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**GRADUATE TRAINEE FELLOWSHIPS FOR ENERGY RESEARCH
IN MISSISSIPPI**

**A PROJECT SPONSORED BY
U.S. DEPARTMENT OF ENERGY
EXPERIMENTAL PROGRAM TO
STIMULATE COMPETITIVE RESEARCH**

AND

MISSISSIPPI RESEARCH CONSORTIUM

FINAL TECHNICAL REPORT

SEPTEMBER, 1995

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I. EXECUTIVE SUMMARY

The Mississippi DOE EPSCoR Project has been a program of education and research which has (1) increased the number of trained professionals in the energy sciences and technology, particularly those from groups traditionally under-represented in the field and (2) interfaced with existing networks of universities, industry, federal, and state institutions involved in energy-related activities.

Key project activities that have been accomplished are shown below:

- Statewide conference on energy related research held
- HBCU pipeline issue addressed
- Research preceptors/problems selected
- Trainees screened and accepted
- Research initiated
- Practicums organized

Mississippi's three comprehensive universities - - The University of Mississippi, Mississippi State University, and the University of Southern Mississippi - - and the urban institution, Jackson State University have provided a foundation for the expansion of energy-related education and research in the state. The activities of the Mississippi DOE EPSCoR program have been executed under a management strategy developed to establish statewide coordination of research and education programs, i.e., the Mississippi Research Consortium. The Consortium has served as the steering committee for the DOE EPSCoR Program Consortium.

The Project Director of the Mississippi DOE EPSCoR Program has been Dr. David L. Wertz, Professor and Chair of the Chemistry and Biochemistry Department at the University of Southern Mississippi. He has been assisted by three cluster leaders, the steering committee, and campus research preceptors. In addition, an Advisory Committee has been formed to provide guidance from representatives of business and industry and federal/state government regarding direction of future activities and resources for future energy-related research and education programs.

The following research clusters were selected for major emphasis in the Mississippi DOE EPSCoR project because of existing expertise at the MRC Universities and national interest in these topics. At the same time, these three areas have allowed for active research participation by scientists at historically black Jackson State University.

A. The FOSSIL FUEL POLLUTANTS CLUSTER has addressed the environmentally sensitive concerns caused by the combustion of fossil fuels (particularly coal and coal products) and has developed methods to minimize the environmentally unacceptable species resulting from use of coal. This cluster has been directed by Dr. Wertz.

B. The ENHANCED OIL RECOVERY CLUSTER has had as its goal the availability of a far higher percentage of our older petroleum deposits. This cluster has been directed by Dr. Robert Woolsey of the University of Mississippi.

C. The POWER SYSTEMS AND CONSERVATION CLUSTER has been directed towards improved efficiency in energy transport and usage. Dr. David Miller (and later Dr. Roger King from Mississippi State University have directed this project.

A schematic of the project and its management plan is presented in Figure One.

From the Fossil Fuel Pollutants Cluster and from the Power Systems Cluster, several multi-investigator projects (which emphasize unusual expertise in the State) have been selected as the principal activities for Mississippi's current and future DOE EPSCoR activities.

FIGURE ONE. PROJECT SCHEMATIC

II. PROJECT GOALS, OBJECTIVES AND STRATEGIES

A. GOALS AND OBJECTIVES

The goal of the DOE EPSCoR Graduate Traineeship project has been to provide the appropriate research and educational mechanisms to attract quality scientists and engineers who will contribute to advances in energy research and energy issues. Specifically, the objectives of the project have been:

- (1) to establish an energy research and education program that compliments the needs of this state and the nation;
- (2) to create a critical mass of scientists and engineers by incorporating inter-institutional links built around a commonality of research expertise;
- (3) to strengthen the state's scientific community by working with other established programs; and by developing the human resource pool to become involved in energy-related research and education;
- (4) to establish technology transfer to appropriate user groups by developing stronger research links with other energy-oriented research communities.

All of the objectives of this project have been met, with varying degrees of success. Several of these successes are discussed below.

B. STRATEGY

Existing faculty, with expertise in energy research, have served as the research preceptors for the Mississippi DOE EPSCoR Traineeship Program. Their facilities have been used to conduct the various research projects discussed herein.

To attract the best students by offering competitive stipends of quality

educational/research facilities has been the principal strategy employed by the DOE EPSCoR Traineeship preceptors. National advertising by the preceptors has been employed as the principal strategy for bringing the DOE EPSCoR Traineeships to the attention of prospective trainees. Excellent stipends helped make the traineeships attractive to such students. It is clear that the caliber of the trainees is consistent with scientific accomplishments and professional recognitions which will continue to produce new science in energy-related fields.

C. MEETING THE GOALS AND OBJECTIVES

Twenty-six trainees, along with their research preceptors, have constituted the resource pool. These trainees would not have been available without the DOE EPSCoR funds to support their research activities. They represent an increase of 40% in energy related research personnel at the graduate level in the State. Since all of the trainees have been enrolled in graduate courses and seminars at their MRC universities, curriculum emphasis and particularly seminar topics have been reoriented towards energy-related topics. The latter trend is continuing and accelerating in several of the participating departments as the DOE EPSCoR project matures and as other DOE initiatives occur.

Several of the research preceptors have submitted proposals to DOE to obtain additional support for their energy-related projects. Publications and presentations from the Traineeship Program have begun (as noted in Appendix II).

III. PROJECT MANAGEMENT PLAN

Personnel in the management structure of the program include the members of the DOE EPSCoR Committee, the Project Director, and the Cluster Leader. The Mississippi DOE EPSCoR Committee designed a management plan to support the administrative requirements as well as the scientific research being conducted by the individual research groups involved in the clusters. This plan (see Figure 1) has been based on the organization of project personnel, the mechanisms for program direction and development, and the methods employed for oversight. Cluster research activities have been grouped around themes of current research, strengths within the state, and high national priority. Within each cluster were several research projects with specific goals and objectives and with a specific trainee (selected by that preceptor) who was involved with the appropriate graduate degree program as well as with an energy related research project.

The MRC Steering Committee has been responsible for formulating, implementing and monitoring appropriate strategies and mechanisms to ensure that the chief goal of the DOE EPSCoR Program has been met. The MRC Steering Committee has managed, coordinated, and established policy for this program.

A. PROJECT DIRECTOR. The Project Director for the Mississippi DOE EPSCoR Graduate Traineeship Project (Dr. David L. Wertz) has represented the committee in all communications with DOE. The Project Director has maintained an effective interface with the Cluster Leaders to ensure rapid, effective communication. Wertz has been responsible for overseeing the three clusters in the program. As Project Director, he has provided regular briefings to DOE. As Project Director, he has maintained overall

budget control. He has devoted approximately 15% of his time to managing the Mississippi Graduate Trainee DOE EPSCoR Project.

