobic groups for displacing oils.

<u>Preliminary results.</u> We have developed more than 10 silica and carbon based porous and non-porous

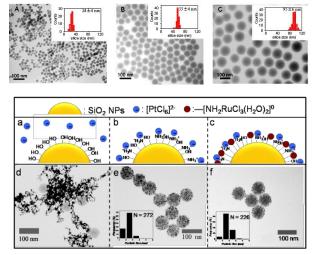


Figure 2. Top: TEM images of different sized silica nanoparticles. Bottom: Surface functionalization of nanoparticles with different chemical groups

nanoparticles during the past 12 years.¹²⁻²⁴ An example of silica nanoparticles with different sizes and surface modification is shown in Figure 2.

Task 2 – Surfactant Screening and Nanoparticle-Surfactant Interactions (*Lead: Dr. Zhang, Team member: Dr. Pu, Dr. Zhao, and Ms. Karen Liu*). Twenty-five developmental and commercial available surfactants (anionic, cationic, nonionic, and amphoteric, a list of the preliminarily selected surfactants is in Appendix B) will be evaluated in terms of their individual oil displacing property, integration of nanoparticles with surfactant, and the effectiveness of fluids containing ultra-low (0.02%-0.2%) and low concentration of nanoparticle-surfactant hybrid. A series of laboratory screening methods will be developed for this evaluation. Specifically, the following tests will be conducted.

<u>2.1 Pre-screening Compatibility Test.</u> Synthetic Bakken formation water of 300,000 ppm TDS and lower salinity (e.g. 150,000 ppm) will be used. Brine compatibility test is a fast and effective method to identify favorable surfactant formulations. Ultra-low and low concentration surfactant solutions will be observed for whether or not precipitation will occur in brine solution at 190°F after three weeks. High-temperature and high-salinity stability test will also be conducted for surfactant solutions. Surfactant solutions with no precipitation and suspension will be prepared for further study.

<u>2.2 Phase Behavior, Optimum Solubilization Ratio and Salinity</u>. The effect of surfactant concentration on equilibrium phase behavior will be investigated by preparing a set of samples with an oil/water ratio of one to one. Winsor phase behavior method will be performed using different surfactant solutions, Bakken crude oil and pure hydrocarbon (i.e., decane). Clarity of interfaces is just one advantage of using pure hydrocarbon for the initial screening, and at the same time, interface will be compared with samples using Bakken crude oil. The salinity of the brine phase is an important parameter influencing type of Winsor phase behavior. Effect of salinity on phase behavior will be evaluated. The salinity at which equal volumes of oil and water are solubilized is defined as the optimal salinity. The ratios of Vo/Vs and Vw/Vs will be varied at different salinity (V represents volume; o, s and w stands for oil, surfactant and water, respectively). When these ratios are plotted, the intersection point within the Type III salinity range is the optimum solubilization ratio at the optimum salinity.

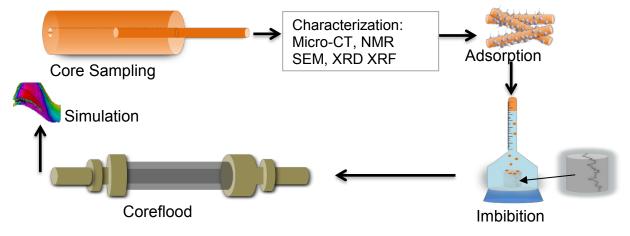
8

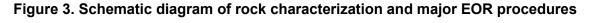
<u>2.3 Interfacial Tension (IFT) Measurement.</u> IFT between Bakken crude oil and different surfactant solutions will be performed at reservoir conditions using a spinning drop tensiometer as a supplement of screening surfactants. Following IFT measurement, nanoparticle-augmented surfactant solutions will be examined as nanoparticle inclusion into surfactant may result in unique interfacial behaviors. Such behaviors, if occurs, will be systematically analyzed and their mechanism will be studied.

<u>2.4 Critical Micelle Concentration (CMC) Measurement.</u> The CMC concentration corresponds to the point where the surfactant first shows the lowest surface tension. The surface tension remains relatively constant beyond this point. Normally, the CMC values is obtained through a conventional plot of the surface tension versus the surfactant concentration. Alternatively, Ultraviolet–visible (UV-Vis) spectroscopy together with pendant drop tests can be employed to measure the CMC of the surfactant in reservoir brine at ambient conditions.

Task 3 – Characterization of Bakken Core Samples (Lead: Dr. Pu, Team member: Dr. Mann, Mr. Hao Fu).

A thorough understanding and characteristics of unconventional reservoirs rock is crucial for studying the mechanisms of the proposed EOR. As shown in Figure 3, cylindrical core plug samples (1 or 1.5 inch in diameter, 3 inch in length) will be cut from whole core samples. Core plug samples will be cleaned, dried, and measured by petrophysical properties such as permeability, porosity, and dimensions.





<u>3.1 Petrographic characteristics.</u> The integrated analysis of minero-petrographical, physical parameters of the rocks can substantially reduce the uncertainty and difficulty in the data interpretation of EOR

experimental study. This integrated analysis will start with the petrographic and physical characteristics of rock samples from Middle Member of Bakken Formation. The physical characteristics of the rocks will be analyzed in the light of their petrographic characteristics. The petrographic data will be collected through optical microscope (OM) analysis of thin sections and a scanning electron microscope (SEM). Besides the regular observation from thin sections and SEM images, special attention will be directed to microfractures, openings and fractures. Further, X-ray Diffraction (XRD) and X-ray Fluorescence (XRF) analysis will also be performed at the UND's Materials Characterizations Laboratory.

<u>3.2 Rock Characterization Using NMR and Micro-CT Techniques</u>. The SEM images will be analyzed in conjunction with results from an advanced Oxford Geospec2 NMR core analyzer (with Green Imaging Technologies Imaging GIT Imaging System) at the UND lab. An advanced high-resolution x-ray micro-CT system at the University of Minnesota will be utilized together with NMR to thoroughly analyze images. Vital data, such as pore size distributions, capillary pressure, porosity, total pore volume, diffusion, 2D imaging, and 3D imaging, bulk and skeletal densities of the study rocks, will be obtained. Other important parameters (permeability and tortuosity) will be measured before and after NMR diffusometry experiment. The methodology also assesses and quantifies pore network features such as grains, pores and pore throats. The results of this image assisted petrophysical analysis will be compared to measurements by conventional core analysis methods, as well as relevant literature.

Task 4. Evaluation and Optimization of the Nanoparticle-surfactant Hybrid for EOR (*Lead: Dr. Pu, Team member: Dr. Zhang, Dr. Zhao, Mr. Hao Fu, and Ms. Karen Liu*)

<u>4.1 Adsorption Measurements.</u> Adsorption of surfactants from aqueous solutions to porous media is very important because surfactant loss attributed to adsorption on the reservoir rocks impairs the effectiveness and renders the process economically unfeasible.²⁵ In this project, adsorption of different concentrations of surfactant and nanoparticle-enriched surfactant solutions onto crushes Bakken sand grains will be measured using batch equilibrium adsorption procedure. The solutions and sand particles will be equilibrated in experimental containers and shaken for 24 hours, which is more than the time needed to reach equilibrium.²⁶ The surfactant solution, nanoparticle-surfactant solution and sand sediments will then be separated by centrifugation. Finally, the surfactant concentration will be

determined using the calibration curves generated from conductivities of surfactant solution and nanoparticle-surfactant solution.

<u>4.2 Oil recovery Experiments.</u> Two parallel experiments of imbibition and coreflood will be conducted to evaluate EOR efficiency. In this study, effect of artificial fractures on oil recovery will be investigated by creating fractures in some of core plugs. Four of the core plugs will be fractured with different orientations using a saw, and assembled with polyoxymethylene (POM) spacers to maintain a constant fracture aperture of 1 mm.

<u>4.2.1.Imbibition Tests.</u> Low concentration surfactant solutions, and nanoparticle-surfactant solutions will be used in imbibition tests in their "original" state and cores with artificially created fractures to represent the hydraulic fractured Bakken Formation. In such fractured reservoirs, spontaneous imbibition of water due to strong capillary forces is important and necessary to expel the oil from the matrix into the fracture system for high displacement efficiency. To enhance the spontaneous imbibition process in fractured reservoirs, surfactants are used as wettability alteration agents to modify the reservoir rock. To ensure effectively delivery of surfactants, a few factors will be investigated such as IFT reduction between oil and water, spontaneous emulsion formation, wettability alteration of silica and the modification of flow character. Meanwhile, NMR measurements will be performed during water imbibition in order to investigate changes in T_2 distributions as the water saturation increased. The core plugs will be protected by removing surface droplets with a moistened paper towel and wrapping the core plugs using plastic wrap to prevent evaporation. The core plugs will then be placed in a glass tube in the NMR instrument. The imbibition will be disrupted to obtain T_2 distributions. The weight of core plugs will be measured after imbibition for calculation of the fluid saturations.

<u>4.2.2 Coreflood experiment.</u> To further study the production and adsorption mechanisms we will conduct an injection of low concentration surfactant fluids and nanoparticle surfactant solutions separately into core plugs. This will enable the collection of oil recovery vs. time data to establish a time frame for the process that will help when setting up an economic analysis.

<u>4.3 Reservoir Simulation of Imbibition and Oil Displacement Processes</u>. A reservoir simulator developed by InPetro Technologies Inc. will be used in this study. InPetro simulator includes a novel formulation

11

of consistently evaluating capillary force and adsorption using pore size distribution (PSD) directly from core measurements and adsorption models using a local density optimization algorithm, those key features are not available in commercial reservoir simulation software. The spontaneous imbibition tests and coreflood test will be simulated using this software. The new findings could better address differences in flow mechanisms in unconventional reservoirs, thus lead to an optimized EOR practice.

Anticipated Results:

The results generated by this research will focus on conceptual validation and thorough understanding of the application of nanoparticle loaded surfactant effects on Bakken tight formation. Those new insights will provide the North Dakota oil industry as a whole with in-state developed nanotechnology that not only fits the pore-throat features of Bakken formation, but also forms a synergy with carefully selected surfactant(s) for enhanced oil recovery in tight formations. Important data and evidence about the effects of nanoparticles surfactant EOR in the Bakken play from macro- to nanoscale levels on mobilizing oil will be generated. Positive results are expected, the application of novel EOR can have a significant economic effect on the ultimate recovery of oil from tight formations.

Facilities and Resources:

The research team has access to the state-of-art facilities both internal and external to perform the proposed research. Details of laboratory facilities are shown in Appendix D.

Environmental and Economic Impacts while Project is Underway:

No significant environmental impacts while the project is underway. The economic impacts are below.

Ultimate Technological and Economic Impacts:

Technological impact: Application of nanotechnology to EOR is in the initial stage. The utilization of nanoparticles for EOR in tight oil recovers is a relatively new concept. If the proposed EOR is successful, it will bring a revolutionary technology for oil industry. The nanoparticle-surfactant based fluid will be a novel approach of making effective fluid for oil recovery. *Economic impacts:* The North Dakota Department of Mineral Resources estimates that original oil in place for the North Dakota-only portion of the Bakken/Three Forks Petroleum System is approximately 170 Bbbl.²⁷ Improvement of 1% of the

recovery rate would lead to an additional 1.7 Bbbl of oil recovered, which equate to over \$85 billion revenue alone in additional to the significant savings on operation cost.

Why the Project is Needed:

Given the very low primary oil recovery in tight formations including the Bakken system, it is imperative to develop new EOR technologies that will increase the oil recovery from the vast oil resources left behind by primary oil recovery. The functional nanoparticles can potentially act as "smart carriers" of surfactants and be delivered to the targeted sites for a more precise and controlled delivery, which can significantly improve the surfactants' oil-displacing efficiency, cut cost, and further minimize potential environmental concerns. Overall, the results of the project will provide petroleum industry and the state of North Dakota with a potential avenue to significantly improve Bakken oil recovery.

