

CHEMICAL ENGINEERING RESEARCH PROJECTS 1999

Kenan Center for the Utilization of Carbon Dioxide in Manufacturing

R.G. Carbonell and J. DeSimone
16 corporate sponsors + Kenan Institute
\$560,000 + \$70,000 (Kenan Institute)
July 1, 1997 to June 30, 2000

This is an industry-university research consortium with over 16 partners from industry and the national laboratories. Research is supported by the membership, as well as through federal and industrial grants to the Center. Projects are a collaborative effort with investigators from the physical sciences at UNC-Chapel Hill and engineering from North Carolina State University. There are four thrust areas for the research: catalysis and reaction engineering, cleaning and extraction technologies, chemical processing and coatings, and computational and analytical technologies.

Mechanisms of Cleaning of Organic Films from Solid Substrates

R.G. Carbonell and C. Grant
National Science Foundation
\$90,000/year
April 15, 1997 to March 31, 1999

The mechanisms of cleaning of organic films from solid substrates by aqueous surfactant solutions and by oil-in-water emulsions are being investigated. A rotating disk apparatus is being used to distinguish the relative roles of solid phase diffusion, liquid phase mass transfer and kinetics of dissolution on the cleaning rates of organic films of different thickness and composition. Of particular interest is the role of surfactant structure on determining the rates of cleaning and the optimization of the surfactant concentration and time required to carry out the cleaning process.

Investigations and Development of CO₂ Based Polymer Delivery Technologies

R.G. Carbonell, J. DeSimone, R. Gould, S. Khan, and J. Edwards
ONR
\$190,000/year
December 15, 1997 to December 14, 2000

Our goal is to develop CO₂-based coatings technologies that will allow the replacement of some of the hundreds of millions of pounds of solvents that are manufactured each year for use in coatings applications. CO₂-based delivery systems are possible for binary systems containing either fluoropolymers or silicones dissolved in CO₂ or for ternary systems comprised of CO₂-insoluble polymers, and CO₂-soluble surfactants that help to solubilize the polymer in the CO₂ solvent. The chemical and physical properties of these systems are being investigated and delivery nozzles are being designed.

Delivery of Polymeric Coatings for the Protection of Monumental and Civil Infrastructure from Homogeneous Solutions of CO₂

R.G. Carbonell, J. DeSimone, and H.B. Hopfenberg
Dupont
\$50,000/year
May 1, 1998 to April 30, 2001

Novel polymeric materials are being developed for the prevention of the deterioration of stone monuments and other civil infrastructure. The objective is to make the surface of the material hydrophobic to prevent the penetration of acid rain. These polymers will be delivered from high pressure solution in carbon dioxide, an environmentally-benign solvent.

Kenan-Italo Consortium for the Preservation of Monumental Works of Art

R.G. Carbonell, J. M. DeSimone and F. Piacenti
Kenan Institute for Engineering, Technology and Science
\$50,000/year
April 13, 1994 to April 12, 2001

Polymeric materials used for the protection of monumental works of art must satisfy stringent requirements. This project characterizes the transport properties of natural stones that have been coated with fluoropolymers to prevent liquid water penetration. In particular, the transport of liquid water and water vapor through the stone samples are being evaluated. The relationship between polymer delivery rates to the surface and polymer penetration and pore blockage are being investigated.

Ligands from Combinatorial Libraries for Virus Detection and Removal

R.G. Carbonell and D. Brown
VITEX Technologies, Inc.
\$100,000
8-17-98 to 8-16-99

Solid phase combinatorial peptide libraries will be used to identify ligands that bind specifically to the surface of viral particles. Such ligands can be used to develop novel detection technologies as well as in the separation and isolation of viruses from plasma products. The technique might also lead to novel anti-viral therapeutic agents.

Affinity Purification of Proteins Using Ligands Derived from Peptide Libraries

Ruben G. Carbonell
Sandia National Laboratories
\$145,000
10/1/97 to 12/31/99

This project involves the screening of solid phase combinatorial peptide libraries to identify ligands for the affinity purification of proteins from complex mixtures. The effects of peptide density and the absorption and elution conditions on the yields of proteins are being investigated. Small peptides offer several advantages as ligands for the large-scale purification of and detection of proteins and other biological molecules by affinity chromatography. These include low cost relative to antibodies, easy coupling chemistry, and stability during sterilization and elution steps.

Composite Polymer Electrolytes Using Fumed Silica Fillers: Synthesis, Rheology, and Electrochemistry

S. Khan, P. Fedkiw, and G. Baker

DOE

\$660,000

9/97 to 8/00

This project involves developing novel composite polymer electrolytes possessing high conductivity, electrochemical stability, mechanical stability, and easy processability. These mutually exclusive properties will be arrived at by a new approach using surface-modified fumed silica fillers. By appropriate choice of these surface groups and the polymeric media, the rheological behavior of these systems can be tailored independently of the electrochemical characteristics, a feat that has eluded researchers to date. These advanced electrolytes will be used as new generation materials for secondary lithium batteries.

Novel Electrodeposited Nanocrystalline Metals and Composites

C. Koch, P. Fedkiw, and J. Narayan

NSF

\$540,000

8/98-7/01

The focus of this project is to develop artifact-free nanocrystalline (nc) materials by electrodeposition. A study of the electrochemical parameters that control the grain size and chemistry of the nc films are a central focus of the research. Both Cu and Zn deposits will be prepared. In addition, nano-sized dispersoids will be co-deposited during the deposition, such as "soft" polystyrene and "hard" diamond, as a means to effect the properties of the deposit.