B. CLUSTER LEADERS. The Cluster Leaders have assisted the Project Director in organizing and managing the Mississippi Graduate Traineeship DOE EPSCoR Project. The Cluster Leaders have conducted the routine administrative functions of each of their respective programs. The Cluster Leaders have been responsible for collecting and maintaining the data concerning relevant scientific activities of individual scientists and engineers participating in the clusters, for organization of meetings, for development of appropriate materials necessary for the management of the program, and for insuring that inter-institutional links have been modified. Each made a presentation at the on-site visit by DOE staff and consultants (January, 1992) to Jackson, MS.

C. EXTERNAL ADVISORY BOARD. To provide guidance to the MRC and the cluster leaders, an external board has been formed. Asked to serve on the Board are:

1. Larry Hartness
Vice President for Engineering
ERGON, Inc.
Jackson, MS
2. Mr. William K. Newman
Vice President for Power Generation
Mississippi Power Company
Gulfport, MS
3. Mr. Joseph Sims
Mid Continent Oil and Gas Association
Jackson, MS
4. Mr. Wes Miller
Department of Economic and Community Development
State of Mississippi Energy Division
Jackson, MS

5. Dr. Mildred B. Perry
U. S. DOE
Pittsburgh Energy Technology Center
Pittsburgh, PA
6. Dr. Riitta Ollendorff
Amoco Research
Naperville, IL

D. FISCAL CONTROL. The University of Southern Mississippi has served as Project Fiscal Agent for the Mississippi DOE EPSCoR Project on behalf of the DOE EPSCoR Committee.

E. PROJECT REPORTING. The members of the DOE EPSCoR committee have reviewed brief progress reports submitted by the cluster leaders to the Project Director at the quarterly meeting of the committee. The project director prepared and submitted annual progress reports for use by the committee and the DOE Program Officer. This reporting process has ensured that data on which to base evaluation of the overall progress of the Mississippi DOE EPSCoR Project and of the various clusters has been readily available. The project director has also accumulated and submitted a final project report.

F. MRC UNIVERSITY SUPPORT. Each of the universities has provided tuition and fee support for each of the trainees and released time support for most of the preceptors. USM has also provided additional research support and extensive travel support to the project director. Each of the universities has also provided travel for the preceptors to attend the statewide DOE EPSCoR meetings.

G. PARTICIPATING DEPARTMENTS AND PROGRAMS

1. Jackson State University (JSU)
Biology, Chemistry and Computer Science

2. Mississippi State University (MSU)
Electrical and Computer Engineering, Mechanical and Nuclear Engineering, Petroleum Engineering and Diagnostic Instrumentation
3. University of Mississippi (UM)
Biological Sciences, Chemical Engineering, Geological Engineering and Mechanical Engineering
4. University of Southern Mississippi (USM)
Chemistry and Biochemistry, Polymer Science and Coal Products Research

IV. NARRATIVE SUMMARY OF PROGRESS

A. PROJECT DIRECTOR TRAVEL FOR THE EPSCoR TRAINEESHIP PROGRAM

(1) In November, 1991, Dr. Wertz attended the National EPSCoR Meeting in Portland, Maine to represent the Mississippi DOE EPSCoR traineeship program and to better familiarize himself with the national goals and objectives of the various EPSCoR activities.

(2) In late November, Wertz visited each of the four MRC campuses to discuss the one year grant activities with each preceptor and to facilitate the various start-up procedures. During these visits, Wertz toured several of the laboratories of the faculty preceptors.

(3) In January, Wertz and the cluster leaders from the DOE EPSCoR Planning Grant participated in a two-day site visit with DOE personnel and a DOE review team to critique and strengthen the Mississippi DOE EPSCoR Planning Grant.

(4) In April, 1992, the three cluster leaders and others who were likely to be involved in future DOE EPSCoR activities visited each of the four MRC campuses. During each visit, two principal activities occurred; (a) presentations from the cluster leaders concerning future activities will be made, and (b) current and potential preceptors provided laboratory tours to illustrate their facilities and capabilities for energy research.

(5) Wertz and other MRC personnel attended the May 5, meeting titled "DOE Research Program Priorities and Initiatives" held in Washington, DC.

(6) Wertz met nine times during the Fall and Winter, 1992, and Spring, 1993, with the MRC Committee that developed the Mississippi DOE EPSCoR proposal. A continuation of these meetings occurred in September, 1993.

(7) During 1994, Wertz met extensively (and often) with other scientists to develop, refine and submit DOE EPSCoR projects.

(8) In November, 1995, Wertz attended a DOE EPSCoR briefing in Washington, DC.

B. THE PIPELINE ISSUE. An excellent opportunity for the Mississippi DOE EPSCoR Project is to attract students from the historically black colleges and universities in this area into energy research related graduate programs at the MRC Universities. To accomplish this, at least fifteen seminars have been given by the various project preceptors to the HBCU's, not only in Mississippi but in several neighboring states as well. During each seminar, the DOE EPSCoR Graduate Traineeship Program and its excellent support for graduate energy researchers have been well advertised. Attempts to select female graduate students into the DOE EPSCoR Program have also been successful.

V. RECRUITING METHODS UTILIZED TO ATTRACT POTENTIAL TRAINEES

A. SEMINARS AND CAMPUS VISITS. Every predominantly minority college and university in Mississippi and most in the surrounding states have been visited by an EPSCoR preceptor in attempts to recruit minorities into the program. The DOE EPSCoR Traineeship Program has been discussed with faculty and potential graduate students during these visits.

B. DISCUSSIONS WITH PREVIOUS RESEARCH COLLABORATORS. Several of the preceptors have had long established relationships with faculty at the Bachelors granting colleges and universities around the country. Informal one-on-one discussions about the DOE EPSCoR traineeships have occurred, with good results!

C. ADVERTISEMENTS IN PROFESSIONAL JOURNALS. Advertisements in the appropriate national journals were submitted in the first annual report. As trainees were replaced, such advertisements were submitted, on an intermittent basis, to the targeted journals.

VI. INFORMATION ABOUT THE TRAINEES CURRENTLY INVOLVED IN THE PROJECT

The credentials of more than sixty candidates were considered for the traineeships of the Mississippi DOE EPSCoR Project. Sixteen fellows were selected originally. Several others have been added. These are listed in Table I.