STANDARDS OF SUCCESS

Success will be measured based on the timely completion of proposed tasks, achievement of project milestones, quarterly reports as well as peer reviewed publications and patents produced in this research. The long-term value to North Dakota oil industry is ultimately realized by the full commercialization and widely deployment of the technologies invented. The short-term value can be generated by the core study and related experimental results that can be referenced in field oil production from the Bakken. Results in this research highly likely impact industry practices directly and lead to oil recovery improvements, with a potential of over 1 billion barrels of incremental oil and extend well production life for several decades. Other than oil generated and associated revenue, the value is also realized by creating additional jobs and enterprises. It has been estimated that a 10-to-20-year life span for the Bakken play in North Dakota will generate and maintain 3000 to 3500 long-term jobs.²⁷ On the other hand, intellectual properties of nanoparticles technologies are also locally developed and owned by the state university. The commercialization of this technology is likely to incubate more local high tech companies, further creating high-tech job opportunities and revenues at North Dakota.

BACKGROUND/QUALIFICIATIONS

The PI, Dr. Pu, has been working on EOR (chemical, CO₂, low salinity water flooding), unconventional reservoir, reservoir engineering, simulation and laboratory studies since 2003. Recently

13

he is working on organic-rich shale formations to investigate EOR in tight formations, and adsorption, nanopore characterization, pore size distribution and capillarity. Dr. Pu has been a key member for Bell Creek Integrated EOR and CO₂ Storage Project in Plains CO₂ Reduction (PCOR) Partnership at the Energy & Environmental Research Center, and he authored/co-authored seven project reports for U.S. DOE on EOR subject. His research on EOR was featured in SPE JPT (Appendix C). The Co-PI, Dr. Zhao, has been working in the field of nanotechnology for 15 years. Her group has developed a number of novel methodologies for making new silica and carbon based nanomaterials, resulting in more than 50 peer-reviewed papers, 11 book chapters, 3 issued patents along with 10 pending patent applications. Her CV is attached in the Appendix C. The Key Personnel, Dr. Zhang, was key personnel in a project for evaluating surfactant on EOR sponsored by ConocoPhilips. His current work focuses on development and characterization of carbon and silica-based nanomaterials. The Key personnel, Dr. Mann, is the Executive Director of the Institute of Energy Studies. He has been involved in multiple research projects for new technology development including energy production from combustion, gasification, wind, and geothermal resources, resulting in 215 publications.

MANAGEMENT

Dr. Pu and Dr. Zhao will be responsible for the overall project management. They will have day-to-day responsibility for ensuring that the activities, both in terms of time effort and fund expenditures, of the scientific team are commensurate with the goals of the proposal. Dr. Zhao will be lead of Task 1, Dr. Zhang will be lead of Task 2, and Dr. Pu will be lead of Task 3 and 4 though they will have overlapping roles in multiple tasks. Effective communications between team members will be established. The team will have biweekly meetings to discuss the progress and problems encountered in project to ensure the project is progressing according to schedule and the goals are being met. Regular and final project reports will be used to communicate project status, achievement, and budget to NDIC and other sponsors. **The evaluation points to be used** *include* scientific merit, the societal and economic impact of project results, student and postdoc performance, and the project effect on institutional practice. Biweekly team meeting, reports, publication, patents will also be points to be evaluated.

14

TIMETABLE

imetable in	six mo	onths ir	ncreme	ents. Tas	k 1 (ye	ellow),	Task 2	(red), Ia	ask 3 (purple), Task	4 (blue
Tasks in proposal	Y1 1-3m	Y1 4-6m	Y1 7-9m	Y1 9-12m	Y2 1-3m	Y2 4-6m	Y2 7-9m	Y2 9-12m	Y3 1-3m	Y3 4-6m	Y3 7-9m	Y3 9-12m
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Timetable in six months increments Task 1 (vellow) Task 2 (red) Task 3 (nurnle) Task 4 (blue)

BUDGET

As shown in the table below, the total cost of the project is \$1,724,006. The amount requested from NDIC is \$769,134, which accounts for 44% of total cost. A private company-InPetro Technologies Inc. provides a total of \$236,250 (\$78,750 per year) as matching fund in the form of in-kind contribution (Letter of support is Appendix E). UND provides a cost share of \$718,622 in the form of contributed equipment, salary, benefits and student tuition for UND participants. Detailed budget and budget justification is listed in Appendix F.

Year	Project Associated Expense	NDIC's Share	Applicant's Share (Cash)	Applicant's Share (In- Kind)	Other Project Sponsor's Share
Year 1	\$565,312	\$251,735	\$0	\$234,827	\$78,750
Year 2	\$569,325	\$253,898	\$0	\$236,676	\$78,750
Year 3	\$589,369	\$263,501	\$0	\$247,118	\$78,750
Total	\$1,724,006	\$769,134	\$0	\$718,622	\$236,250

CONFIDENTIAL INFORMATION

There is no confidential information.

PATENTS/RIGHTS TO TECHNICAL DATA

The concept of nanoparticle-surfactant fluid for EOR would be patentable. The applicants wish to reserve the right of application of an US patent in the future.

STATUS OF ONGOING PROJECTS (IF ANY) N/A

Appendix A: References

- 1. Hoffman, B Todd, Comparison of Various Gases for Enhanced Recovery from Shale Oil Reservoirs. Paper SPE 154329 presented at the SPE Improved Oil Recovery Symposium, Tulsa, Oklahoma, USA, April14-18, **2012**.
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- Li, A.; Jin, Y.; Muggli, D.; Pierce, D. T.; Aranwela, H.; Marasinghe, G. K.; Knutson, T.; Brockman, G.; Zhao, J. X., Nanoscale effects of silica particle supports on the formation and properties of TiO2 nanocatalysts. *Nanoscale* **2013**, 5, 5854-5862.
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- 18. Liang, S.; Shephard, K.; Pierce, D. T.; Zhao, J. X., Effects of a nanoscale silica matrix on the fluorescence quantum yield of encapsulated dye molecules. *Nanoscale* **2013**, 5, 9365-9373.
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- 23. Li, A.; Zhao, J. X.; Pierce, D. T., Silica nanoparticles for template synthesis of supported Pt and Pt-Ru electrocatalysts. *J. Colloid Interface Sci.* **2010**, 351, 365-373.
- 24. Li, A.; Zhao, J. X.; Pierce, D. T., Silica nanoparticles for template synthesis of supported Pt and Pt-Ru electrocatalysts. *J. Colloid Interface Sci.* **2010**, 351, 365-373.
- 25. Curbelo, Fabíola DS, et al. "Adsorption of nonionic surfactants in sandstones." *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 293.1 (**2007**): 1-4.
- Hollander, Agnes F., P. Somasundaran, and Carl C. Gryte. "Adsorption characteristics of polyacrylamide and sulfonate-containing polyacrylamide copolymers on sodium kaolinite." *Journal of Applied Polymer Science* 26.7 (1981): 2123-2138.
- 27. Nordeng, Stephan H., and Lynn D. Helms. "Bakken Source System–Three Forks Formation Assessment." *North Dakota Dept. of Mineral Resources* (**2010**).

Appendix B: List of Preliminarily Selected Surfactants

Tentative List of Surfactants to be Used in Phase Behavior Screening Study

Surfactant	Туре
dodecyl trimethyl ammonium chloride (DTAC)	anionic
octadecyl trimethyl ammonium chloride (OTAC)	anionic
Sodium dodecylbenzene sulfonate	anionic
Sodium laureth sulfate, SLS	anionic
Sodium dodecyl sulfate,SDS	anionic
Alkylaryl Sulfonates XOF-25S	anionic
Alkylaryl Sulfonates XOF-25S XOF-30S	anionic
Isotridecanol (Exxal 13 TDA)	anionic
Isotridecanol (Marlipal™)	anionic
Guerbet (Isofol® 12)	anionic
Guerbet (Isofol® 14T)	anionic
(Isalchem™ 145)	anionic
(Isalchem™ 123)	anionic
Safol® 23	anionic
Neodol®	anionic
Internal olefin sulfonate	anionic
Sulfonic acid of Alkylate A268	anionic
alkyl alcohol amine polyoxyethylene ether	nonionic
alkyl ethoxylate (AEO-7)	nonionic
SURFONIC® L-series surfactants	nonionic
Ethoxylated Alcohols	nonionic
dodecyltrimethylammonium bromide	cationic
Ethoxylated tallow amines	cationic
coco amido propyl betaine	amphoteric
Dimethyl amine oxide	amphoteric

Appendix C: Resumes of Key Personnel

Julia Xiaojun Zhao, Ph.D.

Professor Department of Chemistry University of North Dakota Grand Forks, ND 58203 701-777-3610; Julia.zhao@und.edu

PERSONAL STATEMENT

I have a broad background in the field of nanoscience and nanotechnology, with specific training and expertise in the development of silica and carbon based nanomaterials. As the PI/CoPI I have led a total of 6 federal grants including NSF, DoD and EPA, grants and 17 University- and State- funded grants. I have developed a number of novel silica nanomaterials. These works have resulted in 13 invention disclosures, three of them are issued patents now. I have co-authored 76 peer-reviewed publications, 11 book chapters and one book. The current application builds on my prior work. In addition, I have successfully administered the projects (e.g. staffing, research protection, budget), and collaborated with other researchers including industrial partners.

EDUCATION/TRAINING

Postdoctoral, Nanotechnology, University of Florida, 2004 Ph.D. Analytical Chemistry, Jilin University, China, 1994 B.S. Analytical Chemistry, Jilin University, China, 1984

Issued Patents

- 1. Chen, J.; <u>Zhao, J. X</u>., Hollow Silica Nanomaterials and Method of Making, US Patent, Issued September 22, **2015**, Patent No. 9,139,443.
- 2. <u>Zhao, J. X.</u>; Jin, Y., Manipulation of the Size of Silica Nanoparticles on a Continuous Spectrum. US patent, Issued, **2011**.Patent No.8,183,300
- 3. <u>Zhao, J. X., Pierce, D.</u>, Surfaces Coated with Target-Induced Fluorescent Compounds for Detection of Target Elements., US patent, US 7,629, 179 B2, Issured Dec. **2009**
- 4. <u>Tan, W.,</u> Jin, S., Zhao, X., Tapec, R., Drake, T., Hilliard, L., "Functionalized Nanoparticles and Methods of Use," US Patent, Pub. No. US 2004/0067503A1, **2004,** issued
- 5. <u>Jin Q</u>., Zhao, J. X., Zhang, H., Wang, Z., Xu, H., Liang F., "A Novel Surface Plasmon Resonance Device Based on Measuring Resonance Wavelength," China Patent ZL 98 1 15735.1, **2002**, issued

Pending Patent Applications / Invention Disclosures

- 6. Wu,X.; <u>Zhao, J. X</u>., Graphene Quantum Dots Derived from Glutamic Acid. US Patent, **2013**, pending.
- 7. Wu,X.; <u>Zhao, J. X</u>., *In situ* Green Reduced Graphene Oxide/Metal Nanocomposites, US provisional Patent, pending, **2013**
- 8. Tian, F.; <u>Zhao, J.X.</u>, Vertical Silicon nanowire-Silver Tetracyanoquinodimethane nanocomposites for Enhanced Field Emission Property, US provisional Patent, **2015**,
- 9. Z<u>hao, J.X.; Combs, C</u>., Nanoparticle Based Method for Microglial and Astrocyte Transfection, Invensiton disclosure, **2012**.
- 10. <u>Zhao, J.; Combs, C.;</u> Nanoparticle Based Method for Neuron Transfection. Invension disclosure, **2012**,
- 11. <u>Zhao, J. X.</u>; Nagamoto-Combs, K., A method to control the amount of fluorescence signals in detection of biological target molecules. US patent, **2011**, pending
- 12. <u>Zhao, J. X.</u>, Xu, S., "Engineering of SiO₂-Au-SiO₂ Sandwich Nanoaggregates Using a Building Block," US Patent, **2008**, pending
- 13. <u>Zhao, J. X.</u>, Xu, S., Engineering Rough Surface of Nanoparticles, US Patent, **2008**, pending