Production and Characterization of Electroalvanized Nanocomposites

C. Koch, P. Fedkiw, and J. Narayan

International Lead Zinc Research Organization

\$144,000

1/99-12/01

The main objectives of the research are: (a) the determination and understanding of electrochemical parameters that control grain size, chemistry, and dispersoid morphology and distribution in zinc-based nanocrystalline coatings; and (b) characterization of the structure/property relationships of such coatings, with particular emphasis on coating adherence, hardness, and appearance, all of which are pertinent to automotive applications.

Electrooxidative Destruction of Organic Wastes Using Supercritical CO₂ as a Reaction Media

P. Fedkiw

Kenan Center for the Utilization of Carbon Dioxide in Manufacturing

\$25,000

7/98-12/00

This project will explore the use of near-critical and supercritical (SC) carbon dioxide as a solvent for the electrochemical destruction of organic wastes. The unique solvating advantages of SC carbon dioxide will be coupled with the tuneability and specificity of electrochemistry to oxidatively destroy organics to carbon dioxide, water, and minerals, with an initial focus on chlorinated hydrocarbons.

Gas Transport and Barrier Properties of Novel Aromatic Polyamides

B.D. Freeman and J. Preston

National Science Foundation

\$306,518

July 1, 1998, to June 30, 2001

Proprietary amorphous polyamides are used commercially in gas separation applications; liquid crystalline (LC) aromatic polyamides are among the most impermeable barrier polymers available. However, wholly aromatic polyamides in general and LC ones in particular are typically difficult to dissolve and have melting points near or above their decomposition temperatures, which makes processing quite difficult. This research program explores synthesis and gas sorption, transport, and permeation properties of novel, highly soluble aromatic polyamides, having either rigid rod backbones or semi-rodlike backbones and structural elements which could be precursors for further reactions such as crosslinking.

Novel Nonporous Fouling-Resistant Composite Nanofiltration Membranes and Membrane Separation Systems for Wastewater Treatment Important for CPS0N5/PPSON1: Minimization of Oily and Non-Oily Waste

B.D. Freeman

Department of Defense SERDP Program

\$1,402,106

June 4, 1998 to June 3, 2001

This research program will be a collaborative project between North Carolina State University [NCSU] and Membrane Technology and Research, Inc. [MTR], Menlo Park, CA. This academic/industrial partnership is directed towards removing fundamental wastewater processing limitations related to membrane fouling by developing advanced high flux, low fouling membrane materials and reducing these advances to practice by preparing spiral wound membrane units suitable for field testing.

The Influence of Backpulsing on Fouling Properties of Novel Nanofiltration Membranes for Wastewater Remediation

B.D. Freeman

Office of Naval Research

\$80,000

November 15, 1997, to October 30, 1999

The use of backpulsing (or reverse filtration) has been shown to markedly enhance average permeate flux (by providing in situ membrane cleaning to remove foulants) in conventional ultrafiltration and microfiltration membranes, separations that are commonly carried out at 10-40 psi transmembrane pressure difference. Thus, in this program, we will construct a novel apparatus to evaluate the effect of backpulsing on fouling resistance of both novel materials being produced and characterized as part of our ongoing ONR program and commercial nanofiltration and ultrafiltration membranes, which will serve as standards. We will perform a variety of backpulsing experiments to explore the efficacy of this technique to enhance average permeate flux.

Development and Testing of New Nanofiltration Membranes for Application to Water Treatment

B.D. Freeman, F. DiGiano, and J.M. DeSimone
American Water Works Association
 \$149,447
 October, 1997, to October, 1999

Minimizing the frequency with which membranes must be rinsed and/or cleaned to remove foulant material is key to the success of membrane technology in water treatment. In this research program, we are synthesizing new heterophase polymers that have high water productivity, excellent selectivity and are more fouling-resistant than conventional drinking water treatment membranes.

Gas and Vapor Transport Properties of Amorphous, Glassy Fluoropolymers

B.D. Freeman and V.T. Stannett
Civilian Research and Defense Fund
 \$20,000
 December, 1996-January, 1999

The objective of this research program is to organize and execute a cross-disciplinary, cooperative research with Professor Yuri Yampolskii and his colleagues at the Russian Academy of Sciences in Moscow. Professor Yampolskii is among the best membrane scientists in the former Soviet Union and we share a common interest in the transport of small molecules in high permeability fluoropolymers. Therefore, we have embarked on a joint research program to characterize the transport properties solid state structure of these materials using a variety of advanced spectroscopic techniques.

Very Low Surface Energy (<11 dynes cm⁻¹) Heterophase Polymeric Materials for Membrane Separations: An Integrated Polymer Chemistry/Engineering Approach

B.D. Freeman and J.M. DeSimone
Office of Naval Research
 \$1,080,101
 April 1, 1994 to September 31, 1999

The composite experimental research effort includes materials synthesis, ultrafiltration characterization, biological foulant adhesion characterization, and oversight involving membrane module fabrication companies. This research program is among the first to combine a materials synthesis effort with bio-foulant adhesion *and* membrane separation performance characterization. Composite transport data and transmission electron microscopy results on a series of new materials is providing fundamental insight into the effect of morphology on transport properties.

Sorption and Transport of Organic Molecules in Polyethylene terephthalate

B.D. Freeman
 \$177,262
Eastman Chemical Company
 January 1, 1996 to December 31, 1999

The migration of flavor molecules in poly(ethylene terephthalate) (PET) packaging can limit business opportunities by deleteriously affecting food and beverage package performance through flavor adulteration, scalping and, in refill applications, flavor carryover. Fundamental permeant partitioning and diffusion properties are critically important in understanding and controlling flavor adulteration, scalping,

and flavor carryover. However, little information regarding flavor molecule partitioning and transport in PET is available. These composite considerations have stimulated the proposed cooperative research program. The primary objective of the research program is to determine equilibrium partitioning and sorption transport kinetics of model organic penetrant molecules in poly(ethylene terephthalate).