At the conclusion of the project, thirty four trainees had been supported by this project. A brief academic profile of the trainees is provided in Table One. Nineteen

TABLE I. TRAINEES AND PRECEPTORS

<u>Trainee</u>	<u>Preceptor</u>	<u>Department and University</u>	<u>Degree</u>
Charles Smithhart	Wertz	Chemistry & Biochemistry, USM	Ph.D.
Patricia McCormick	Cannon	Chemistry & Biochemistry, USM	Ph.D. ^a
Mark Logan	McCormick	Polymer Science, USM	M.S.
Steven Davis	Mauritz	Polymer Science, USM	Ph.D. ^a
Scott Bailey	Ward	Biological Sciences, UM	M.S.
Kevin Daye	Ward	Biological Sciences, UM	M.S. ^a
Kendall Harris	Roux	Mechanical Engineering, UM	M.S., Ph.D. ^a
Robert Tavlin	Chen	Chemical Engineering, UM	M.S.
Eddie Gossett	Chen	Chemical Engineering, UM	
Brent Johnson	Davis	Ctr. for Spatial Data Res., JSU	M.S.
Roger Carpenter			
Eric Beasley			
Meschelle Thomas	Lindes	Dial Lab., Dept. of Chem., MSU	
John Lowther	Jung	Computer Science, JSU	M.S.
Mildred Leonard	Jung	Computer Science, JSU	M.S.
Eric Peters	Jung	Computer Science, JSU	M.S. ^a
Juanita McDonald	Hussein	Chemistry, JSU	
Burnell St. Cyr	Hussein	Chemistry, JSU	
Kenneth Collins	Hussein	Chemistry, JSU	
Christopher Walker	Hussein	Chemistry, JSU	

William Grantham	King	Electrical Engineering, MSU	M.S.
Daniel Brooks	King	Electrical Engineering, MSU	M.S.
Minh-Luan Ngo	King	Electrical Engineering, MSU	M.S.
Carl S. James	Hodge	Mechanical Engineering, MSU	M.S., Ph.D. ^a
Meschelle Jacobs	Cook	Diagnostic Inst. Lab., MSU	M.S.
Gary Reid	Sawyer	Petroleum Engineering, MSU	Ph.D.
Jane Sessions	Van Besien	Geological Engineering, UM	
Robert Stewart	Aughenbaugh	Geological Engineering, UM	

^aDegree requirements to be completed within the next six months.

percent of the trainees have been women, and 38% have been black, evidencing a strong interest in energy research and the Mississippi DOE EPSCoR activity from underrepresented segments of the scientific higher education population. These high percentages also indicate the success with which the Mississippi DOE EPSCoR Traineeship Program is reaching these underrepresented populations. Brief progress reports from the trainees are provided in Appendix I.

VII. FAVORABLE DEVELOPMENTS DUE TO THE TRAINEESHIP PROJECT

A. VISITING THE CAMPUSES OF THE PARTICIPATING FACULTY PRECEPTORS. The Project Director, in November, 1992, visited each of the participating MRC campuses to discuss the objectives and the specific goals of this project with each of the faculty preceptors. Many of the individual preceptors used the visit to review their facilities and their research projects with the project director and to discuss specific problems related to the initiation of each traineeship project.

B. SYMBIOSIS IN FOSSIL FUEL BENEFICIATIONS. During each campus visit, the Fossil Fuel participants met together to develop a symbiotic major theme project while utilizing the characterization expertise of some and the fuel beneficiation expertise of others. Emerging from these discussions was a nationally competitive project involving at least six experts -- Cannon, Hussein, Ward, Chen, Cook and Wertz -- all of whom were in Mississippi and none of whom were professionally acquainted with their colleagues at the other MRC campuses. Segments of this group have submitted joint proposals and attached

joint research projects since the DOE EPSCoR facilitated their involvement(s) with one another.

C. TRAINEE PRACTICUMS. Practicums were taken at National Wetlands Research Center, Kentucky Center for Applied Energy Research, Mississippi Power Company, Argonne National Laboratory, and Exxon Energy Research Laboratory, Cyanobacteria Workshop, Advanced Technology Systems, Inc. and the National Institutes of Health. These are listed in Table II.

D. SOME FORTUNATE OCCURRENCES. (1) One preceptor has been able to make a "double use" of the trainee stipend. In this situation, a terminal year doctoral candidate was supported at a time when it is critical that he have maximum laboratory time (which the traineeship provides). The preceptor then, however, used the traineeship to attract an excellent new student to his university.

(2) Quotes from Preceptors. (a) "Without the DOE EPSCoR funds, it would have been impossible to attract a student of ****'s caliber to our program." (b) "The project is off and running with a very positive start -- the organisms now in hand show great potential. Already publications and presentations to internationally important audiences have begun. Shown in Appendix II are those which have been accepted to date (including some galley proofs, etc.)" (c) "Our group has become competitive in terms of consideration for research funding and national prestige. Our publication rate has doubled because of this program."

Several graduations have occurred. Masters thesis and doctoral dissertations as included in Appendix III.

TABLE II. PRACTICUMS

<u>Trainee</u>	<u>Preceptor</u>	<u>Location</u>
Charles Smithhart	Wertz	DOE and Kentucky Center for Applied Energy Research DOE and Pittsburgh Energy Technology Center
Patricia McCormick	Cannon	National Cyanobacteria Laboratory
Mark Logan	McCormick	Exxon Energy Research Center
Steven A. Davis	Mauritz	ABB Systems
Scott Bailey	Ward	National Institute for Petroleum and Energy Research
Kevin Daye	Ward	
Kendall Harris	Roux	Oak Ridge National Laboratory
Robert Tavlin	Chen	
Eddie Gossett	Chen	
Jane Sessions	Van Besien	
Robert Stewart	Aughenbaugh	
Meschelle Thomas	Lindner	Argonne National Laboratory
John Lowther	Jung	
Mildred Leonard	Jung	
Eric Peters	Jung	
Brent Johnson	Davis	National Wetlands Research Center American Congress of Surveying & Mapping
Juanita McDonald	Hussein	
Burnell St. Cyr	Hussein	

Kenneth Collins

Hussein

Oak Ridge National Laboratory

William Grantham

King

Mississippi Power Company

Daniel Brooks

King

M. -L. Ngo

King

Carl James

Hodge

Heat Craft, Inc.

Gary Reid

Sawyer

VIII. CURRENT PROBLEMS AND/OR UNUSUAL DEVELOPMENTS

Timing was a problem for some of the preceptors during the first year of the project. Availability of stipend support on October 1, precluded project involvement of trainees on a full-time basis during the Fall, 1991, semester because the potential trainees were committed to other activities. Several of the preceptors (see below) were fortunate enough to be able to recruit a trainee for the start of the Spring, 1992, semester. The problem mentioned above has no effect during the second and third years of the project and was the justification for the fourth year no-cost project extension.

During the initial period of the project, two of the faculty preceptors -- Dr. Cass Parker and Dr. William Lupton -- resigned their positions and left this project. Dr. Wedad Hussein replaced Parker and Dr. G. S. Jung replaced Lupton. Dr. Hussein, in the Fossil Fuel Pollutants cluster, has introduced the technique of bio-remediation of coal into the overall project theme and has already selected a trainee. Dr. Jung has also selected a trainee and developed a project which compliments other activities in the area of computer pattern recognition applied to energy research.

Dr. David Miller, a preceptor (jointly with Dr. King) and the cluster leader for the Power Systems and Conservation cluster, has retired. Dr. King has assumed his role as cluster leader.