A. Positions and Honors Positions and Employment

2015— present Professor, Department of Chemistry, University of North Dakota, Grand Forks, ND
2009—2015 Associate Professor, Department of Chemistry, Univ. of North Dakota, Grand Forks, ND
2004—2009 Assistant Professor, Department of Chemistry, Univ. of North Dakota, Grand Forks, ND
2001—2004 Postdoctoral Research Associate, University of Florida, FL

1999—2001 Visiting Scholar, Department of Chemistry, Hong Kong Baptist University, Hong Kong 1984—1994 Editor and Senior Editor, Chemical Journal of Chinese Universities, Jilin, China

Other Experience and Professional Memberships

2016 President, North Dakota Academy of Science

- 2012- Member, American Nano Society
- 2002- Member, American Chemical Society

<u>Honors</u>

2013 UND Faculty Research Collaboration Award, University of North Dakota, Grand Forks, ND

2012 North Dakota Spirit Faculty Achievement Award, University of North Dakota, Grand Forks, ND

2010 UND Faculty Stars, University of North Dakota, Grand Forks, ND

2008-2014 A total of 14 Certificates of Acknowledgement for the Advancement of Intellectual Property Commercialization and Economic Development, Division of Research, University of North Dakota, Grand Forks, ND

2007 The Annual Phenomenal Woman Award, University of North Dakota and the Office of Multicultural Student Services, Grand Forks, ND

B. Contribution to Science & Technology

1. Development of Various Fluorescent Nanoparticles. We have developed eight fluorescente nanomaterials that could be categorized to three types: 1) target-induced fluorescence nanoparticles. These nanoparticles can serve as reaction centers for identification, collection and *in situ* determination of trace amounts of selenium, zinc and mercury.2) metal-enhanced nanocomposites, including a gold nanoparticle-enhanced fluorescent nanocomposite, an Au nanoparticles-silicon nanowire nanohybrid that exhibited strong capacity for surface enhanced fluorescence. In addition, we have developed serval nanocomposites, such as silica-TiO₂ nanocatalysts for studying nanoscale effect on catalytic activities, Au-silica nanowire nanohybrid and silica-Au-silica sandwich nanocomposite for phototherapy studies in the NIR region. We have also developed a new method for the synthesis of highly fluorescent graphene quantum dots (GQDs) using a pyrolysis of a natural amino acid. The GQDs emitted NIR fluorescence with the excitation-dependent manner. This work opens a new door for the development of more sensitive NIRF nanomaterials.

- Wu, X.; Tian, F.; Wang, W.; Chen, J.; Wu, M.; **Zhao, J. X.**, Fabrication of highly fluorescent graphene quantum dots using L-glutamic acid for/imaging and sensing. *J. Mater. Chem. C* **2013**, 1, 4676-4684.
- Liang, S.; Chen, J.; Pierce, D. T.; **Zhao, J. X.**, A turn-on fluorescent nanoprobe for selective determination of selenium(IV). *ACS Appl Mater Interfaces* **2013**, 5, 5165-5173.
- Chen, J.; Jin, Y.; Fahruddin, N.; **Zhao, J. X.**, Development of gold nanoparticle-enhanced fluorescent nanocomposites. *Langmuir* **2013**, 29, 1584-1591.
- Xu, S., Hartvickson, S., and **Zhao, J.X.**, "Engineering of SiO₂-Au-SiO₂ Sandwich Nanoaggregates Using a Building Block: Single, Double and Triple Cores for Enhancement of Near Infrared Fluorescence," *Langmuir*, **2008**, 24(14), 7492-7499

- John, C. L.; Strating, S. L.; Shephard, K. A.; **Zhao, J. X.**, Reproducibly synthesize gold nanorods and maintain their stability. *RSC Advances* **2013**, 3, 10909-10918.
- Xu, S.; Hartvickson, S.; **Zhao, J. X.**, Increasing surface area of silica nanoparticles with a rough surface. *ACS Appl. Mater. Interfaces* **2011**, 3, 1865-1872.
- Li, A.; **Zhao, J. X.**; Pierce, D. T., Silica nanoparticles for template synthesis of supported Pt and Pt-Ru electrocatalysts. *J. Colloid Interface Sci.* **2010**, 351, 365-373.
- **Zhao, J. X.**, Bawge, R., and Tan, W., "Synthesis of Organic Dye Doped Silica Nanoparticles in Reverse Microemulsion," *Adv. Mater.*, 2004, 16(2):173-176.
- Tan, W., Wang, K., He, X. Zhao, J. X., Drake, T., Wang, L., and Bagwe, R., "Bionano- technology Based on Silica Nanoparticles," *Med. Res. Rev.*, 2004, 24(5): 621-638.
- **Zhao, J. X.**, Dytocio, R.T., and Tan, W., "Ultrasensitive DNA Detection Using Highly Fluorescent Bioconjugated Nanoparticles," *J. Am. Chem. Soc.*, 2003, 125 (38): 11474-11475.
- **Zhao, J. X.**, Dytocio, R.T., and Tan, W., "Collection of Trace Amounts of DNA/mRNA Molecules Using Genomagnetic Nanocapturers," *Anal. Chem.*, 2003, 75(14): 3476-3483.
- Zhao, J. X., Pierce, D., Surfaces Coated with Target-Induced Fluorescent Compounds for Detection of Target Elements., US patent, US 7,629, 179 B2, Issured Dec. 2009
- Tan, W., Jin, S., **Zhao, J. X.**, Tapec, R., Drake, T., Hilliard, L., "Functionalized Nanoparticles and Methods of Use," US Patent, Pub. No. US 2004/0067503A1, **2004**, issued
- **Zhao, J. X.**; Jin, Y., Manipulation of the Size of Silica Nanoparticles on a Continuous Spectrum. US patent, Issued, **2011**.
- 2. Fundamental Study of Molecular Properties at the Nanoscale. To understand whether/how molecular properties change after they are bound on a small domain of nanoparticles, a series of comparison between the free molecules and the functionalized nanoparticles were conducted. These studies included metal binding kinetics, fluorescence quantum yields, and catalytic properties. The effects of nanoparticle size on their phototoxicity were investigated as well.
 - Li, A.; Jin, Y.; Muggli, D.; Pierce, D. T.; Aranwela, H.; Marasinghe, G. K.; Knutson, T.; Brockman, G.; **Zhao, J. X.**, Nanoscale effects of silica particle supports on the formation and properties of TiO2 nanocatalysts. *Nanoscale* **2013**, *5*, 5854-5862
 - Liang, S.; Shephard, K.; Pierce, D. T.; **Zhao, J. X.**, Effects of a nanoscale silica matrix on the fluorescence quantum yield of encapsulated dye molecules. *Nanoscale* **2013**, *5*, 9365-9373.
 - Liang, S.; Hartvickson, S.; Kozliak, E.; Zhao, J. X., Effect of Amorphous Silica Nanomatrix on Kinetics of Metalation of Encapsulated Porphyrin Molecules. J. Phys. Chem. C 2009, 113, 19046-19054.
 - Zhao, Y.; Ye, Y.; Zhou, X.; Chen, J.; Jin, Y.; Hanson, A.; Wu, M.; **Zhao, J. X.**, Photosensitive Fluorescent Dye Contributes to Photoxicity of Dye-Doped Silica Nanoparticles in Cells and Mice, *Theranostics* **2014**, *4*, 445-459.

3. Application of the Fluorescent Nanomaterials for Bioimaging. The applications included the *in vitro* luminescence imaging of cellular structures, monitoring of the presence of metal ions inside cells, *in vivo* imaging of mice using the fluorescent nanoparticles as the biomarker, and the detection of trace elements.

 Gao, Y.; Zou, X.; Zhao, J. X.; Li, Y.; Su, X., Graphene oxide-based magnetic fluorescent hybrids for drug delivery and cellular imaging. *Colloids Surf. B Biointerfaces* 2013, 112, 128-133.

- Liu,S.; Shi, F.; **Zhao, J. X.**; Chen,L.; Su, X., 3-Aminophenyl boronic acid-functionalized CuInS₂ quantum dots as the near-infrared fluorescent probe for the determination of dopamine, *Biosens. Bioelectron* **2013**, 47, 379-384
- Jin, Y., Kannan, S., Wu, M., **Zhao, J. X.**, "Toxicity of Luminescent Silica Nanoparticles to Living Cells," *Chem. Res. Toxicol.*, **2007**, 20(8), 1126-1133. [Article featured in *ACS News Service Weekly PressPac* on Aug. 8, 2007 and *Medical News Today, First Science, Bio-Medicine, Science Daily, Quick News etc*].
- Jin, Y., Lohstreter, S., Pierce, D.T., Parisien, J., Wu, M., Hall, C. III, **Zhao, J. X.**, "Silica Nanoparticles with Continuously Tunable Sizes: Synthesis and Size Effects on Cellular Imaging," *Chem. Mater.*, **2008**, 20 (13), 4411-4419.
- **Zhao, J. X.**, Hilliard, L.R., Mechery, J. M., Wang, Y., Jin, S., and Tan, W., "A Rapid Bioassay for Single Bacterial Cell Quantization Using Bioconjugated Nanoparticles," *Proc. Natl. Acad. Sci. USA*, **2004**, 101(42):15027-15032 [Featured in *Nature*, **2004**, 43, 923].
- John, C. L.; Huan, Y.; Wu, X.; Jin, Y.; Pierce, D. T.; **Zhao, J. X.**, A target-induced fluorescent nanoparticle for in situ monitoring of Zn(II). *Analyst* **2013**, 138, 4950-4957.

D. Research Support Ongoing Research Support

- NSF Naima Kaabouch (PI), 09/15/2014-09/14/2017 NUE: "Exposing Engineering students to Nanoscience and Nanotechnology at the University of North Dakota" Role: Co-PI
- Society for Analytical Chemists of Pittsburgh, Julia Zhao (PI), 05/01/05-12//30/17 "Development of Novel Fluorescent Nanosensors for Rapid, Sensitive and Selective Determination of Selenium in Organic Matter" Role: PI
- North Dakota Department of Commerce ND Venture, 03/01/2016-02/28/2017 "Graphene-based Infrared Fluorescent Quantum Dots for Bioimaging and Biodetection" Role: PI
- University of North Dakota UND VPR Office, 07/01/2016-06/30/2018 "Near Infrared fluorescent Graphene Quantum Dots for Bioimaging" Role: PI
- North Dakota Department of Commerce ND Ventrue, 01/01/2017-01/12/2018
 "Development of Novel Carbon Adsorbent for Removal of Pharmaceutical Herbicides and other Emerging Containment form in Drinking Water" Role: PI

Completed Major Research Support

- DoD, Zhao (PI) 09/12/2011-10/18/2013 "Absorbable Antimicrobial Battlefield Hemostat" Role: PI
- NSF, Zhao (PI) 08/01/09-09/30/13 "Near-Infrared Fluorescent Nanoparticles for Detection of Trace Analytes" Role: PI
- EPA, Zhao (PI) 05/15/2011-08/14/2012
 "Development of a Biosensor for Monitoring of Mercury Pollution in Natural Water" Role: PI
- NSF Hoffmann (PI) 01/01/10-12/31/12 CRIF, "Acquisition of a Cyber-enabled Scanning Electron Microscope for Research and Education in Nanochemistry, Role: Co-PI
- NSF, Zhao (PI) 08/01/06-07/30/09

"Surface Functionalized Nanoparticles for Enrichment and Detection of Trace Analytes" Role: PI

Hui Pu, Ph.D.