Structure and Properties of Polyelectrolyte Brushes

Jan Genzer
National Research Council, COBASE Project Development Grant (98PD1)
 \$2,200
 Two weeks: May/June 1999

Project description: I plan to establish a new research partnership between Dr. Petr Vlcek's group of anionic polymerization at the Institute of Macromolecular Chemistry, Prague, The Czech Republic, and my research group at the Department of Chemical Engineering at North Carolina State University, Raleigh, NC. The IMC group will synthesize the macromolecules for this study and the NCSU group will carry out the measurements of the polyelectrolyte interfacial properties. The purpose of my travel to Prague is to set up the collaboration with my Czech colleagues, discuss the details of the planned synthesis and characterization experiments and to initiate writing a joined NSF proposal, which would provide necessary funding for this program.

Substrate Pattern Recognition by Copolymer Chains

Jan Genzer
NC State University, Faculty Research & Professional Development Program
 Award \$5,000 + \$5,000 matched by the College of Engineering, NC State University
 1/1/1999 to 12/31/1999

Project description: The purpose of the proposed program is to initiate experimental and modeling studies, which aim at studying the adsorption of heteropolymers with defined microstructures (e.g., copolymers) on substrates with pre-defined chemical heterogeneity. During the course of the research we plan to investigate: i) the role of the copolymer microstructure (block vs. random vs. alternating) and the copolymer molecular weight on adsorption, and ii) the effect of size and spatial distribution of the substrate chemical heterogeneities on the amount of adsorbed copolymer. The experimental part of the project will be complemented with calculations using a 2- and 3-dimensional self-consistent field model and Monte Carlo simulations. These will provide useful physical insight into the various phenomena governing polymer adsorption on chemically heterogeneous substrates.

Fabrication of Nanoparticle Assemblies in Ordered Polymeric Matrices - Faculty Early Career Development (CAREER) Program

Jan Genzer
National Science Foundation, DMR-9875256
 Award \$359,992
 1/15/1999 to 12/31/2003

Project description: In this project I propose to investigate experimentally the formation, organization, and physical properties of nanocomposites made of periodically modulated arrays of nanoparticles embedded in self-organized two-component polymer matrices deposited onto solid substrates. The organization and spatial distribution of the

nanoparticles will be adjusted by tailoring the structural properties of the host polymer material. In particular, by using mixtures of diblock copolymers or by using laterally phase-separated polymer blends, the dimensions of the structural features in the polymer matrix, and thus the distribution of the embedded nanoparticles, will range from several nanometers to several micrometers, respectively. Our initial research efforts will focus on studying the formation and morphology of the mesoscopic structures, investigating the interplay between the polymer/particle interactions, and probing the ordering mechanisms as a function of the host- and guest- properties. Subsequent work will be devoted to determining the optical, electrical, magnetic, and transport properties of the newly generated structures.

POWRE: Interfacial Analysis of the Deposition and Removal of Organic Films from Solid Surfaces

Christine S. Grant

National Science Foundation Professional Opportunities for Women in Research and Education (POWRE) Program

\$65,000

6/98-5/00

The POWRE grant will enable our group to access highly specialized analytical equipment at the University of Minnesota's Center for Interfacial Engineering to study the interfacial properties of films during decontamination and fouling processes. The following systems will be studied: the hydration and swelling of supported phospholipid films in aqueous surfactant solutions and the high temperature degradation and deposition of lubricant films on metal surfaces. The proposed research represents a departure from our earlier work by focusing on a closer evaluation of the changes in the chemical and physical properties of the contaminant film using interfacial characterization techniques.

Development of International Environmental/Chemical Engineering Computer Educational Aids: A Partnership Between NCSU and the University of Science and Technology (UST) in Ghana, West Africa

Christine S. Grant

NCSU Committee on International Programs

\$5000

4/97-6/99

Engineering Information Foundation

\$75,000

1998-1999

United Technologies Faculty Activity Grants (COE)

\$1000

1998-1999

The major goal of this project is the compilation of environmental/ChE computer based problems with an international perspective. The key stages of the project include: (1) acquisition of environmental data by ChE students at UST by either chemical sampling, or from industrial/municipal waste information, (2) utilization of software programs to analyze and model the Ghana environmental data, and (3) development of a textbook of problems and case studies that can be incorporated into the undergraduate chemical engineering curriculum. This project represents the first joint research/educational program between the NCSU's COE and UST.

Mechanisms of Cleaning Organic Films from Solid Substrates

Christine S. Grant and Ruben Carbonell

National Science Foundation

\$180,000

4/97-4/99

This work studies the factors that control the rate of cleaning of organic residues from solid surfaces using non-ionic surfactants. The surfactant structure (i.e., size of the hydrophobic tail and hydrophilic head group), is an important parameter in determining the chemical features of the surfactant which control the cleaning rate. The experiments include a study of the performance of organic cosolvent/surfactant solutions during cleaning. The effect of temperature on cleaning is also investigated, since the temperature can control the state of aggregation of the surfactant. An investigation of the recycling and regeneration of spent surfactant solutions can assist in the development of environmentally benign cleaning processes.