Dr. Nolan Augenbaugh replaced Dr. Alphonse Van Besien, who retired unexpectedly due to illness.

While trends in Federal funding cannot be predicted, it is unfortunate that the DOE Practicum requirement for these trainees cannot now be followed up by post-doctoral

appointments and employment at the DOE facilities. One serious problem deals with insufficient funds for travel and housing for the trainee during the practicum experience.

The Mississippi DOE EPSCoR award was split into sixteen traineeships, so there has been minimal implementation funding for most of the projects. However, because each of the trainees has entered a functioning research environment, and the minimal start-up funds (ca. \$4,000 per trainee per year) has not presented an insurmountable problem, that the DOE EPSCoR project did not have to provide tuition allowed the research funds to be spent for research.

APPENDIX I. PROJECT SUMMARIES.

- A. Fossil Fuel Pollutants
- B. Enhanced Oil Recovery
- C. Power Systems and Conservation

QUANTIFICATION OF SULFUR IN COALS BY WAVELENGTH DISPERSIVE X-RAY SPECTROSCOPY

Trainee: (Dr.) Charles B. Smithhart; 1-1-90 to 12-30-94

Preceptor: Professor D. L. Wertz, Dept. of Chemistry & Biochemistry
University of Southern Mississippi

Much has been accomplished towards the project's goal of developing a quantitative elemental analysis technique for measuring sulfur in coals using wavelength-dispersive x-ray fluorescence spectroscopy. The principal achievement was the development of a novel transmission-emission experimental procedure which compensates for matrix-absorption effects by using thin films of coal dispersed in a polymer. This allows the non-destructive, simultaneous measurement of both the x-ray transmittance and the x-ray emission properties of the coal and requires only one standard for instrument calibration. Both are significant advances for the x-ray fluorescence measurements of environmentally important elements such as sulfur.

A schematic of the sampleholder is shown in figure 1.

The research results demonstrate the laboratory application of an analytical technique that could be modified to provide on-line sulfur analysis and therefore contribute to the regulatory compliance required of commercial users of coal.

A side benefit of the project was realized in collaborations with others in the analytical x-ray field (such as equipment and accessory suppliers) resulting in their development of new, higher performance x-ray detector windows and monochromators. These advances were used to participate in the DOE sponsored study of the resids from the Two Stage Liquefaction Project. The technique was also used to study the phosphoric uptake by coals from the KCAER hot phosphoric acid process (see figure 2).

Several publications and presentations to national/international audiences resulted from this study. The publications are included in the appropriate Appendices.

The research effort culminated in the preparation of a doctoral dissertation entitled "Sulfur Determination in Coals by Wavelength-Dispersive X-Ray Fluorescence Spectrometry". This was successfully defended in April, 1995. The author wishes to express his gratitude to the Department of Energy for their financial support.

WAVELENGTH DISPERSIVE X-RAY SPECTRA OF THE IBC SAMPLES TREATED WITH THE KCAER HTT TREATMENT OVER THE RANGE FROM AMBIENT TO 550°C. SULFUR AND PHOSPHORUS PEAKS ARE LABELED.

Diagnostic Instrumentation and Analysis Laboratory

DATE: July 31, 1995

TO: Dave Wertz, USM Chemistry and Biochemistry

FROM: Jeff Lindner, DIAL - MSU

RE: RESPONSE TO MEMO OF 7/24/95

1. Meschelle Jacobs Thomas, Employed from Oct. 1992 - August 1994; Female, White; MS - Chemistry, August 1994; Practicum - Two Weeks Spent in Dr. Jack Demirgian's Laboratory at Argonne National Laboratory, Argonne IL. Summer of 1993.
2. SUMMARY OF RESEARCH PROGRESS (NEXT PAGE)
3. PUBLICATIONS/PRESENTATIONS COPIES (FOLLOWING ITEM 2)
4. COPY OF THESIS ENCLOSED

Diagnostic Instrumentation and Analysis Laboratory

2. SUMMARY OF RESEARCH PROGRESS

The work performed by Meschelle Jacobs Thomas concerned extending the use of Fourier Transform Infrared (FTIR) Spectroscopy as an on-line monitor for products of incomplete combustion (PIC's) and Volatile Organic Compounds (VOC's). Such molecules may arise from a number of processes including waste remediation and direct combustion. The work involved the determination of the IR absorption coefficients for compounds such as CO, CH₄, benzene, toluene, xylene and simple chlorinated organics followed by studies on mixtures using the partial least squares chemometric model. An initial goal of the work was to characterize the limitations of the chemometric method and to determine the number of molecules it was capable of quantifying. Such information would establish the information content available from on-line FTIR analysis of process effluents.

A main result from this work was the determination of the absorption coefficients. These values are dependent on temperature, pressure, and instrument resolution and, contrary to popular belief, many of these constants have not been confirmed. Results in the literature are fragmentary and in most cases the agreement obtained was within 15% and most likely these differences arise from the different instrument resolutions used in different laboratories. A paper is planned for comparing these results with other workers in the field.

At the 0.58 cm⁻¹ resolution of the interferometer used in this work the rotational structure of many of the smaller molecules was observable and this lead to additional questions regarding the performance of the partial least squares algorithm with mixtures containing small and large molecules. It was found that the chemometric model was capable of quantitative predictions of multicomponent mixtures with broad band overlap and with rotational structure overlap. From measurements on these multicomponent mixtures, the largest of which contained 10 different molecules, it was observed that a correlation did not exist between concentration error and calibration size. False positives were easily identified and concentration prediction was most degraded when one of the mixture components had a considerably larger absorption than the other molecules. The model did not take into account a number of other molecules which may be normally present in the exhaust from a waste treatment or combustion facility such as H₂O, CO₂, NO, NO₂, etc. The identification of over 20 different compounds appears possible based on the methodology developed in this work.

DOE Graduate Traineeship Program

Patricia McCormick (WF), student trainee

January, 1992 - December, 1994

projected date of graduation - 1996

practicum: Molecular Biology of Cyanobacteria Workshops (6/93 & 7/95)

project: *A Study of Recombinantly Modified Organisms for Heavy Metal Detoxification*

RESEARCH UPDATE

8/1/95

The cyanobacterium *Synechococcus* sp. strain PCC 7942, also very commonly known as *Anacystis nidulans* R2, was chosen for development and expression of the construct. It is unicellular, non-heterocystous, and naturally competent for DNA uptake. Natural tolerance levels for various heavy metals was determined for the wild type PCC 7942. This initial assessment involved the metals cadmium, copper, and zinc. The organism was most sensitive to copper and cadmium. Growth was inhibited at concentrations greater than 2 and 5 μ M, respectively, for these metals. Zinc was found to be somewhat less toxic, as growth was maintained at levels up to 25 μ M.