Assistant Professor Department of Petroleum Engineering University of North Dakota Grand Forks, ND 58202 701-777-6861; hui.pu@engr.und.edu

EDUCATION

2010 Ph.D. Petroleum Engineering, University of Wyoming 2006 M.S. Petroleum Engineering, Daqing Petroleum Institute, Daqing, China 2003 B.S. Petroleum Engineering, Daqing Petroleum Institute, Daqing, China

PRINCIPAL AREAS OF EXPERTISE

> Enhanced/Improved Oil Recovery, CO₂ Sequestration

- Studied CO₂ EOR and storage, and evaluated CO₂ EOR potential in some of fields in Wyoming;
- Worked on chemical flood (polymer, surfactant, surfactant-polymer flood) mechanism study, field evaluation, and pilot test;
- Conducted study on low salinity waterflood;
- Conducted study on waterflooding evaluated performance, well pattern optimization, designed surveillance, and studied feasibility in for non-fractured and naturally fractured low permeability reservoirs;
- Worked on thermal recovery: B.S thesis on cyclic steam stimulation.
- > Reservoir Engineering, Modeling & Simulation
- Strong experience and solid knowledge of petroleum engineering, especially reservoir engineering, modeling & simulation, and unconventional reservoirs;
- Simulated waterflood, CO₂ flood, polymer flood, and cyclic steam stimulation;
- Worked on enhanced recovery in unconventional reservoirs;
- Strong experience in pressure/rate transient analysis, decline curve analysis, material balance, and volumetric analysis;
- Set up pattern models, sector models and large reservoir models from scratch;
- Wrote programs to process data and generate the required formats of data for different reservoir simulators.

PVT Analysis, and SCAL

- Was responsible for the PVT tests simulation, equation of state (EOS) tuning, and minimum miscibility pressure (MMP) study on oil samples from Bell Creek oilfield;
- Worked on the core analysis in lab;
- Planned special core analysis testing programs, analyzed lab data, and utilized SCAL data in the simulation models.
- Laboratory Experimental Studies
- Conducted experimental study on surfactant flood, surfactant-polymer flood, polymer flood, and low salinity waterflood;
- Applied low-field Nuclear Magnetic Resonance (NMR) and Magnetic Resonance Imaging (MRI) to recovery mechanisms study of low salinity waterflood;
- Evaluated, screened and optimized the properties of surfactant, surfactant-polymer, polymer solutions, low salinity brine, and optimum formulations for field applications;
- Investigated wettability, spontaneous imbibition, chemical-based EOR processes, crude oil/brine/rock interactions, and interfacial tension;
- Conducted coreflood, core analysis, measurement and evaluation of different types of chemicals for EOR experiments.

WORK EXPERIENCE

July 2016 – Assistant Professor, Department of Petroleum Engineering, Univ. of North Dakota

- Teach petroleum engineering courses
- Conduct research in the areas of EOR, tight formations, reservoir engineering, modeling and simulation.

2015 Reservoir Engineer, InPetro Technologies Inc, Houston, TX

- Enhanced recovery, reservoir engineering; CO₂ EOR and storage mechanisms;
- Reservoir modeling and simulation, and field development plan.
- 2010 2014 Reservoir Engineer, Energy & Environmental Research Center, Univ. of North Dakota
 - Responsible for reservoir engineering studies for large-scale injection of carbon dioxide into the Bell Creek oil field for CO₂ EOR and long-term CO₂ storage, sponsored by DOE NETL and Denbury Resources;
 - Planned PVT and special core analysis testing programs and analyzed lab data;
 - Conducted MMP, PVT simulation, EOS (equation of state) tuning, and slimtube;
 - Co-Authored 8 Bell Creek project reports as a principal contributor for DOE NETL and Denbury Resources;
 - Worked on "Denbury Sequestration Project Phase 2b Evaluation of Selected CO₂ EOR and Sequestration Sites, funded by Denbury Resources" project, the results were utilized by Denbury's decision making for Bakken Sale and Asset Exchange with Exxon;
 - Worked on "Optimizing and Quantifying CO2 Storage Resource in Saline Formations and Hydrocarbon Reservoirs", funded by U.S. DOE NETL.

2006–2010 Graduate Assistant, Department of Chemical & Petroleum Engineering, Univ. of Wyoming

- Worked in Petrophysics & Surface Chemistry Lab: coreflood, evaluation of fluids, petrophysics, mineralogy, lithology, capillary pressure, IFT, and imbibition test;
- Investigated the mechanisms of low salinity waterflood, and evaluated its potential application in Tensleep, Minnelusa and Cottonwood reservoirs in Wyoming;
- Taught and graded students of *Rock and Fluids* course.

PUBLICATIONS

Selected U.S. DOE Project Reports

- Liu, G., Braunberger, J., Pu, H., et al.: "Bell Creek Test Site Simulation Report", Report for Denbury & U.S. DOE, Contract DE-FC26-05NT42592, Plain CO₂ Reduction (PCOR) Partnership, Energy & Environmental Research Center, Grand Forks, North Dakota, 2014,
- Braunberger, J., Pu, H., Gorecki, C., Hamling, J., Bailey, T., Ge, J., Gao, P., and Steadman, E.: "Bell Creek Test Site – Simulation Report", Report for U.S. DOE and Denbury, Contract DE-FC26-05NT42592, Plain CO₂ Reduction (PCOR) Partnership, Energy & Environmental Research Center, Grand Forks, North Dakota, 2013
- Ge, J., Klenner, R., Braunberger, J., Ayash, S., Pu, H., Gao, T., Bailey, T., Saini, D., Hamling, J., Sorensen, J., Gorecki, C., Steadman, E., and Harju, J.: "Bell Creek Test Site Geomechanical Modeling Report", Report for U.S. DOE and Denbury, Contract DE-FC26-05NT42592, Plain CO₂ Reduction (PCOR) Partnership, Energy & Environmental Research Center, Grand Forks, North Dakota, 2013
- Kurtz, B.A., Heebink, L.V., Eylands, K.E., Smith, S.A., Hamling, J.A., Klapperich, R.J., Thompson, J.S., Stepan, D.J., Botnen, B.W., **Pu, H.**, Gorecki, C.D., Steadman, E. N., Harju, J. A.: "Bell Creek Test Site – Preinjection Geochemical Report", Report for U.S. DOE and Denbury, Contract DE-FC26-05NT42592, Plain CO₂ Reduction (PCOR) Partnership, Energy & Environmental Research Center, Grand Forks, North Dakota, 2013
- Saini, D., Braunberger, J., **Pu, H.**, Hamling, J., Bailey, T., Ge, J., Gorecki, C., and Steadman, E.: "Bell Creek Test Site Simulation Report", Report for U.S. DOE and Denbury, Contract DE-

FC26-05NT42592, Plain CO₂ Reduction (PCOR) Partnership, Energy & Environmental Research Center, Grand Forks, North Dakota, 2012

- **Pu, H.**, Hamling, J., Bailey, T., Braunberger, J., Ge, J., Gorecki, C., and Steadman, E.: "Bell Creek Test Site Simulation Report", Report for U.S. DOE and Denbury, Contract DE-FC26-05NT42592, Plain CO₂ Reduction (PCOR) Partnership, Energy & Environmental Research Center, Grand Forks, North Dakota, 2011
- Saini, D., Ge, J., Botnen, L., **Pu, H.**, Sorensen, J., Gorecki, C., and Harju, J. "Denbury Sequestration Project Phase 2b Evaluation of Selected CO₂ EOR and Sequestration Sites, funded by Denbury Resources", sponsored by Denbury, 2012
- Gorecki, C., **Pu, H.**: "Evaluation of MMP for Bell Creek Field", Report for Denbury Resources Incorporated, EERC, 2011

Papers Published in Refereed Journal Papers

- **Pu, H.**, Yin, D., "A Numerical Simulation Study on Surfactant Flooding and Its Field Application in Daqing's Pilot Test", Journal of Petroleum Science and Technology, Vol. 27(5), 2009
- Yin, D., **Pu, H.**, "Numerical Simulation Study on Surfactant Flooding for Low Permeability Oilfield in the Condition of Threshold Pressure", Journal of Hydrodynamics, Ser. B, Vol.20(4), 2008
- Wang, W., **Pu, H.**, Yin, D., Gao, P., "The Study on Reasonable Infilling Method for Low Permeability Reservoir", Small Hydrocarbon Reservoirs, Vol.12(2), 2006
- Yin, D., **Pu, H.**, Wu, Y., "Numerical Simulation of Imbibition Oil Recovery for Low Permeability Fractured Reservoir", Journal of Hydrodynamics, Ser. A, Vol.19(4), 2004

Papers Published in Conference Proceedings

- Pu, H., Wang, Y., Li, Y.: SPE 180080 "How CO₂ Storage Mechanisms are Different in Organic Shale: Characterization and Simulation Studies", to be presented at SPE Europec featured at 78th EAGE Conference and Exhibition held in Vienna, Austria, 30 May–2 June 2016
- **Pu, H.**, Li, Y.: SPE 179533 "Novel Capillarity and Adsorption Quantification Method in IOR Process in Bakken Shale Oil Reservoirs", SPE Improved Oil Recovery Conference, Tulsa, OK, 11-13 April 2016
- **Pu, H.**, Li, Y.: SPE 178943 "Study of Condensate Blockage and Its Remedy in Eagle Ford Gas-Condensate Zone", to be presented at SPE International Conference & Exhibition on Formation Damage Control, Lafayette, Louisiana, USA, 24–26 February 2016
- **Pu, H.**, Li, Y.: CMTC 439769 "CO₂ EOR Mechanisms in Bakken Shale Oil Reservoirs", Carbon Management Technology Conference, Sugarland, Texas, 17–19 November 2015
- Li, Y., Pu, H., CMTC 439561 "Modeling Study on CO₂ Capture and Storage in Organic-Rich Shale", Carbon Management Technology Conference, Sugarland, Texas, 17–19 November 2015
- Jin, L., **Pu, H.**, Wang, Y., Li, Y., SPE 178507/URTeC-2148314 "The Consideration of Pore Size Distribution in Organic-rich Unconventional Formations May Increase Oil Production and Reserve by 25%, Eagle Ford Case Study", Unconventional Resources Technology Conference, San Antonio, TX, 20-22 July 2015
- **Pu, H.**, Xie, X., Yin, P., Morrow, N. R., SPE 134042 "Low-Salinity Waterflooding and Mineral Dissolution", 2010 SPE Annual Technical Conference and Exhibition
- **Pu, H.**, Wang, G., Han, G., SPE 120127 "Production Enhancement Through Pattern Modification: Analysis and Field Results", 2009 SPE Production and Operations Symposium
- **Pu, H.**, Xu, Q., SPE 118746 "An Update and Perspective on Field-Scale Chemical Flood in Daqing Oilfield, China", 2009 SPE Middle East Oil & Gas Show and Conference
- **Pu, H.**, Wang, G., Li, Y., SPE 118836 "Reservoir Simulation Study on Improvement of Waterflooding Effect for a Naturally Fractured Low Permeability Field in Daqing, China: a Successful Case", 2009 SPE Middle East Oil & Gas Show and Conference
- **Pu, H.**, Yin, D., SPE 109546 "Study of Polymer Flooding in Class III Reservoir and Pilot Test", 2008 16th SPE/DOE Improved Oil Recovery Symposium
- **Pu, H.**, Yin, D., SPE 114200 "Field Practice of Improvement of Waterflooding Effect for Mature Naturally Fractured Low-Permeability Field", 2008 SPE Western Regional Meeting