Mechanisms of Cleaning Organic Films from Solid Substrates

Christine S. Grant and Ruben Carbonell

CORPEX Technologies, RTP, North Carolina

\$22,500

Three years

This project represents industrial support for above NSF grant through the NSF Grant Opportunities for Academic Liaison with Industry (GOALI) program. The effect of temperature on cleaning and subsequent regeneration of spent surfactant solutions is also investigated. The experiments include a study of the role of various organic solvent/surfactant solutions in the cleaning process.

Molecular Simulation of Fluid Behavior in Narrow Pores and Pore Networks

K.E. Gubbins

NSF, NRAC

\$110,000/year

4/1/98-3/31/99

The work under this project is aimed at understanding the behavior of simple fluids in porous media, in particular the effects of such confinement on phase transitions, chemical equilibria and transport properties. Current projects being pursued are studies of shifts in the freezing transition in porous carbons, improved molecular models of activated carbons, effect of confinement on diffusion rates in carbons, and the influence of pore size, material and state conditions on chemical reaction equilibria. These large simulations are carried out on supercomputers at several NSF national centers (San Diego, Illinois and University of Texas) under a large NRAC grant.

Adsorption and Diffusion in Well-Characterized Adsorbent Materials

K.E. Gubbins

DOE

\$230,000/year

8/1/98-10/31/2001

A wide-ranging study, using molecular simulation and experimental methods, of adsorption, separation and diffusion of gases and liquids in nanoporous materials is supported under this project. The main emphases are on (a) studies of water and gas hydrates in activated carbons and large pore

aluminosilicate materials, (b) the development of improved and realistic models of porous glasses, sol-gel materials and polymers, and (c) the design of highly selective adsorbent systems for removal of trace components.

CISE Postdoctoral Research Associate: Molecular Simulation of Phase Transitions

K.E. Gubbins

NSF

\$46,174

7/1/97-6/30/2000

This grant supports a postdoctoral research associate to develop advanced molecular simulation methods for the study of phase transitions in realistic models of mesoporous glasses and activated carbons. Both equilibrium and kinetic aspects of capillary condensation (gas-liquid transitions) and liquid-liquid separation are being investigated.

Diffusion and Transport of Fluid Mixtures in Porous Media

K.E. Gubbins

Civilian Research & Development Foundation

\$38,860

7/1/97-6/30/99

This grant supports a collaborative research program between NC State University and a research team of the Ukraine Academy of Sciences on the theory and simulation of diffusion and viscous flow in porous media.

U.S.-Venezuela Cooperative Research Project: Modeling Adsorption and Dynamics of Water and Aqueous Solutions on Activated Carbons

K.E. Gubbins and E. Muller

NSF

\$38,256

3/1/97-2/29/00

This project involves a cooperative research program between the PI at NC State University and Prof. E. Muller at Simon Bolivar University in Caracas, Venezuela. The behavior of water and aqueous solutions adsorbed in activated carbons is under investigation, with particular interest in selective adsorption and the formation of gas hydrates in carbons.

Phase Separation and Molecular Dynamics of Fluids and Solids in Porous Media

K.E. Gubbins and M. Sliwinska-Bartkowiak

U.S.-Poland Maria Sklodowska Curie Foundation

\$38,000

12/1/97-11/30/00

This project is a joint molecular simulation/experimental effort to investigate the freezing and liquid-liquid separation of fluid mixtures adsorbed in well-characterized porous media. The experimental work is carried out by a team of professors at the Institute of Physics, Adam Mickiewicz University, Poznan, Poland. The complementary theoretical and molecular simulation program is carried out at NC State University.

Computer Simulation Studies of the Thermodynamics of Protein Folding and Aggregation

Carol K. Hall

NSF

\$200,500

12/1/97-11/30/00

This research program is aimed at understanding the mechanisms by which solutes prevent protein aggregation. By simulating the properties of model proteins and solutes on the computer, we explore how protein folding and kinetics are influenced by protein type and concentration, denaturant concentration, solute type and concentration, and temperature. Our theoretical work should assist scientists in (1) choosing and/or designing solutes to suppress unwanted aggregation, (2) optimizing the in vitro refolding of recombinant proteins by manipulation of process variables, and (3) providing a future basis for the modeling of medically-important proteins such as β -amyloid, the protein whose aggregation is associated with Alzheimer's disease.

Theoretical Treatment of the Thermophysical Properties of Fluids Containing Chain-like Molecules

Carol K. Hall

DOE

\$300,000

6/1/97-5/31/00

This research program has three main objectives: (1) to further develop and refine the Generalized Flory theory for mixtures, particularly those of practical interest to the polymer industry, e.g. polymer solutions, polymer blends, and copolymer-containing blends, (2) to enhance our understanding of the static and dynamic properties of polymer networks and gels, with special focus on network deformation and gel swelling, and (3) to develop a detailed molecular level understanding of the role played by entanglements in the dynamics of polymer melts.

Theories for Fluid Mixtures Containing Chain-like Molecules

Carol K. Hall

American Chemical Society-Petroleum Research Fund

\$60,000

7/1/98-8/31/00

This research program is aimed at developing an equation of state that is capable of predicting the experimentally observed thermodynamic properties, including phase equilibria, of fluids and fluid mixtures containing chain-like molecules ranging in length from alkanes to polymers. The Generalized Flory Dimer theory is being extended to mixtures of practical interest to the petroleum, chemical and polymer industries, i.e. mixtures with asymmetries in segment size, chain length, and attractions, either between species or along the chain.