Two plasmids have been used for cloning and transformation studies. The first is a cyanobacterial shuttle vector pUC303. This relatively large plasmid (10.9 kb) is a hybrid vector based on the small natural vector endogenous to this species. It has been constructed with resistance markers for chloramphenicol and streptomycin and has a unique *Eco*RI restriction site within the chloramphenicol marker for insertion of foreign DNA. The second plasmid, pAM1213, is a derivative of pBR322 and has been designed for integration by recombination into the PCC 7942 genome. Lacking a cyanobacterial origin of replication, it is unable to replicate in the cyanobacterial host. The plasmid contains a random segment of the *Synechococcus* chromosome which allows homologous recombination between the transforming plasmid DNA and the recipient cyanobacterial chromosome. A spectinomycin-resistance cassette to confer selection for transformants is located within and flanked by portions of the random segment. Also located within this area is the *E. coli conII* promoter, a unique *Sma*I site behind this promoter, and portions of the *lac* operon positioned to allow for IPTG induction.

The large *cup I* locus (1.95 kb) for the yeast copper metallothionein possesses two transcriptional units, the smaller of which contains the actual coding region (less than 200 bp) for the metal binding protein. While yeast may require multiple copies of this sequence for increased metal tolerance, the gene dosage effect has not been demonstrated in a cyanobacterium. A 309 bp fragment encompassing this metallothionein coding region was excised by digestion with *Rsa*I from the cloned sequence of the gene. This fragment was inserted into the unique *Eco*RI site of the pUC303 vector, interrupting the chloramphenicol marker. Similarly, the fragment was inserted into the *Sma*I site of the pAM1213 vector. Molecular hybridization (using the 309 bp fragment as probe) confirmed the presence of inserts for both vectors.

PCC 7942 was transformed with the pUC303 construct, selected initially for streptomycin resistance, and subsequently examined against controls for increased metal tolerance. Since the coding sequence was cloned into the vector without concomitant insertion of a promoter, any expression of the metallothionein protein would probably be controlled by an endogenous promoter already present on the vector. With the foreign DNA inserted into the coding region for chloramphenicol acetyl transferase (CAT), expression via transcriptional read-through from the CAT promoter or creation of a fusion protein are possibilities. It is known that the CUP1 protein undergoes post-translational modification in yeast; however, absence of this process in the prokaryotic cyanobacterium would not be expected to greatly alter the metal-binding capability of this fairly simple protein. Chelating activity depends primarily on metal

coordination to the multiple cysteine residues, and even the unmodified protein is expected to be capable of metal sequestration.

Transformation of PCC 7942 with the pAM1213 construct has also been successfully accomplished. The cells were selected for spectinomycin resistance, transferred to a medium supplemented with IPTG, and examined against controls for increased metal tolerance. Recombination was confirmed by molecular hybridization of cyanobacterial genomic DNA using the 309 bp fragment of the *cup I* gene as probe.

Initial screening of the pUC303 cyanobacterial transformants was encouraging. Earliest results indicated a substantial increase in tolerance to copper relative to controls. Resistance to 10 μ M copper and possibly greater concentrations was observed with several transformants. However, with repeated culturing, these levels were unable to be maintained at a constant level of tolerance. Whether these results might be due to loss of the plasmid, recombination, or other events was unclear.

It was decided to concentrate efforts on the other plasmid construct which contains a promoter and provides for inducible expression. The *con II* synthetic promoter has been shown to function well as a heterologous promoter in a cyanobacterial system. Recombinants having increased tolerance to copper have been identified. Tests are currently underway to assess levels of protein expression from this construct and to determine physiological effects on the cyanobacterial cell. The construct is also being modified to include a cyanobacterial ribosomal binding site upstream of the inserted metallothionein coding region as an aid to transcription. Primers were devised to facilitate this addition and flanking restriction sites were added to the design for ease of ligation. Additionally, attempts are being made to obtain a special strain of PCC 7942, mutant in its ability to express the endogenous metallothionein. Use of this cyanobacterium should eliminate any problems which may arise distinguishing expression levels of the recombinant protein from the endogenous.

FINAL REPORT - May 12, 1995

Nitrous Oxide Formation and Destruction in Fluidized Coal Combustion

Professor Wei-Yin Chen

Department of Chemical Engineering, Anderson Hall, University of Mississippi,
University, MS 38677

phone: (601)232-5651, fax: (601)232-7023, e-mail: cmchen@vm.cc.olemiss.edu

1. Trainees:

- a. Robert F. Tavlin, Jr.
January, 1992 - May, 1993
Male
White

Progress in Research: This trainee addressed the experimental observations and theoretical analysis of the random phenomenon during primary coal fragmentation. Parametric examinations were made of the dynamic and final-state size distributions of coal particles in a basket suspended in a 3-inch fluidized bed with sand. To explore the relative importance of processing variables in analyzing the particle multiplication at the end of primary fragmentation, the experiments were conducted according to a two-level factorial design. The dynamic data were analyzed by means of two probability-based formalisms; the master equation and maximum entropy. In addition, a N_2O was selected and ordered during this course of this study.

MS, Chemical Engineering, University of Mississippi, degree awarded in May, 1993.
Thesis Title: Observations and Experimentations on Pre-Combustion Fragmentation of Coal A Fluidized Bed Combustor

- b. Eddie Gossett
August, 1993 - June, 1994
Male
White

Progress in Research: This trainee's study centered around the "pattern" of coal particle size distribution after primary fragmentation. Specifically, the objective of the investigation was aimed at the fractal dimension of coal fragmentation. Since particle over a wide range of size is was to be collected, various designs of sample basket was tested. Previous size distributions, however, can not be reproduced. During this period, it was discovered that the N_2O analyzer was set at $4.5 \mu m$, instead of the desired $7.8 \mu m$. The analyzer was sent back to Horiba for repair.

The trainee decided to pursue other research topics after June, 1994; and he is currently pursuing MS in Environmental Engineering.

- c. Te-Chang Lu
August, 1994 -
Male
Asian Pacific (U.S. Permanent Resident)

Progress in Research: The objectives of this trainee's research have been 1. to obtain the fractal dimension of primary coal fragmentation, 2. to learn the trace metal distribution in the various fractions after primary fragmentation, and 3. to investigate the kinetics of N_2O formation and destruction during fluidized bed coal combustion. It was discovered that heating rate is an important variable governing particle size distribution during primary coal fragmentation. To obtain high particle heating rate similar to the fluidized bed combustor and collect particle over a wide size range, a new reactor system was designed, fabricated and tested. Three fractions of coal pyrolysis products have been collected: 1. char particles larger than 0.7 mm, 2. char particles smaller than 0.7 mm, 3. tarry compounds condensed on the reactor wall, and 4. the soot particles on the 0.3 μm filter paper. These samples have been sent out for the measurements of particle size distribution and three trace metal contents: Hg, Se and As. Since trace metals in the fractions 2, 3 and 4 are expected to be carried by the flue gas during combustion, and require control technology for their collections. The N_2O analyzer was repaired and has been tested with the on-line combustor.