- **Pu, H.**, Yin, D., Chen, Y., Yang, F., SPE 111720 "Feasibility Study and Pilot Test of Polymer Flooding in Third Class Reservoir of Daqing Oilfield", 2008 SPE North Africa Technical Conference and Exhibition
- **Pu, H.**, Xie, X., Yin, P., Morrow, N., SPE 113410 "Application of Coalbed Methane Water to Oil Recovery from Tensleep Sandstone by Low-Salinity Waterflooding", 2008 16th SPE/DOE Improved Oil Recovery Symposium
- Xu, Y., **Pu, H.**, Shi, L., SPE 114199 "An Integrated Study of Mature Low-Permeability Reservoir in Daqing Oil Field, China", 2008 SPE Western Regional Meeting
- Yin, D., **Pu, H.**, SPE 112424 "A Numerical Simulation Study on Surfactant Flooding and Its Field Application in Daqing Oilfield", 2008 SPE EUROPEC Conference
- Xie, X., **Pu, H.**, Morrow, N., "Aspects of Coalbed Natural Gas Water and Oil Recovery", 2007 National Meeting of the American Society of Mining and Reclamation

TRAININGS

- Modern Production Data Analysis for Unconventional Reservoirs by Fekete, September, 2013
- Fundamentals of Geostatistics by Prof. Clayton Deutch, August 2013
- OilField Manager (OFM) Fundamentals by Schlumberger, July 2012
- CO₂-Based EOR (Miscible Flood) and CMOST by CMG, February 2011
- Fundamentals of Reservoir Simulation by Mike Carlson, September, 2011
- Petrel Reservoir Engineering by Schlumberger, September 2011

HONORS/AWARDS

- Featured in the article of supplement to February 2014 *JPT (Journal of Petroleum Technology)*: "Daqing: an Old Field is at the Center of New EOR Testing"
- SPE Scholarship by Denver Section, 2007-2008, 2008-2009
- Graduate Assistantship, University of Wyoming, 2006-2010
- National Scholarship for M.S. Study, China Ministry of Education, 2003-2006
- Outstanding Undergraduate Student of Year 2003, Daqing Petroleum Institute, China

Uncovering ASIA PACIFIC

EXECUTIVE INTERVIEWS

Sinopec Petronas Pertamina PTTEP KNOC Inpex Talisman Roc Oil <u>Me</u>dcoEnergi

COUNTRY ANALYSIS

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FEATURES

Shale Resources Map Technology Case Studies Converging Interests of NOCs and IOCs

SUPPLEMENT TO JOURNAL OF PETROLEUM TECHNOLOGY

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DAQING an old field is at the center of new eor testing

STEPHEN RASSENFOSS, JPT EMERGING TECHNOLOGY SENIOR EDITOR

aqing remains China's top oil field, producing nearly 290 million bbl (40 million tons) per year of oil, defying predictions of serious decline after more than 50 years of production.

Those involved take pride in the fact production has remained steady since the target was reduced 20% in 2004. "We want that to continue," said He Liu, in charge of production engineering and enhanced oil recovery [EOR] research at the Research Institute of Petroleum Exploration and Development (RIPED), which is part of Petrochina.

Liu's career has been tied to maintaining the field's output. Before moving to RIPED, he was the deputy general engineer at the Daqing oil field. He worked for 28 years there as part of a team whose responsibilities included managing the ever-evolving polymer-enhanced waterflooding system that is credited with 25% of the field's output.

Extending the life of the field will require another generation of EOR methods to postpone the natural decline of the reservoir as older zones are produced, Liu said. The push provided by water plus polymer adds to the amount of oil recovered, but there is a limit to how much of the oil is likely to be produced before the well reaches the point when the fraction of oil in the produced water is too low to justify further production.

RIPED is working on improved chemical combinations to extend Daqing's life past that point, and new methods that will allow it to efficiently identify and flood the many layers that have been missed, which tend to be thin and less permeable. Liu said that without improved EOR methods the benefit of polymer flooding would likely decline to 5% to 7% of the reservoir's total output.

China's domestic production depends on maximizing production from older fields. Daqing has been a center for developing these methods.

"The polymer-flooding technology has been implemented for a long time. It is very much their own," said Hui Pu, a research engineer at the University of North Dakota Energy & Environmental Research Center who has written about Chinese chemical EOR methods along with



Pipes throughout the Daqing field, located in the frigid northeast corner of China, are needed for the polymerenhanced water injection and production systems used to enhance production. *Photo: Jiamaomimi, depositphotos.com.*

employees of PetroChina working at Daqing. "They have a lot of experience in all types of reservoirs."

Nearly every sort of enhanced recovery method has been tried at Daqing. The list includes polymers; polymers plus various combinations of alkali and surfactants; colloidal dispersion gel; and carbon dioxide flooding. Even biological methods are being considered.

"They have tried everything available in EOR," said James Sheng, an associate professor at Texas Tech University, who worked at RIPED early in his career, before working as a reservoir engineer at several international oil and service companies.

Researchers in China are looking for ways to get at what is left in the complex formation. Liu calls it "redevelopment."

One of Liu's major interests is developing a system for efficiently putting zones into production that have been missed. The device, called the Integrated Bridge Eccentricity Packer with Steel Cable measuring and adjustment technology (IBEPC), is used to precisely manage flooding in zones that other tests have indicated failed to produce using waterflooding, often because they were bypassed because they are less permeable. The device combines a

DAQING FACTS

Major reservoirs: Lamadian, Saertu, Xingshugang Reservoir thickness: 2,300-3,900 ft Reserves: 36 billion bbl 00IP Reservoir description: multiple layers of sand Permeability: varies widely with layers Average reservoir temperature: 45°C [113°F] Crude: medium grade Salinity: low-6,000-9,000 mg/L [TDS 5,000-7,000 ppm]

Source: PetroChina

DAQING TIMELINE

Work on enhanced oil recovery [EOR] began early in the Daging oil field:

- **1959** Sognli-3 discovery well comes in
- 1960 First oil delivered to a refinery
- 1965 Waterflood testing begun
- **1972** First polymer-flood test
- 1976 Production first exceeds peak rate of 1 million B/D (50 million tons per year)
- 1987 Thin-layer reservoir development method developed
- **1988** Deeper horizons (to 4000 m) drilled adding 7 oil layers and 3 gas layers
- **1994** First test of alkaline-surfactant-polymer flood
- **1995** Start of polymer flooding throughout field
- 1996 Production peaks at 56 million tons per year
- 1998 First horizontal well drilled
- 2002 Qingshen gas field discovered
- 2004 Daging output target reduced by 20%

Source: PetroChina



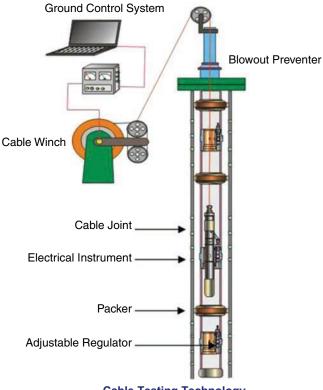
Workers maintain a pump in the aging Daging field where intense management has been used to maintain a high level of production. Photo: Jiamaomimi, depositphotos.com.

measuring tool that is able to measure water and polymer flows in specific areas in vertical wells, and adjustable injection control devices to precisely control the fluid flow and chemical mix.

Improved chemicals are also a priority in Daging, with expanding use of chemical combinations-primarily alkalinesurfactant-polymer (ASP). This combines the ability of polymer to increase the viscosity of water to near that of oil, and the ability of surfactants and alkali to reduce interfacial tension and free oil adhering to reservoir rock.

Making it work on a large scale will require improvements that reduce ASP's cost and side effects. Strong alkali can lead to scale deposits and broken sucker rods, plus increased pump maintenance and failures. It can also cause oil and water emulsions, making it difficult to separate out the oil. Finding a combination that is as effective, without strong alkali, is one of the difficult challenges Chinese EOR researchers are working on, Pu said.

Sheng is updating a paper delivered in 2013 surveying ASP test results worldwide. The initial paper covering projects six tests at Daging, making it the most active test site in



Cable Testing Technology

PetroChina developed a device to flood layers within a reservoir which have not been produced effectively using targeted waterflooding. The Integrated Bridge Eccentricity Packer with Steel Cable measuring and adjustment system, is used to regularly adjust flooding aimed at reaching all the productive zones in a well. The electrical instrument measures water-polymer flow in areas that previous testing indicated were likely missed by previous flooding, which likely went to more permeable zones. Several times a year it is placed near water injection points and the information is used to precisely manage injection. The packers are used to isolate the area, focusing the fluid injected by the adjustable regulator on previously unproduced areas.

the global survey. Information on more recent ASP projects six tests at Daiqing, making it the most active test site in the global survey. Information on more recent ASP projects in Daqing shows the scale of later ASP flooding tests is much larger.

What has been learned in Daqing is being applied in other parts of China. RIPED is working on chemicals that can tolerate conditions in reservoirs elsewhere in the country with higher temperatures and salinity levels than Daqing, which is a hospitable environment for polymers, Liu said. RIPED is also looking for ways to deliver productionenhancing chemicals more effectively, and match what is used to reservoir conditions.

"There are some new-generation techniques such as horizontal wells, separated-zone injection, intelligent completion, and integrated reservoir and production management systems and so on," Liu said.

"Each year we derive new data from downhole to analyze production performance so we can make adjustments to the production strategy," he said. Looking ahead, "there are many, many new techniques."

Driven by Need

Daqing is the largest field in China based on reserves and production, according to the US Energy Information Administration (EIA), at a time when demand for oil is more than twice what the country can produce. It has also long been a symbol of China's drive to become a world economic power.

The field has its own oil company, PetroChina's Daqing Oilfield Company; a museum; and a legendary oilman, Wang Jinxi, known as the Iron Man. The story of how he and his crew overcame the elements and rudimentary drilling equipment to drill the discovery well was used in the past to exhort workers to demonstrate "the spirit of diligence, loyalty, bravery, and tenacity."

Daqing was discovered in 1959, first oil was delivered to a refinery the next year, and waterflooding began 5 years later as China pushed to accelerate development of the field to demonstrate that Chinese workers could build a domestic oil industry.

By 1976 Daqing, which encompasses three large reservoirs, hit its peak level of 1 million B/D (50 million tons a year). That level was maintained until 2004, when the target was lowered by 20%.

The drive to maintain Daqing production led PetroChina to begin working on polymer flooding more than 30 years ago. It began large-scale use of it in the mid-1990s. China stuck with its program to use chemicals to improve waterflooding on a large scale at a time when interest in chemical EOR had waned in other countries because prices were low and oil was plentiful.

China's national oil industry was focused on the government mandate to produce all the oil available in its fields. "Because production levels were based on what the state wanted, they did it even though at that time prices were low or prices were high," Sheng said.

In the past decade, there has been a strong economic need for Daqing's output. China's domestic oil output is rising—it totaled 4.42 million B/D in 2012, up 1.6% from the previous year according to the EIA—but demand rose 4.3% to 10.3 million B/D in 2012, a shortfall of 5.88 million B/D.

While Daqing's 36 billion bbl of original oil in place make it among the world's largest fields, maintaining its production has tested the energy and ingenuity of those running it because there is little gas pressure or natural water drive pushing the oil out of the ground.