Biocatalysis Near and Above 100°C: Physiological, Enzymological and Engineering Studies

Michael W.W. Adams (Univ. of Georgia), John A. Baross

(Univ. of Washington), and Robert M. Kelly (CSU)

National Science Foundation

\$285,000 (RMK)

9/96-8/99

This project examines both basic and applied issues of enzymes from two model hyperthermophiles, the archaeon

Pyrococcus furiosus ($T_{\max} = 105^{\circ}\text{C}$) and the bacterium *Thermotoga maritima* ($T_{\max} = 90^{\circ}\text{C}$). This will be done in light of sequence information on the entire genomes of both organisms which will be available during the course of the project. We will also continue to explore the diversity of hyperthermophiles in both deep sea and continental geothermal sites, and the types of enzymes they contain will be examined for their physiological significance, thermostability/thermoactivity and/or biotechnological potential.

Bioenergetic and Physiological Studies of Hyperthermophilic Archaea

Robert M. Kelly
Department of Energy
 \$285,000
 8/96-7/99

The physiological and bioenergetic characteristics of the hyperthermophilic archaea must be better understood, if this novel group of organisms is to be used to the best scientific and technological advantage. This project addresses these issues from the perspectives of intracellular proteolysis and bioenergetics for members of the order Thermococcales.

Rheological Characterization of Enzymatically-modified Galactomannans

Saad A. Khan and Robert M. Kelly
Department of Agriculture
 \$194,612
 9/96-8/99

In this project, newly discovered hemicellulases from high temperature microorganisms are being used to modify galactomannans to achieve favorable thickening properties. These enzymes are functional and stable at temperatures approaching and exceeding 100°C . As such, the galactomannan solutions that are treated have significantly less viscosity and are more amenable to enzymatic processing. The ability to use enzymes in this way and at these high temperatures has not yet been available but could expand the applications for thickening agents. Fundamental insights into the relationship between biochemical modifications and thickening properties of the resulting galactomannan solution will be sought in this investigation.

Synthetic Biocatalysis by Hyperthermophilic Enzymes

Michael W.W. Adams (Univ. of Georgia) and Robert M. Kelly
Department of Energy BCTR Program
 \$90,000
 10/97-2/99

The work proposed here focuses on strategic opportunities to catalyze synthetic reactions with enzymes from hyperthermophiles, which are organisms that grow near and above 100°C . Using several hyperthermophilic enzymes which have been recently purified in the PIs laboratories, a series of model synthetic reactions will be investigated which make strategic use of the remarkable stability and activity of these enzymes at extreme temperatures. The results will be essential in defining the areas of opportunity for enzymes from these unique microorganisms in the chemical process industries.

Biological and Chemical Energetics of Deep Sea Subsurface Life

Michael W. Adams (Univ. of Georgia), John A. Baross (Univ. of Washington), and Robert M. Kelly
National Science Foundation
 \$225,000 (RMK)
 8/98-7/01

The isolation of hyperthermophiles (microorganisms that grow at 90°C and above) from both deep sea (< 2 km) subsurface fluids immediately following a volcanic eruption and from diffuse flow vent fluids associated with stable hydrothermal vents indicates the existence of a deep sea, subsurface microbial biosphere. The overall objective of this project is to examine the specific nature of the microbial population within deep sea subsurface environments, such that the results will serve as a paradigm for the characterization of extraterrestrial environments and potential life forms therein.

Thermophilic Enzyme Hydrolysis of Water-soluble Polymers: Interrelationship Between Biocatalysis and Rheology

Saad A. Khan, Robert M. Kelly and Robert K. Prud'homme (Princeton Univ.)
National Science Foundation
 \$226,051 (RMK and SAK)
 8/97-8/00

Novel approaches to extend the useful temperature range for enzymes used for hydraulic fracturing of oil and gas wells have been identified that rely upon newly discovered hyperthermophilic enzymes and new concepts in copolymer blends for galactomannan polymers. Preliminary research has resulted in the identification of several hyperthermophilic microorganisms capable of producing hemicellulosic enzymes active in galactomannan hydrolysis. As such, the objectives of this collaborative study are to understand the physical chemistry and rheology of existing and proposed polymeric fracturing solutions during enzymatic hydrolysis at high temperatures.

Biomolecular and Engineering Studies of Extremely Thermophilic Xylose (Glucose) Isomerases

J. Gregory Zeikus (Michigan State Univ.) and Robert M. Kelly
National Science Foundation
 \$244,915 (RMK)
 11/98-10/01

Enzymes from thermophiles and hyperthermophiles represent attractive candidate biocatalysts when applications require high-temperature operations. One such operation is glucose conversion to fructose by xylose (glucose) isomerases (XIs) in the production of high fructose corn syrup. This study, containing both biochemical engineering and biomolecular aspects, focuses on the XIs from the thermophilic and the hyperthermophilic eubacterial genera *Thermoanaerobacterium* and *Thermotoga*.

Rheological Studies on Blends of Dendritic and Linear Polymers

Saad Khan
RTI/EPA
\$110,000
8/98-10/01

Dendrimers and hyperbranched polymers offer tremendous potential for use as coating systems because of their uniqueness in displaying low viscosities even at high molecular weights. The proposed research will focus on obtaining a fundamental understanding of the rheology of dendritic polymers and their blends in terms of the micro-/molecular-structure of the system so as to facilitate development of systems that show low viscosity at high solids level.

Predictive Modeling of Flow Properties in Food Systems Containing Dairy-based Ingredients

Brian Farkas, Christopher Daubert and Saad Khan
DMI/SDFRC
\$175,025
1/97-12/99

This project focuses on investigating the rheological behavior of milk and hydrocolloid mixture under aseptic processing conditions and developing empirical models to predict the viscosity of this system under these conditions. Results from the research could be used by the dairy industry to improve products and manufacturing processes.