Progress towards degree : Master of Science in Chemical Engineering, expected to graduate in August, 1996.

2. Graduations

- a. Robert F. Tavlin, Jr., May, 1993.
- b. Eddie Gossett, unknown.
- c. Te-Chang Lu, August, 1996 (expected).

3. Publications

Chen, W.Y., X.Y. Chen, R.F. Tavlin, Z.P. Zhang, B.C. Shen, and L.T. Fan, "Randomness of Primary Coal Fragmentation," the Proceedings of the 7-th International Conference on Coal Science, Volume 1, pp.355-358, International Energy Agency, 1993 (attached).

4. Presentations

Chen, W.Y., X.Y. Chen, R.F. Tavlin, Z.P. Zhang, B.C. Shen, and L.T. Fan, "Randomness of Primary Coal Fragmentation," presented at the 7-th International Conference on Coal Science, Banff, Alberta, Canada, September 12-17, 1993.

Chen, W.Y., R.F. Tavlin, Z.P. Zhang, B.C. Shen, and L.T. Fan, "Dynamics of Primary Coal Fragmentation during Fluidized Bed Combustion," Presented at the Annual Meeting of the American Institute of Chemical Engineers, St., Louis, Missouri, November 7-12, 1993.

Lu, T.C., L. Ma, and W.Y. Chen, "Flue Gas Desulfurization By Manganese Nodule under Equilibrium Conditions," Presented at the Annual Meeting of the American Institute of Chemical Engineers, San Francisco, California, November 13-18, 1994.

5. Co-op Experience: none

The first trainee, Robert F. Tavlin, graduated in May, 1993 and immediately started working as a process engineer of Rubicon Incorporation at Baton Rouge. He was not able to visit an energy related laboratory during the course of his study. The second trainee, Eddie Gossett, pursued practicum for the summer of 1994, but changed research topics before he can accept an offer.

Our previous study indicated that coal nitrogen had very similar evolution rate as the carbon oxidation rate under combustion conditions. The delayed nitrogen evolution to HCN is generally considered the major cause of N_2O formation in a circulating fluidized bed. Therefore, knowledge concerning the dynamics of particle size distribution in a fluidized bed is significant to pollutant emissions. In addition, coal fragmentation also profoundly influences combustion efficiency. Our first phase of the program was devoted to the experimental investigation and theoretical analysis of coal fragmentation.

This fragmentation research addresses the experimental screening of potentially important variables and the correlation of the dynamic particle size distribution data with a previously established stochastic model. Parametric examinations were made of the dynamic and final-state size distributions of coal particles in a basket suspended in a 3"-fluidized-sand bed. This fluidized bed provided both rapid heating and quenching. It was accomplished by lowering the basket from and raising the basket into a water-cooled, reversed-gas flow section. The parameters investigated included coal type, temperature, initial size, and oxygen content for the final state; residence time was an additional parameter for the dynamics. Two-level factorial design was adopted in analyzing the final-state distributions. It has revealed that the coal type, oxygen content, and initial particle size affect appreciably the particle multiplication; specifically, higher rank coal, higher oxygen content and larger particles favor the particle multiplication. Nevertheless, it has also been observed that lignite structure is fragile after drying; for example, it breaks up easily along the geological bedding planes after drying at room temperature. Factorial design has indicated some interactions take place among the variables. The interactions between the initial particle size and temperature imply the potential significance of temperature gradient in the particle.

The dynamic data have been analyzed by resorting to the master-equation approach based on the stochastic population balance. The particles have been lumped into a limited number of states representing the different sizes (volumes) of particles. Each particle at state i undergoes fragmentation into two particles at states j and k , where $i=j+k$. The governing equations have been derived from the master equation for means, variances, and covariances of the random variables, each representing the number of particles in an individual state in the system. The transition intensities in the governing equations or model have been recovered by fitting the model to the experimental data. A good agreement has been obtained between the model and data.

The maximum entropy formalism has also been applied to predict the size distribution at a particular moment during primary fragmentation. The maximum entropy formalism has its origin in the derivation of the Boltzmann distribution of particles at various energy levels; it has become a highly useful tool for predicting the most likely particle distribution when the knowledge about the process is limited. Considering that only two constraints have been imposed in the resultant equation and the good agreement between the model and experimental data, the maximum entropy formalism appears to be a viable first approximation to the size distribution.

The on-going fragmentation research focuses on three tasks: 1. to obtain the fractal dimension of primary coal fragmentation, 2. to learn the trace metal distribution in the various fractions after primary fragmentation, and 3. to investigate the kinetics of N_2O formation and destruction during fluidized bed coal combustion. It was discovered that heating rate is an important variable governing particle size distribution during primary coal fragmentation. To obtain high particle heating rate similar to the fluidized bed combustor and collect particle over a wide size range, a new reactor system was designed, fabricated and tested. Three fractions of coal pyrolysis products have been collected: 1. char particles larger than 0.7 mm, 2. char particles smaller than 0.7 mm, 3. tarry

compounds condensed on the reactor wall, and 4. the soot particles on the 0.3 μm filter paper. These samples have been sent out for the measurements of particle size distribution and three trace metal contents: Hg, Se and As. Since trace metals in the fractions 2, 3 and 4 are expected to be carried by the flue gas during combustion, and require control technology for their collections. The present literature does not contain any information concerning particle size distribution below 1 μm . Therefore, the information concerning fractal dimension of coal fragmentation will not only shed light about fragmentation pattern, but also yield valuable insights about the emissions of fine particulates.

An on-line N_2O analyzer (Horiba VIA510) has been ordered through the Associates' Fund of the University of Mississippi. This instrument was finally delivered in January 1995, and linked to the reactor for the investigation of kinetics of N_2O formation from various coals and destruction with various catalysts in the next phase of the program.

DOE-EPSCoR Traineeship
Final Report
Wedad R. Hussein
Department of Chemistry
Jackson State University
31 July, 1995

Students:

1. Juanita McDonald: Female, African American
February-March, 1992.
2. Burnell St.Cyr: Male, African American,
September-December, 1993.
3. Kenneth Collins: Male, African American,
June, 1993-May 1994
Graduated from JSU, BS Chemistry, May 1993.
4. Michael Price Male, African American
August-December, 1994
Graduated from JSU, BS Chemistry, May 1994
5. Christopher Walker Male, African American
August 1994 - May, 1995,
Will be back in August 1995
Summer Internship: With Advanced Technology System, Inc.
Monroeville, Pennsylvania

Research Status & Progress:

This summer, we have A.C. in the building !!! but no students !!!.

Michael and Chris are the latest addition to the list of students supported by the grant. Michael has been accepted by the graduate school in UC Davis (Ph.D. in Chemistry), so he's gone. Christopher is still here, he went this summer to work (traineeship) with Advanced Technology Systems. He spent the last two semesters taking courses and doing extensive literature survey. He did not start any experimental work. Hopefully, he will do that when he returns next fall in August.