Waterflooding was started early on. Without it, Daqing's production would have been minimal. Water injection should



Pumps pull water and oil from Daqing oil field which has long been a major testing ground for enhanced oil recovery methods. Photo: Jiamaomimi, depositphotos.com.

push ultimate recoveries from 40% to as high as 45%, Pu said. Adding polymer is likely to get recovery past 50%, and going to ASP holds the promise of a 60%-plus ultimate rate of recovery of the oil, he said.

Post Polymer

Polymer has its limits. Sheng, who recently wrote a book on the subject, *Modern Chemical Enhanced Oil Recovery: Theory and Practice*, said polymer can add 5% to 10% to what waterflooding could ultimately produce, and ASP could add 20% to 30%. ASP is an option when a polymer waterflood reaches its economic limit, with a water cut of about 98% in Daqing.

The six Daqing ASP tests, reported in a 2013 paper by Sheng, resulted in incremental recoveries of about 20% and significant reductions in the water cut, and led to expanded testing. "They are using ASP on a large scale now," Sheng said. There are ASP applications with more than 100 injection wells, compared to an average of 5 injectors in early tests. Mass use will depend on the cost and reduced operating issues. "There are two sides to everything," he said.

Since strong alkali is blamed for costly maintenance issues, PetroChina is seeking a substitute—a weak alkali that limits side effects without reducing EOR performance or a breakthrough technology that allows a combination of surfactant and polymer to be as effective as ASP, Pu said.

Another method tried in the field has been carbon dioxide injection. Liu said they have had "some success" with

 $\rm CO_2,$ which is used to free oil adhering to reservoir rock and to reduce the viscosity of heavier crude.

"For 5 years we have had CO_2 flooding," he said. There is CO_2 available in Daqing because "You must separate natural gas from CO_2 ," which produce together around Daqing and are separated during processing, Liu said. Reinjection answers the question: "After separation, where does it (CO_2) go?"

Well by Well

PetroChina has intensively managed the huge field with the meticulous attention to detail that traditional Chinese farmers apply to their small plots.

Polymer flooding has been credited with 25% of the production at Daqing, but there is a significant human element required to match specific chemicals to the location. "They need experts with a range of expertise to make it all work together efficiently," Sheng said.

Daqing's complexity rewards attention to detail. It is a thick sandstone reservoir with large variations in rock properties from layer to layer, as well as barriers, such as fractures, leaving many pockets of oil.

In one area researchers studied the properties of 36 oil-bearing layers, with permeability ranging from 15 mD to 710 mD. The goal of that test was to target the water injection so that it flooded hard-to-penetrate layers which had been missed, despite drilling more closely spaced wells and reducing the number of producing wells per injection well. One paper described a pattern where there were two wells injecting water for every three producing wells.

Injection programs are adjusted on a well-by-well basis. The polymer concentration and type of polymer is chosen based on the reservoir to be flooded. The molecular weight of the polymer and the concentration of the polymer solution are adjusted to fit the injection interval, Pu said.

PetroChina is looking for the right polymers to match with lower-permeability layers. Core testing to see which molecular weight of polymer can flow through rocks from different locations in a conventional reservoir found that samples with the same measured permeability may not be able to accommodate the same-sized polymer molecules. One paper (SPE 14595) said that testing "suggests that the pore structures of the oil layers in different areas are not the same."

A 2006 paper coauthored by Liu, said there is a clear need for EOR after polymer flooding. "During the thicklayer polymer flooding period in Daqing oil field," the paper said, "the recovery rate can only reach 52% approximately, and nearly half of the geological reserves will remain underground, so conducting new after-polymer enhanced oil recovery technology is demanded." **JPT**

For further reading:

SPE 100855 Commercial Success of Polymer Flooding Application in Daqing Oilfield—Lessons Learned by Wang Yupu and Liu He, Daqing Oilfield Co.

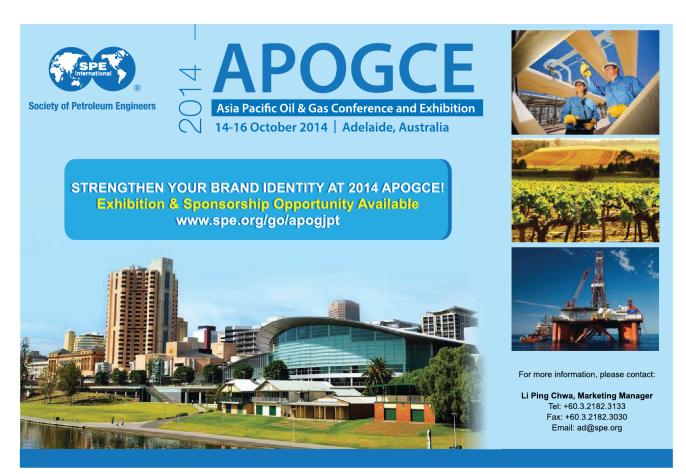
SPE 165358 A Comprehensive Review of Alkaline-Surfactant-Polymer (ASP) Flooding by James J. Sheng, SPE, Texas Tech University

SPE 89175 Advances in Polymer Flooding and Alkaline/ Surfactant/Polymer Processes as Developed and Applied in the People's Republic of China by H.L. Chang, SPE, Intratech Inc., and Z.Q. Zhang, PetroChina

SPE 118746 An Update and Perspective on Field-Scale Chemical Floods in Daqing Oilfield, China by Hui Pu, SPE, Department of Chemical and Petroleum Engineering, University of Wyoming, and Qinglong Xu, Exploration and Development Research Institute of Daqing Oilfield Co.

SPE 109546 Study of Polymer Flooding in Class III Reservoir and Pilot Test by Hui Pu, SPE, University of Wyoming, and Daiyin Yin, Key Laboratory of Enhanced Oil Recovery of Ministry of Education, Daqing Petroleum Institute

SPE 164595 The Polymer Flooding Technique Applied at High Water Cut Stage in Daqing Oilfield by Wang Yuming and Pang Yanming, Daqing Oilfield Company, et al.



Michael D. Mann, PhD

Chester Fritz Distinguished Professor, Department of Chemical Engineering Executive Director, Institute for Energy Studies, University of North Dakota 701-777-3852; michael.mann@engr.und.edu

EDUCATION AND TRAINING

Ph.D. Energy Engineering, University of North Dakota, 1997 M.B.A. Business Administration, University of North Dakota, 1987 M.S. Chemical Engineering, University of North Dakota, 1981 B.A. Chemistry, Mathematics, Mayville State University, 1979

RESEARCH AND PROFESSIONAL EXPERIENCE

2014 – Present: Executive Director, Institute for Energy Studies

- Help realize the Institute for Energy Studies goal of developing UND into a premier "Energy University" that "inspires the creation of new knowledge to enable the development of revolutionary energy technologies, train the next generation of energy experts, and establish advanced industries required to make affordable emissions free energy technologies a reality".
- Responsibilities include identifying key technical and economic barriers to the development of secure, affordable, and reliable energy production technologies; identifying proposal opportunities and develops new relationships with potential partners; and drawing from resources across campus building teams to deliver the research, education, and outreach required to meet the needs of public and private partners.

2009-14: College of Engineering (Associate Dean 2013-24; Associate Dean for Research 2009-13)

- Provide advice and support to the Dean in issues related research and development within the college and support academic affairs.
- Responsible for the implementation of the college's major research goals, promoting a culture of research in the college, enhancing research opportunities for faculty and students, and providing administrative oversight for proposal submittal and grant accounting.

2008: Interim Dean, UND School of Engineering and Mines

• Responsible for all academic and research activities within SEM. In this role he expanded his leadership experience and broadened his overview of the campus wide talents and opportunities for enhancing UND's reputation as a leader in energy research and education.

1999 – Present: UND Department of Chemical Engineering (Professor, 2006-present; Chair 2005-13; Associate Professor, 1999-2006)

- Developed a reputation as an engaging teacher, excellent researcher, and inspirational leader.
- Awarded UND's highest honor, the Chester Fritz Distinguished Professorship in 2009 in recognition for his accomplishments in research, teaching, and service.
- Led the Department of Chemical Engineering to UND's top departmental awards for Excellence in Research in 2005 and 2011 and Excellence in Teaching in 2007.
- Co-founder of the SUstainable eNergy Research, Infrastructure, and Supporting Education (SUNRISE) group in 2004.
- SUNRISE now has over 30 faculty participants from 12 different departments and 4 North Dakota Universities with over \$20 million in research grants.

1981-99: UND Energy & Environmental Research Center (Sr. Research Mgr, Advanced Processes and Technologies 1994-99; Research Mgr, Combustion Systems 1985-94; Research Engineer 1981-85):

- Activities evolved from hands on research to the development and marketing of ideas and technology.
- Involved in a wide range of technology development, including energy production from combustion and gasification, wind, and geothermal resources. Highlights include management of over \$15 million in research projects; design, installation, and operation of a 1 MW_{th} CFBC; design, installation, and operation of a 250 lb/hr gasifier; manager for project for the development of small power systems for Alaskan villages; and the development of a small-modular fluid-bed combustion system (0.5 to 5 MW)

PUBLICATIONS (selected from over 150)

- Hussain, M.; **Mann, M.D.**; Swanson, M.L.; Musich, M.; "Testing of Lithium Silicate and Hydrotalcite as Sorbents for CO₂ Removal from Coal Gasifiction", *in* Proceedings of the 24th Annual International Pittsburgh Coal Conference, Johannesburg, South Africa, September, 2007.
- Karki, S., **Mann, M.**; Salehfar, H.; "Substitution and Price Effects of Carbon Tax on CO₂ Emission Reduction from Distributed Energy Sources", *Asian Journal of Energy & Environment*"
- Bandyopahdyay, G.; Bagheri, F.M.; **Mann, M.D.**; "Reduction of Fossil Fuel Emission in US: A Holistic Approach Towards Policy Formulation", *Energy Policy*; 2007, 35 (2) 950-965.
- Knutson, R. (Presenter & Author), **Mann, M. D.**, Bayless, D., Karim, T., 2011 Clearwater Clean Coal Conference, "CFD Modeling of Hydrogen Production from Coal via Lewis Ultra-Superheated Steam (USS) Fluidized Bed Gasification," Clearwater, FL. (June 2011).
- Nel, M.V.; Mann, M.D.; Folkedahl, B.; Timpe, R.; "Comparison of Sodium Chloride Removal Abilities of Kaolin Clay and Calcined Bauxite as Possible Sorbents for Gasification", *in* Proceedings of the 24th Annual International Pittsburgh Coal Conference, Johannesburg, South Africa, September, 2007.
- Zhao, Y., **Mann, M.D.**, Pavlish, J.P., Mibeck, B.A.F.; Dunham, G.E.; Olson, E.W.; "Application of Gold Catalyst for Mercury Oxidation by Chlorine", *Environmental Science and Technology*; 2006 40: 1603.
- Karki, S; Kulkarni, M.; **Mann, M.D.**; Salehfar, H.; "Efficiency Improvements through Combined Heat and Power for On-Site Distributed Generation Technologies", *Cogeneration and Distributed Generation Journal*, Vol 22, No 3, 2007, pp 19-34.
- Pavlish, J.P.; Sondreal, E.A.; Mann, M.D.; Olson, E.S.; Galbreath, K.C.; Laudal, D.L.; Benson, S.A. "A Status Review of Mercury Control Options for Coal-Fired Power Plants" *Fuel Process. Technol.* 2003, 82: 89-165
- Sondreal, E.A.; Benson, S.A.; Hurley, J.P.; **Mann, M.D.**; Pavlish, J.H.; Swanson, M.L.; Weber, G.F.; Zygarlicke, C.J. "Review of Advances in Combustion Technology and Biomass Firing". *Fuel Processing Technology* 2001, 71 (1-3), 7-38.
- Mann, M.D.; Knutson, R.Z.; Erjavec, J.; Jacobson, J.P.; "Modeling Reaction Kinetics for a Transport Gasifier", *Fuel* 83 2004 1643-1650.