Thermophilic Enzyme Hydrolysis of Water-soluble Polymers: Interrelationship Between Biocatalysis and Rheology

Saad A. Khan and Robert M. Kelly
National Science Foundation
\$341,000
9/97-6/00

Crosslinked gels of galactomannan-based natural polymers are used as hydraulic fracturing fluids to enhance productivity from gas and oil wells. Subsequently, chemical or enzymatic hydrolysis of the polymer is necessary to remove the polymer and open the fracture. The objectives of this project is to understand the physical chemistry and rheology of existing and proposed polymeric fracturing gels during enzymatic hydrolysis at high temperatures. A backbone cleaving enzyme, β -mannanase, is used for gel degradation.

Rheological Characterization of Enzymatically-modified Galactomannans

Saad A. Khan and Robert M. Kelly
Department of Agriculture
\$194,612
9/96-8/99

Enzymes will be used to systematically modify the galactose to mannose ratio in guar galactomannans, a natural biopolymer with extensive use as food additives. The goal of this project is to use these enzymatically-modified guar in conjunction with other polysaccharides to form synergistic cogels with unique properties and functionalities for food applications. This project attempts to correlate the rheological and morphological properties of these cogels with the role of the enzyme and the properties of the enzymatically-modified guar.

Composite Polymer Electrolytes using Surface-functionalized Fumed Silica: Synthesis, Rheology & Electrochemistry

Saad A. Khan, Peter S. Fedkiw and Gregory L. Baker
Department of Energy
\$660,359
10/97-9/00

This project involves developing novel composite polymer electrolytes with high conductivity, electrochemical stability, mechanical stability and easy processability. These mutually exclusive properties will be arrived at using a new approach utilizing surface modified fumed silica fillers. By appropriate choice of these surface groups and the polymeric media, the rheological behavior of these systems can be tailored independently of the electrochemical characteristics, a feat that has eluded researchers to date. These advanced electrolytes will be used as new generation materials for secondary lithium polymer batteries.

Characterize and Modify Microstructure in Water-borne Inks

Maury Balik, Richard Spontak, Saad A. Khan
Environmental Protection Agency
\$245,000
10/96-10/99

Polystyrene latexes with chemically or physically grafted polyethylene oxide chains are being synthesized for use as zero VOC water-borne inks and coatings. This multidisciplinary effort combines synthesis, materials characterization and an understanding of the colloidal interactions among the latex particles, that is needed to develop this into a viable technology.

On the Origins of Petroleum-Water Emulsions: The Role of Asphaltene, Resins, and Colloidal Aggregation

Peter Kilpatrick
Shell Oil Company
\$355,000
7/1/97 – 6/30/99

The central role of polar resins and asphaltenes in fossil fuels in stabilizing water-in-oil and oil-in-water emulsions has been elucidated with 19 different crudes. The asphaltenes aggregate through discotic π -bond or electron-transfer interactions to form primary agglomerates which subsequently crosslink at the oil-water interface to stabilize these emulsions. Our laboratory has developed mechanistic understanding of this phenomenon and how it is related to and governed by asphaltene composition and chemistry, resin-to-asphaltene ratio, aromatic solvency, and aromatic hydrocarbon type.

Structural Analysis of Heavy Crudes: Role of Acid-Base Imbalance in Surface Activity and Emulsion Stabilization

Peter Kilpatrick
Exxon Production Research Company
\$25,000
1/1/98 – 12/31/99

Asphaltenes from differing crudes can have dramatically differing effects in their ability to stabilize water-in-oil emulsions. It is believed that the origin of these differences lies in the cohesive energy density of asphaltene-asphaltene in-

teractions. Specifically, π -bonding and H-bonding both contribute to asphaltene aggregation and the complementarity between acidic and basic sites is believed to be essential for film stability. In this study, the detailed chemistry and structure of asphaltene aggregates from four heavy crudes are studied.

Towards a Greening of the Petroleum Industry: Minimizing Emulsion and Foam Formation

Peter Kilpatrick and Richard Spontak
National Science Foundation
\$200,001
7/1/98 – 6/30/01

Asphaltenes play the central role in the stabilization of emulsions and foams, serious production and refining issues for the petroleum industry. The relationships among asphaltene chemistry; aggregation; film strength, rheology, and microstructure; and emulsion stability will be explored. Chemistry will be probed by a variety of spectroscopies and chromatographies, aggregation by small angle neutron scattering, film strength and rheology by microscopy and interfacial stress rheometry, and emulsion strength by critical voltage. This is the first molecular chemical study of asphaltene film-forming mechanisms.

Mechanisms of Foam Formation in Petroleum-Gas Mixtures

Peter Kilpatrick
Nalco-Exxon Energy Chemicals
\$35,000
2/1/99 – 12/31/99

One of the major challenges associated with deep-sea production of petroleum is the creation of large foam heads due to expansion and decompression of dissolved gases in the crude (methane, ethane, etc.). Due to the limited space offshore, foam must be rapidly broken. In this mechanistic study, we apply our knowledge of emulsion stabilization to study the basic causes of foam stability: asphaltenes, waxes, and asphaltene aggregation.

Selected Energy Epitaxial Deposition (SEED) & Low-Energy Electron Microscopy (LEEM) of AlN, GaN and SiC Thin Films

R. F. Davis and H. H. Lamb
ONR
\$700,000
11/1/97-10/31/99

Wide bandgap (WBG) semiconductors (e.g., SiC and III-V nitrides) have a variety of realized and potential commercial applications in optoelectronics and in high-power, high-frequency microelectronic devices. The quality of heteroepitaxial GaN thin films remains a significant hindrance to further commercial development. The objectives of this research are to improve heteroepitaxial growth of III-V nitrides and SiC by performing (a) epitaxial growth using supersonic molecular beams, (b) in-situ real-time characterization of growth processes using LEEM and other techniques, (c) Monte Carlo simulations of nucleation and growth processes.