I am sorry I don't have much to report, as you can see we have problems, what can I say ? not much help to you.

Hope you had a nice summer

Wedad

DOE-EPSCoR Graduate Trainee Project

Final Report

Name: Mark Logan

Gender: Male

Race: Caucasian

Progress to Date: Significant progress has been made toward biosynthesis of *de novo* amphiphilic proteins for water remediation.

Progress Towards Degree: This student has successfully defended his thesis with an anticipated graduation date of August 1995.

Publications:

Logan, M.J., Cannon, G.C., Heinhorst, S, and McCormick, C.L., "pH Responsive Association of *De Novo* Amphipathic α -Helices." *Manuscript in Progress*

Logan, M.J., Cannon, G.C., Heinhorst, S, and McCormick, C.L., "*De Novo* Design and Expression of a Hydrophobically Associating, pH Responsive Polypeptide." Abstracts, 46th ACS Southeast Regional Meeting, Birmingham, AL, October 16-19, 1994

Logan, M.J., Cannon, G.C., and McCormick, C.L., "The Use of Recombinant DNA Technology to Produce Water-Soluble Polymers." Abstracts, Sixth Annual Southeastern Graduate Polymer Conference, University of Southern Mississippi, April 13, 1994

Logan, M.J., Cannon, G.C., and McCormick, C.L., "Design and Synthesis of a *De Novo* Hydrophobically Associating Polypeptide for Phase Transfer Applications." Abstracts, 45th ACS Southeast Regional Meeting, Johnson City, TN, October 17-20, 1993

Logan, M.J., Cannon, G.C., Heinhorst, S., and McCormick, C.L., Design and Synthesis of *De Novo* Hydrophobically Associating Polypeptides." Polymer Preprints, 34(1), 140, (1993)

Presentations:

"Hydrophobically Associating Polypeptides as Polymeric Micelles" Mississippi Academy of Sciences" Biloxi, MS, February 1995

"*De Novo* Design and Expression of a Hydrophobically Associating, pH Responsive Polypeptide." 46th ACS Southeast Regional Meeting, Birmingham, AL, October 16-19, 1994

Poster Sessions: "The Use of Recombinant DNA Technology to Produce Water-Soluble Polymers." Sixth Annual Southeastern Graduate Polymer Conference, University of Southern Mississippi, April 13, 1994

"Design and Synthesis of a *De Novo* Hydrophobically Associating Polypeptide for Phase Transfer Applications." 45th ACS Southeast Regional Meeting, Johnson City, TN, October 17-20, 1993

Logan, M.J., Cannon, G.C., Heinhorst, S., and McCormick, C.L., Design and Synthesis of *De Novo* Hydrophobically Associating Polypeptides." (1993)

Co-op Experience: In November 1991, Mark was given the opportunity to visit the Exxon corporate research facility in Annondale, New Jersey. While at Exxon, Mark was given instruction in the various aspects of using the Polymerase Chain Reaction (PCR) to amplify, mutate and sequence synthetic oligonucleotides. As a result, Mark was able to minimize significantly the time normally associated with learning these techniques expediting his research.

Accomplishments: Molecular cloning techniques have been utilized in the synthesis of a novel biopolymer protein designated as DN3Lx1. The placement of hydrophobic and ionizable functionalities in DN3Lx1 are intended to facilitate the formation of pH responsive hydrophobic interactions.

At this time the DNA encoding for this novel biopolymer/protein has been synthesized, characterized and placed into a specially modified prokaryotic expression vector. Expression of a protein with the anticipated molecular weight of DN3Lx1 was observed shortly after protein induction.

The microstructure of DN3Lx1 has been characterized by SDS-PAGE, laser desorption mass spectroscopy, amino acid analysis, isoelectric focusing and peptide sequencing. The results of these characterizations are in agreement with those anticipated for DN3Lx1.

The solution properties of DN3Lx1 have been examined using Fourier transform infrared spectroscopy (FTIR), circular dichroism (CD), fluorescence probe analysis and gel filtration chromatography (GFC). These analyses indicate that DN3Lx1 adopts an α -helical structure in solution which is unperturbed upon reducing the pH from 6.6 to 1.7. Unlike the structural content the aggregation properties of DN3Lx1 are dependent upon solution pH, with octamer aggregates forming at pH 6.6 and tetramer aggregates forming at pH values less than 3.7. The formation of hydrophobic microdomains as indicated by fluorescence probe measurements, is maximized at pH 3.7 suggesting that tetramer aggregates of DN3Lx1 contain regions of greater nonpolarity relative to the octamer aggregates.

The EPSCOR Traineeship gave me a unique opportunity to engage in research for which I had no other funding resources. The results of the project, thanks to one very capable and dedicated graduate student (Scott Bailey) who served as a Trainee, have been very rewarding. The project provided data to be essential for leading into other promising investigations into novel biological tools for MEOR. Furthermore, the two Trainees that worked on the project now are working at national energy labs (Scott Bailey at NIPER, Kevin Daye at PETC)

1. Name: Scott A. Bailey
 Gender: Male
 Race: Caucasian

Progress in Research: Excellent progress. Three presentations, two at national meetings; manuscript in progress. Now employed at NIPER with Dr. Rebecca Bryant (MEOR) and in Ph.D. program, University of Tulsa.

Progress toward Degree: Completed M.S. thesis and degree - May, 1994.

- Name: Kevin Daye
Gender: Male
Race: Caucasian

Progress in Research: Began during last year of project, unimpressive work; continued research in progress; co-author of abstract. Progress toward Degree: Elected not to work toward degree on the project - completed thesis research for another faculty member on a different topic.

2. Graduations: Scott A. Bailey, M.S. May, 1994.

3. Publications: Abstracts published in meeting programs attached. Manuscripts in progress.

4. Co-op experience: Scott A. Bailey. Summer Internship at NIPER, Bartlesville, OK, with Dr. Rebecca Bryant working on MEOR. Scott continued our EPSCOR project while at NIPER in addition to working on other projects with Dr. Bryant. Scott gained valuable experience using new research tools and approaches to MEOR - indeed he did so well there that he was asked to return to work at NIPER as he now pursues his Ph.D. at the University of Tulsa.