POSTDOCTORAL SCHOLAR: Eduardo Hernandez Pacheo, 2005, Intel; Motjaba Sadraemli, 2009, University of Tehran, Iran.

- PH.D. ADVISEES: Eduardo Hernandez Pacheo, 2004, Intel; Yongxin Zhao, 2005, ARCADIS, Shankar Karki, 2006, Electric Power Research Institute; Kevin Harrison, 2006, National Renewable Energy Laboratory; Rhonda Hill, 2007, Clipper Energy; Gopal Bandyopadhyay, 2007, Pacific Northwest National Laboratory; Philip Hutton, 2009, Energy & Environmental Research Center; Nilesh Dale, 2009, Bhanuaben Patel, 2010; Taehee Han, 2010, Nissan; Christian Biaku, 2010; Ryan Knutson, 2011; Udhe Corp of America; Kirtipal Barse, 2014, UND; Stacy Bjorgaard, 2015 UND. Jivan Thakare, 2016, Energy & Environmental Research Center (I currently advise two Ph.D. students)
- **M.S. ADVISEES:** Jermie Moberg, 2001, Intel; Jason Jacobson, 2001, Trane; Muhammad Amanullah, 2003, University of Minnesota-Duluth; Biplab Mukerjee, 2004, Washington University, St. Louis,

Ph.D. program; Kyle Martin, 2004, EERC; Devinder Singh, 2005, Kansas State University, Ph. D. program; Devdutt Shukla, 2005, University of Missouri-Rolla, Ph.D. program; Srinivas Koli, 2005, University of Kentucky, Ph.D program; Joshua Strege, 2005, EERC; Carol Horabik, 2006, University of Minnesota-Duluth; Xi Hong, 2006, Michigan Tech, Ph.D. program; Prasana Sardenia, 2006, Microbeam Technologies; Christopher Kinchin, 2006, National Renewable Energy Laboratory, Prasad Chavan, 2007, Hutchinson Technologies; Taskin Karim, 2007, University of Texas; Marika Nel, 2007, Northwest University, South Africa; Mohammed Hussain, 2007, Kansas State University PhD program; Jason Hrdlicka, 2007, National Renewable Energy Laboratory; Rahul Pandry, 2008, private company in India; Taskin Karim, 2010, University of Houston; Shankar Lande, 2011; Sam Cowart, 2012, West Point Military Academy; Kirt Leadbetter, May 2014; Chris Haugen, 2015, Hess Corporation. (I currently advise two MS students)

Xuefei Zhang, Ph.D.

Postdoctoral Researcher Department of Chemistry University of North Dakota Grand Forks, ND 58202 xuefei.zhang@gmail.com

EDUCATION

2013 PhD Chemical Engineering, University of North Dakota, Grand Forks, ND 2007 M.S. Physics University of Maine, Orono, ME 2001 B.S. Material Science & Engineering, Tianjin University, China

WORK EXPERIENCE

Feb, 2015 - Postdoc Researcher, Department of Chemistry, University of North Dakota

- Synthesized and characterized graphene and silica nanoparticles and nanowires.
- Developed tooling and methods for Autosorb iQ Chemsorption & Physisorption nanometric analyser.
- Taught general chemistry laboratories and tutoring sessions (Chem 121 & 122).

2013-2014 Research Engineer, Dept. of Geology & Geological engineering, Univ of North Dakota

- Investigating the use of surfactant imbibition to enhance oil recovery from Bakken shale.
- Constructed wettability and permeability test experimental setup and data acquisition system.

2009-2013 Research Assistant, Department of Chemical Engineering, University of North Dakota

- Explored and proved the idea of using polymeric membrane contactors for CO₂ recovery; designed and established an experimental and data acquisition system for conceptual validation.
- Proficient with Design of Experiment (DOE) and process optimization, as well as analytical instrumentation of Gas Chromatography (GC-TCD/FID with methanizer), GC/MS, Scanning Electron Microscope (SEM), UV-vis Optical Absorption Spectroscopy, Scanning Differential Calorimeter (DSC)
- Prepared technical presentation and reports for funding agents as well as research proposals and journal articles.

2007-2008 Engineer, IC Interconnect, Colorado Springs, CO

- Developed customized process recipes to meet customers' reliability requirements and design rules
- Engaged with customers regarding process qualification and failure modes effect analysis (FMEA)
- Proficient with various coating technologies including sputtering, evaporation, eletroless plating.
- Installed a new electroless plating line in China and passed (internal and external) process qualification
- Specialized on flip chip wafer level chip scale bumping by electroless Ni/Au plating for electronic packaging processes
- Skilled with products characterization methods by optical microscopy, surface profilometer, bump shear and reliability tests

2007 Adjunct Faculty, Community College of Denver, Denver, CO

- Taught Introductory Physics lectures and labs (PHY 111)
- Communicated effectively with students, staff and faculty regarding course expectations and outcomes

2003-2007 Research Assistant, Lab for Surface Science & Technology, Univ. of Maine, Orono, ME

• Investigated novel Zr-Si-N thin film materials including synthesis, processing, and characterization

- Experienced with RF/DC magnetron sputter deposition, electron beam evaporation, ECR plasma-assisted deposition
- Proficient with materials characterization techniques including X-ray Photoelectron Spectroscopy (XPS), X-ray Diffraction (XRD), X-ray Reflectivity (XRR), Auger Electron Spectroscopy (AES), Reflection High Energy Electron Diffraction (RHEED), Atomic Force Microscopy (AFM), and UVvis Spectroscopy

2003-2007 Teaching Assistant, Department of Physics and Astronomy, Univ. of Maine, Orono, ME

- Instructed Lab for Introductory Physics courses (PHY107, PHY121, PHY122) and Introductory Astronomy courses (AST110, AST110–online)
- Operated facilities at Maynard F. Jordan Planetarium & Observatory

2001-2003 Building Materials Test Engineer, China State Construction Engineering Corporation, Beijing, China

- Developed work procedures and manuals for quality assurance system
- Sampled in-field statistically and characterized in-lab the building materials including steel bar, concrete, cement, soil, waterproof materials
- Customized concrete mix design based on parameters for different projects

SELECTED PUBLICATIONS

- **X. Zhang**, and B. M. Tande, "*Regeneration of carbon dioxide from CO*₂ *saturated monoethanolamine by membrane contactors*", Separation Science and Technology, Vol. 49(1), 2014, 1-11
- **X. Zhang**, M. S. Byrne, and R. J. Lad, "*Structure and optical properties of Zr_{1-x}Si_xN thin films on sapphire*", Thin Solid Films 518 (2009) 1522-1526.

Book Chapters

• X. Zhang and H. Li, "Corrosion Science and Technology", 7 Chapters, China Machine Press, in Press

PRESENTATIONS

- X. Zhang, "Efficient Regeneration of Chemical Solvents for CO₂ Capture by Polymeric Membranes", Department of Petroleum Engineering, Montana Tech of The University of Montana, Butte, MT, September, 2014.
- X. Zhang, "Synthesis and Characterization of Nitride/Oxide Thin Films", Ningbo Institute of Materials & Technology, Chinese Academy of Sciences, Ningbo, Jiangsu Province, P. R. China, September, 2013.
- X. Zhang, "*Polymeric Membranes for CO*₂ *Capture*", Scholarly Forum, The University of North Dakota, Grand Forks, ND, April, 2012.
- X. Zhang and B. M. Tande, "*Regeneration of CO*₂ *Solvent with Polymeric Membranes*" Annual Meeting of the American Institute of Chemical Engineers, Minneapolis, MN, October, 2011.
- X. Zhang, "Synthesis and Characterization of Zr-Si-N Thin Films", Department of Chemistry, The University of North Dakota, Grand Forks, ND, April, 2010.
- **X. Zhang** and Ř. J. Lad, "*Growth and Structure of Zr-Si-N Thin Films*" March Meeting of the American Physical Society, Denver, CO, March, 2007.

Appendix D: Relevant UND Laboratory Facilities and Capabilities

The research team has access to advanced laboratory facilities both internal and external to perform the proposed research. Access to instrumentation in the UND Department of Petroleum Engineering, Department of Chemistry, and Microscopy Imaging Center at the UND School of Medicine is not restricted and is not currently charged. The access to the Materials Characterization Laboratory at UND is not restricted and a nominal laboratory service fees have been budgeted in this proposal. Micro-CT and imaging scan on several core plug samples will be conducted at external lab-Micro-CT Laboratory at the University of Minnesota, and a small amount of it lab service fees is shown in the budget.

The Co-PI's Nano Chemistry Labratory has a dedicated chemistry wet laboratory for synthesis of nanoparticles, 900 sq ft. The laboratory is fully equipped for all synthesis studies required and has ample desk space, internet/phone connection for students, fellows, and staff. There is a dark room laboratory equipped with a Scanning Electron Microscope and a Fluorescence Microscope.

Petroleum Engineering Laboratory

- Vinci EOR core flood system for polymer and chemical injection (CHEMFLOOD)
- Vinci Visual Fluid-Eval PVT system
- High Pressure Pump Hastelloy
- CSC Interfacial Tensiometer
- TKA209 Gas Permeameter
- TPI219 Helium Porosimeter
- Hassler Coreholder
- Advanced Attrition Test System
- Tracer IV-SD System
- Wettability Test
- Viscometer
- Rehometer
- Interfacial Tension Cell

Materials Characterization Laboratory

- Oxford Geospec2 NMR
- SEM (FEI Quanta 650 FEG, and Hitachi SEM)
- Rigaku Smartlab XRD
- Rigaku Supermini 200 x-ray fluorescence (XRF)
- OFITE 740 Gamma Ray Logger
- High-pressure porosimeter
- Micropore and Chemisorption analyzer (Autosorb-iQ-C Chemisorption with built in TCD)
- Malvern Zetasizer Nano ZS with NIBS
- PerkinElmer Lambda 1050 UV/Vis/NIR spectrophotometer

Nano Chemistry Laboratory

- IX71 Olympus fluorescence microscope
- HORIBA fluorolog-3 spectrofluorometer
- HORIBA fluorescence microplate reader
- Malvern Zetasizer Nano ZS with NIBS
- Micropore and Chemisorption analyzer (Autosorb-iQ-C Chemisorption with built in TCD)
- PerkinElmer Lambda 1050 UV/Vis/NIR spectrophotometer
- Eppendorf centrifuge 5810 R with temperature control

- Hitachi SU8010 field emission scanning electron microscope with an Oxford X-Max energy dispersive X-ray spectrometer (EDS).
- Matson FT-IR spectrometer
- Philips X'Pert Pro X-ray diffractometer
- Bruker advance 500 high-field superconducting NMR spectrometer
- Siemens P-4 single crystal X-ray diffractometer(XRD)
- Brinkmann (Eco-Chemie) PGSTAT 302A with Frequency Response Analyzer

Microscopy Imaging Center at the UND School of Medicine

- Olympus Fluoview II confocal laser scanning microscope (inverted)
- Hitachi 4700 field emission scanning electron microscope
- Hitachi 7500 transmission electron microscope.