Equipment for Selected Energy Epitaxial Deposition (SEED) and Low Energy Electron Microscopy (LEEM) of Group III-Nitride Thin Films

H. H. Lamb
ONR
Amount of Grant: \$160,000
3/2/98-3/1/99

Instrumentation for research and research-related education in selected energy epitaxial deposition (SEED) and in-situ, real-time low-energy electron microscopy (LEEM) of Group III nitride (III-N) thin films.

Demonstration and Development of a Novel Biphasic Column Reactor

P. K. Lim
NC Department of Environment & Natural Resources
\$10,000
2/1/98-5/31/99

The objective of the project is to demonstrate the feasibility and advantages of a novel biphasic column reactor that permits simultaneous reaction, product removal and catalyst recycle, using the industrially-important conversion of nitrobenzene to p-aminophenol as a test case. A bench-scale, thermostated column reactor is built for the demonstration study. A nitrobenzene suspension of fine platinum catalyst supported on activated carbon is sprayed onto the reactor wall at the top of the column reactor by means of a pump and a distributor. The liquid film is allowed to fall through a hydrogen-containing vapor phase, where nitrobenzene is reduced successively to nitrosobenzene and N-phenylhydroxylamine, and then through an aqueous sulfuric acid solution, where N-phenylhydroxylamine is removed and converted to p-aminophenol before it is reduced to aniline, an undesired side product. The unreacted nitrobenzene and the suspended catalyst are collected at the reactor bottom and recirculated. The experimental study is supported by a model study.

SUCCEED Student Transitions Focus Team

David F. Ollis
National Science Foundation/Univ. Florida
\$49,000/yr
9/97-8/99

Chair 8 campus team to identify best practices and hold workshops on transitions “in” and “out” of university, including (i) summer bridge programs, (ii) women and minority peer mentoring, (iii) multidisciplinary capstone design, and (iv) practice experiences (co-op, international)

E123 Scale-up

David F. Ollis, Sarah Rajala
National Science Foundation/Univ. Florida
\$20,000/yr(DFO portion)
9/97-8/99

Prepare “take-apart” lab to handle much/all of entering first year engineering class

Photocatalytic Air Purification

David F. Ollis
National Science Foundation
\$15,000 (3 yr travel grant)
5/98-4/00

Travel allowance for research collaboration with Dr. Pierre Pichat, Director, French lab studying photocatalysis.

Industrial Ecosystem Development Project, Phase I

J. Kincaid, M. Overcash, P. Berke, M. Powell
U.S. EPA
\$192,184
May, 1997 - April 1999

Joint Project with Triangle J Council of Governments, UNC, and Duke. Project will demonstrate the value of specific tools and techniques in achieving local partnerships between businesses that will reduce the amount and cost of waste and pollution within a six-county region of North Carolina.

Investigation of Pollution Prevention Alternative Technologies in the Garment and Textile Care Industries

Perry Grady, Gary Mock, Robert McCall, M. Overcash
U.S. EPA
\$450,000
May, 1998 - April, 2001

Concern over perchloroethylene has led to development and testing of alternative technologies for cleaning clothes labeled "dry clean only". The dry cleaning community has asked for help in the identification of potentially promising technologies and information on making decisions. This work is to provide guidance for the evaluation of the cleaning systems, to identify a feasible technology, and provide the required support to make it successful.

High-K Dielectrics by Remote PECVD

Gregory N. Parsons
SRC/SEMATECH Center for Front End Processing
\$73,000
April 1, 1998 to March 31, 1999

High dielectric-constant (high-k) insulating layers are critical for advanced sub 100nm CMOS IC devices. In collaboration with researchers in Physics, Materials Science, and Electrical Engineering, our group is developing plasma deposition approaches for ultra-thin high-k metal oxide dielectric layers (including Al_2O_3 and Y_2O_3). Auger Electron Spectroscopy is used to characterize the thermodynamic and kinetic stability of these layers in contact with silicon, and materials are characterized using IV and CV analysis. The goal is to achieve $< 1\text{A}/\text{cm}^2$ when the equivalent oxide thickness is less than 1 nm, and demonstrate good n- and p-channel transconductance in CMOS devices.

Plasma Chemical Vapor Deposition of Amorphous Silicon Thin Films Near Room Temperature Using Inert Ion Enhanced Processes

Gregory N. Parsons
Agency: NSF CAREER Award
\$210,000
Supplement \$25,000 March 1998
June 1996-May 2000

Novel approaches for film synthesis have been identified that result in dense, non-porous materials near room tem-

perature. Preliminary results indicate that an ion-enhanced hydrogen desorption process in plasma activated chemical vapor deposition, which has previously not been documented, may be critical for achieving high film density. The objective of this study include the desorption and analysis of these plasma activated deposition processes. The results will lead to new low temperature capabilities in silicon thin film materials, and devices, and extend our understanding of low temperature surface chemistry in plasma activated materials synthesis and plasma/surface interactions.