Micro-CT Laboratory at the University of Minnesota

- X5000 high resolution microCT system with a twin head 225 kV x-ray source and a Dexela area detector (3073 x 3889 pixels)
- 3D reconstructions are processed using 4 Nvidia Tesla C2075 GPUs

Appendix E: Letter of Support



Yinghui Li, Ph.D. President and Co-Founder InPetro Technologies Inc. 9800 Centre Pkwy, Suite #860 Houston, TX 77036 855-894-6863 yinghui.li@inpetrotechnologies.com

October 26, 2016

Hui Pu, Ph.D. Assistant Professor Department of Petroleum Engineering University of North Dakota Grand Forks, ND 58202

Dear Hui:

InPetro Technologies Inc.(InPetro) is very pleased to support your proposed effort entitled "Functional Nanoparticle-Augmented Surfactant Fluid for Enhanced Oil Recovery in Williston Basin". InPetro will provide support, specifically, we will provide our newly-developed reservoir simulation software for unconventional oil & gas reservoirs and relevant training and consulting for this software as in-kind support. Our simulator includes the effect of capillarity with the consideration of pore size distribution characteristics, a key features of shale formations which are not considered in commercial software. In addition, adsorption models using a local density optimization algorithm were developed and included to our simulator to better address the incapability of Langmuir model for wet and liquid-rich formations. We believe that our reservoir simulator can significantly complement your proposed important EOR study in Bakken Formation.

We understand that you and your research team at UND is seeking the support of the North Dakota Industrial Commission's Oil & Gas Research Council (NOIC-OGRC). We are confident that the proposed effort will greatly contribute to the EOR efforts in Bakken Formation, and could develop a new pathway to efficiently improve Bakken hydrocarbon recovery in an environmentally sustainable manner.



InPetro's in-kind contribution will include the provision of our reservoir simulator, training and consulting for usage of our software. The contribution of our reservoir simulation software is worth at least \$75,000 per year, plus \$3,750 for training and consulting per year. Our three years' contribution will be at a value of \$236,250 in total.

InPetro's commitment is, of course, contingent on Hui's attainment of the necessary funding from NDIC-OGRC. Please do not hesitate to contact me if you need further clarification or would like to discuss this effort further.

Sincerely,

Yinghui Li President and Co-Founder InPetro Technologies Inc.

Appendix F: Detailed Budget and Budget Justification

The following three tables are the detailed breakdown costs of each personnel and nonpersonnel for Year 1, Year 2 and Year 3, respectively. We request a total of \$769,134 (direct costs: \$562,212; indirect costs: \$206,922) from NDIC-OGRC to undertake this crucial study described above. The majority of funds will go towards personnel expenses for the essential but time-consuming activities of developing a promising enhanced oil recovery methodologies for Bakken tight formation. This includes summer support for the PI and Co-PI, two other key personnel, and support for a graduate student assistant. Salaries/benefits are escalated at a rate 5% per annum from 2016/2017. In addition, UND policies require a 39% overhead to cover facilities and other support provided by our institution.

A. PERSONNEL

Dr. Hui Pu, PI, and Dr. Julia Zhao, Co-PI, will be responsible for the overall coordination and supervision of all aspects of the study, and the successful execution of the project. This includes supervising staff/students; coordinating team member efforts; scheduling and staff assignments; quality control and project management. In addition, PI and Co-PI will lead the efforts of each tasks, assist with data analyses, and be responsible for reporting the study's findings. PI and Co-PI request 2 months of summer salary and associated benefit for each year. The total of three years of request will be: Dr. Pu's salary: \$56,039, benefits \$14,009; Dr. Zhao's salary: \$73,801, benefits \$18,450. UND will provide the same amount of match of two month salary and benefits for Dr. Zhao and Dr. Pu.

Dr. Michael Mann, key personnel, will be participating in the Task 3 – Characterization of Bakken Core Samples efforts. He will also assist in project reports preparation. Dr. Mann will receive \$15,000 salary and associated benefits of \$3,750 during the three years of the project.

Dr. Xuefei Zhang, key personnel, will be leading Task 2 – Surfactant Screening and Nanoparticle-Surfactant Effects and conduct experimental study as a key member in all other three tasks. Dr. Zhang will put 36 calendar month efforts for experimental study, experimental data analysis and interpretation, reports preparation. The salary for Dr. Zhang will be \$151,320 during the three years of project with a total of associate benefits of \$60,528.

B. OTHER PERSONNEL

Three Research Assistants (2 PhD students and 1 Master student): each research assistant will contribute an effort of 36 Calendar Months. These individuals will be trained by key personnel to conduct scientific research for the ultimate goal of developing EOR process for Bakken Formation. Two PhD students' salary and benefits will not come from NDIC-OGRC grant, but will be supported by funding from the UND. So only 1 Master student's salary requests from NDIC, the salary for this research assistant will be \$40,800 and associated benefits will be \$33,915 during the three years of project.

C. MATERIALS AND SUPPLIES

General Research supplies are calculated at approximately \$16,000 per year to purchase materials to synthesize functional nanoparticles, surfactants, reagent grade salts, gases, glassware, nuts, bolts, and piping used in the laboratory studies.

D.EQUIPMENT

A variety of advanced laboratories at UND are equipped with most of laboratory instruments needed in the proposed study. Funds are requested to purchase one oven (\$7,000). The oven with adjustable thermostat temperature control is essential for reservoir temperature conditions experiments, such as surfactant and nanoparticles screening tests, adsorption tests, stability test, phase behavior, and spontaneous imbibition test.

E. LABORATORY FEES

Most of our rock characterization experiments will be conducted at UND's Materials Characterization Laboratory. Materials Characterization Laboratory is very supportive of this proposed project and will only charge an institutional service fee. \$3,000 is budget for this purpose. The mirco-CT and imaging studies will provide key insight to the understanding of Bakken core samples, these tests will be conducted at an external lab-- Micro-CT Lab at the University of Minnesota, and a total of \$3,600 is planned for micro-CT laboratory fee.

F. TRAVEL

Travel is estimated on the basis of UND travel policies which can be found at <u>https://und.edu/finance-operations/accounting-services/travel-employee.cfm</u>. Travel may include site visits, professional meetings, and conference participation as indicated by the scope of work and/or budget. The estimated travel costs of \$10,000/yr will enable the researchers to travel to professional conferences to present findings associated with the investigation.

G. OFFICE SUPPLIES

Office supplies estimates are based on prior experience. \$3,000 is planned for costs of office supplies for the proposed project.

	Year 1, N	DIC Share		Year 1, UN	D Share		Industry
EXPENSES, Personnel	Salary	Benefit	Tuition	Salary	Benefit	Tuition	Sponsor's Share
Hui Pu, Pl	17,776	4,444		17,776	4,444		
Julia Zhao, Co-Pl	23,410	5,853		23,410	5,853		
Michael Mann, Key personnel	5,000	1,250		10,000	2,500		
Xuefei Zhang, Key personnel	48,000	19,200					
Three Graduate Students	13,200	3,000	7,973	45,990	6,000	21,029	
Total Personnel	\$149,105			\$137,001			
EXPENSES, Nonpersonnel							
Supply/Materials-Professional	16,000						78,750
Equipment>\$5,000	7,000			49,816			
Lab fees: Materials Characterization Lab@UND	1,000						
Lab fees: Micro-CT Lab @Univ. of Minnesota	1,200						
Travel, Meetings, Conferences	10,000			2,000			
Office Supplies	1,000						
Total Nonpersonnel	\$36,200			\$51,816			
Total Direct Expenses	\$185,305			\$188,818			
F&A (39% of Direct Costs)	\$66,430			\$46,009			
TOTAL EXPENSES	\$251,735			\$234,827			

	Year 2, N	DIC Share)	Year 2, UN	D Share		Industry
EXPENSES, Personnel	Salary	Benefit	Tuition	Salary	Benefit	Tuition	Sponsor's Share
Hui Pu, Pl	18,665	4,666		18,665	4,666		
Julia Zhao, Co-Pl	24,581	6,145		24,581	6,145		
Michael Mann, Key personnel	5,000	1,250		10,000	2,500		
Xuefei Zhang, Key personnel	50,400	20,160		0	0		
Three Graduate Students	13,596	3,090	8,212	47,370	3,090	21,427	
Total Personnel	\$155,765			\$138,444			
EXPENSES, Nonpersonnel							
Supply/Materials-Professional	16,000						78,750
Equipment>\$5,000	0			49,816			
Lab fees: Materials Characterization Lab@UND	1,000						
Lab fees: Micro-CT Lab @Univ. of Minnesota	1,200						
Travel, Meetings, Conferences	10,000			2,000			
Office Supplies	1,000						
Total Nonpersonnel	\$29,200			\$51,816			
Total Direct Expenses	\$184,965			\$190,260			
F&A (39% of Direct Costs)	\$68,934			\$46,416			
TOTAL EXPENSES	\$253,898			\$236,676			

	Year 3, N	DIC Share)	Year 3, UN	D Share		Industry
EXPENSES, Personnel	Salary	Benefit	Tuition	Salary	Benefit	Tuition	Sponsor's Share
Hui Pu, Pl	19,598	4,900		19,598	4,900		
Julia Zhao, Co-Pl	25,810	6,452		25,810	6,452		
Michael Mann, Key personnel	5,000	1,250		10,000	2,500		
Xuefei Zhang, Key personnel	52,920	21,168		0	0		
Three Graduate Students	14,004	3,183	8,458	48,791	6,183	21,838	
Total Personnel	\$162,742 \$146,071						
EXPENSES, Nonpersonnel							
Supply/Materials-Professional	16,000						78,750
Equipment>\$5,000	0			49,816			
Lab fees: Materials Characterization Lab@UND	1,000						
Lab fees: Micro-CT Lab @Univ. of Minnesota	1,200						
Travel, Meetings, Conferences	10,000			2,000			
Office Supplies	1,000						
Total Nonpersonnel	\$29,200			\$51,816			
Total Direct Expenses	\$191,942			\$197,887			
F&A (39% of Direct Costs)	\$71,559			\$49,231			
TOTAL EXPENSES	\$263,501			\$247,118			

Summary of Budget

EXPENSES, Personnel	Year 1, NDIC Share	Year 1, UND Share	Year 1, Industry Sponsor Share	Year 2, NDIC Share	Year 2, UND Share	Year 2, Industry Sponsor Share	Year 3, NDIC Share	Year 3, UND Share	Year 3, Industry Sponsor Share
Hui Pu, Pl	22,220	22,220		23,331	23,331		24,498	24,498	
Julia Zhao, Co-PI	29,263	29,263		30,726	30,726		32,262	32,262	
Michael Mann, Key personnel	6,250	12,500		6,250	12,500		6,250	12,500	
Xuefei Zhang, Key personnel	67,200			70,560			74,088		
Three Graduate Students	24,173	73,019		24,898	71,887		25,645	76,811	
Subtotal	149,105	137,001		155,765	138,444		162,742	146,071	
Total Personnel	\$286,107			\$294,208			\$308,813		
EXPENSES, Nonpersonnel									
Supply/Materials- Professional	16,000		78,750	16,000		78,750	16,000		78,750
Equipment>\$5,000	7,000	49,816			49,816			49,816	
Lab fees: Materials Characterization Lab@UND	1,000			1,000			1,000		
Lab fees: Micro-CT Lab @Univ. of Minnesota	1,200			1,200			1,200		
Travel, Meetings,Conferences	10,000	2,000		10,000	2,000		10,000	2,000	
Office Supplies	1,000	-		1,000			1,000	-	
Total Nonpersonnel	\$36,200	\$51,816	\$78,750	\$29,200	\$51,816	\$78,750	\$29,200	\$51,816	\$78,750
Total Direct Expenses	\$185,305	\$188,818		\$184,965	\$190,260		\$191,942	\$197,887	
F&A (39% of Direct Costs)	\$66,430	\$46,009		\$68,934	\$46,416		\$71,559	\$49,231	
TOTAL EXPENSES	\$251,735	\$234,827	\$78,750	\$253,898	\$236,676	\$78,750	\$263,501	\$247,118	\$78,750