Plasma Sensing and Analysis

Gregory N. Parsons
NSF Engineering Research Center for Advanced Electronic Materials Processing
\$60,000 /year
August 1992 - September 30, 1999

This project is aimed at demonstrating mass spectroscopic techniques for real-time sensing of gas phase chemical species during plasma processing, and the description of physical and chemical processes occurring in thin film deposition. Our goal is to demonstrate a process sensing and simulation tool that is capable of detecting and characterizing process fluctuations and faults, as well as, supplying information from each run (i.e. film thickness) that could be carried forward in the process stream. Real time integrated sensing and modeling can minimize the impact of statistical process fluctuations to improve product yield and throughput.

Membrane-Based Biological Destruction of Toxic Organics

S.W. Peretti
EPA
\$153,450
5-1-98 to 5-1-99

Microporous coated and uncoated hollow fibers are used to support extraction of VOCs from a gas into a liquid and to support a biofilm capable of completely degrading the VOCs. Small pilot-scale units will be constructed and operated at the EPA paint facility in RTP and at Tyndall AFB. This work will establish the potential for this technology to be applied in a full-scale demonstration at other USAF aircraft painting sites. Currently, we are evaluating polyalkylsulfone-coated membranes for VOC separation, and the performance of the biomembrane reactor under multi-species, multi-substrate conditions.

Promoted Zinc Chromite Catalysts for Higher Alcohol Synthesis in a Slurry Reactor

George W. Roberts
Department of Energy
\$199,811
10/1/97-9/30/00

Higher alcohols, specifically 2-methyl-1-alcohols, are being synthesized from hydrogen and carbon monoxide in a slurry reactor. A novel family of slurry liquids, consisting of fused-ring cyclic compounds, is being used. These compounds permit the reactor to be operated at temperatures of approximately 375°C, and they also influence the activity and selectivity of the catalyst. Zinc chromite catalysts promoted with potassium and cesium are the focus of the research.

In-Situ Generation of Hazardous Reactants for Chemical Synthesis

George W. Roberts
National Science Foundation/Lucent Technologies
Foundation
\$99,910
10/1/97-9/30/99

The dehydrogenation of methanol to formaldehyde is being studied in a slurry reactor using copper-based catalysts. The emphasis of the research is on reaction selectivity, the stability of the slurry liquid, and the deactivation of the catalyst. Kinetic models are being constructed to describe the rate and selectivity of the reaction over the various catalysts. In the next phase of the research, methanol dehydrogenation will be coupled with a formaldehyde-consuming reaction in a single reactor.

Continuous Polymerization in Supercritical Carbon Dioxide

Joseph M. DeSimone/George W. Roberts
Kenan Center for the Utilization of Carbon Dioxide in
Manufacturing
ca. \$125,000/yr.
ca. 10/1/97-9/30/00

The continuous dispersion polymerizations of acrylic acid and vinyl fluoride are being carried out in a stirred-tank reactor (CSTR) using supercritical carbon dioxide as the reaction medium. The decomposition kinetics and initiator efficiencies of various free-radical initiators are also being studied in supercritical carbon dioxide. Models will be developed to describe the effect of chemical and process variables on polymerization rates and polymer properties.

Direct Synthesis of Acetic Acid via Carboxylation of Methane

George W. Roberts
Environmental Protection Agency
\$369,623
1/1/99-12/31/02

The objective of this project is to synthesize lower aliphatic acids (e.g., acetic acid) by the direct reaction of carbon dioxide with a paraffin (methane in the case of acetic acid). The research will include a study of potential catalysts, as well as the effect of process conditions on reaction rates and selectivities. Diffuse reflectance infrared spectroscopy (DRIFTS) will be used to understand reactant/product/catalyst surface interactions.

Morphological and Property Studies of Dibenzylidene Sorbitol Gels

R.J. Spontak
Milliken Chemicals
\$80,000
1/98-12/00

Addition of sugar acetals such as dibenzylidene sorbitol (DBS) to low-molar-mass organic solvents or high-molar-mass polymers often results in the formation of a recognition-driven fibrillar network. These "tectonic" additives afford the possibility of physical gelation, thixotropy and viscosity control. We are currently examining the morphological and rheological characteristics of several model systems, as well as using molecular mechanics and dynamics simulations, to investigate the mechanism and kinetics of DBS-induced gelation and, ultimately, network dynamics and structural reorganization upon deformation.

Modification of Polymer Blend Miscibility through the Use of Supercritical Carbon Dioxide

R.J. Spontak and S.A. Khan
Kenan Center for the Utilization of Carbon Dioxide in
Manufacturing
\$90,000
1/98-12/00

Supercritical carbon dioxide is currently used to alter the viscosity and thermal properties of a polymer melt by plasticizing the polymer and increasing its free volume. This project aims to exploit this neutral solvation effect by controllably altering the miscibility of polymer blends, which in turn could lead to processing windows of homogeneous systems previously unavailable. Thus far, we have demonstrated that supercritical carbon dioxide lowers the cloud point of an upper critical solution temperature (UCST) polymer blend at moderate pressures. This reduction is countered at high pressures due to the effect of hydrostatic pressure on compressible polymers.

Towards a Greening of the Petroleum Industry: Minimizing Emulsion and Foam Formation

P.K. Kilpatrick and R.J. Spontak
National Science Foundation
\$200,000
7/98 - 6/01

Asphaltenes play the central role in the stabilization of emulsions and foams, serious production and refining issues for the petroleum industry. The relationships among asphaltene chemistry; aggregation; film strength, rheology, and microstructure; and emulsion stability will be explored. Chemistry will be probed by a variety of spectroscopies and chromatographies, aggregation by small angle neutron scattering, film strength and rheology by microscopy and interfacial stress rheometry, and emulsion strength by critical voltage. This is the first molecular chemical study of asphaltene film-forming mechanisms.

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