5. The project began with a focus on screening a variety of environmental isolates for indications of possibilities for MEOR applications. Several bacterial cultures gave indications for further investigations. Especially promising were cultures that exhibited relatively rapid and extensive emulsification of crude oil - one of several features that we were exploring for possible MEOR applications. Given the limited budget with stipend only for the Trainee with no allowance for equipment or a laboratory assistant, we elected to focus efforts on evaluations of oil emulsification at the expense of other promising approaches. Emulsification has potential application as an oil-mobilizing procedure for EOR. Scott Bailey then narrowed the focus on only a few microbial cultures, developed assay techniques and analytical methods, and then characterized the chemistry and kinetics of bioemulsification of crude oil. Scott developed methods for gas-chromatographic and gravimetric quantification of crude-oil fractions affected by microbial activity, and established a standardized assay for emulsification. Kevin Daye then took over from Scott and focussed on characterization of crude-oil fractions degraded during emulsification. The research revealed the following: several kinds of naturally occurring microorganisms are capable of emulsifying crude oil. Some of the cultures are effective at salt concentrations up to about 10% and temperatures up to 40 degrees Celsius. A strong emulsifier from one organism can be isolated and semipurified in active state. During growth on crude oil as bacteria use lightweight fractions the oil as carbon and energy source, emulsification greatly enhanced degradation of most fractions. Possibilities for applications to EOR (and for further investigations): 1. living cells growing on the oil itself as they produce emulsifiers. 2. dead cells or extracted emulsifier injected into the well to mobilize residual oil.

Presented at the 94th General Meeting, American Society for Microbiology,
Washington D.C., May 20-26, 1995

Biodegradation of Crude Oil Fractions by Rhodococcus erythropolis
ST-2. K. DAYE, S.A. BAILEY, and H.B. WARD*. Univ. of Mississippi,
University, MS 38677

Applications of oil-emulsifying microorganisms to enhanced oil recovery might require that living cells grow on fractions of the oil as the same cells form oil-in-water emulsions. Moreover, bioremediation of oil spills and similar hydrocarbon pollutants using naturally occurring microbial isolates offers the potential for direct on-site cleanup. Especially promising are biopolymers that act as emulsifying agents to enhance bioavailability of hydrophobic pollutants. We observed in preliminary tests that the superior oil-emulsifying strain ST-2 (ASM Abstract No.* , Las Vegas, NV, 1994) also grew on the oil as the only carbon source. To determine the amounts and kinds of alkanes removed from oil by ST-2, we used time-course assays of replicate shake-flask cultures grown in a mineral medium plus 0.2% crude oil as the only carbon source. Total oil removed was measured gravimetrically on hexane-soluble (HSF) or insoluble fractions; alkane fractions were monitored by gas chromatography. About 35% maximum of total crude oil corresponding to the HSF was consumed by growing cultures. Some lightweight and some heavyweight alkane fractions were rapidly attacked and completely consumed within the first 60 h of culture, other fractions in both weight ranges were slowly consumed and persisted until after 80 h. Pristane and phytane were only slightly removed until about 75 h, then were rapidly attacked. No detectable fractions remained after 85 h of culture. Emulsification of the oil did not occur until about 54 h of culture, after which the rate of degradation some fractions increased. Strain ST-2 exhibits the potential to metabolize completely all crude-oil alkane fractions and to enhance degradation of some fractions by emulsification of the oil. In enhanced oil recovery, organisms such as ST-2 might mobilize the oil as they grow on some oil fractions. In bioremediation applications, the organism might serve both to enhance general bioavailability of hydrophobic substrates and to participate in biodegradation.

GIS-BASED EXPLORATION MODELS FOR MISSISSIPPI

Trainees: Jane Sessions, Robert Stewart

Preceptor: Alphonse Van Besien, Dr. Nolan B. Aughenbaugh

PROJECT

Activities on the DOE EPSCoR Traineeship project were initiated in June, 1992, on the University of Mississippi campus. Emphasis on the development of each trainee's skills with the remote sensing systems, image processing software, and geographic information systems (GIS) has been maintained throughout the duration of the project. Trainees gained experience in literature-based selection of promising exploration sites in Mississippi as well as subsequent acquisition and preparation of remotely sensed data for further processing. Site selection involved the trainee's review of exploration data, lithologic logs, geophysical information, etc. and satellite image preparation consisted of georeferencing and initial image filtering. ATLAS and AGIS software were used by trainees for the processing of georeferenced images of candidate exploration sites. This DOE EPSCoR project successfully provided students with opportunities to better understand GIS applications to petroleum exploration as well as gave students hands-on experience with state-of-the-art remote sensing systems and image processing software.

PROJECT SUMMARY

The trainees were involved in a project with the goal of developing petroleum exploration models using a combination of conventional geologic methods and remote sensing and geographic information systems technologies. Monroe County, Mississippi was selected as a target area for analysis due to known buried faults in the area and the possible presence of several linear features identified earlier using analog remote sensing techniques.

Multispectral scanner (MSS) imagery for the project area was acquired from the EROS Data Center and georeferenced to the Universal Transverse Mercator (UTM) coordinate system using the Summagraphics Table Digitizer. AGIS software was used for image processing to extract surface lineations with a Public Land Survey (PLS) township grid incorporated into the images to facilitate comparison to previous investigations of the study area.

FINAL REPORT
October 1991 - September 1995

Application of Intelligent Concepts to the Control of Power
Systems for Improvement of Power System Stability and
Efficiency

Preceptor: Dr. Roger L. King
Department of Electrical and Computer Engineering
Mississippi State University

Trainee: William C. Grantham, B.S.E.E.

Male, Caucasian

Summer 1992 practicum with Mississippi Power Company, Hattiesburg, MS

Research products: 1 research report, 1 publication, 1 presentation

Completed M.S.E.E., December 1993

Presently employed at Southern Company Services, Birmingham, AL

Mr. Grantham's research focused on two areas -- investigating the sources of error in power system control signals and developing a prototype controller utilizing fuzzy set theory. The electric utility industry in the United States is divided into many interconnected areas for the purpose of improved reliability. Each of these areas is responsible for generating enough power to supply its own customers or "native load." By maintaining the generated power equal to the power consumed by the load, the utilities will maintain system frequency at 60 Hz. Under-generation will cause the system frequency to fall while over generation will cause it to rise. Since the loads are constantly changing, the generation level in each area must also be changing to maintain the scheduled frequency. Since the different areas are interconnected, changes in one area will be "felt" by all the other areas that are connected. Therefore, the power flow on the tie-lines used to connect areas are also essential to the control of the generation within an area.

One of the goals of his research was to design a fuzzy controller that would measure the tie-line power flow of the system and the frequency and determine the correct generation response. The design of this controller was accomplished using the Fuzzle PC Based Logic Shell (Version 1.8). The fuzzy controller was tested using a simplified model of an interconnected power system. The results of the test indicate that it is possible to design a fuzzy controller to regulate generation of a power system. Further research in this area should include a more complex real time dynamic model of the system and the controller. A research report documenting this research was prepared and is included as a part of this final report.

The other goal of his research was to investigate the effect of filtering the control signal received from the power system sensors before using these signals for generation control. This work demonstrated the use of the EASY5 Engineering Analysis System in modeling a power system and its AGC cycle. The research also showed how control effort can be increased by over 200% by high frequency load variations and noise in the ACE signal. These unnecessary control actions result in excess wear on plant equipment and an increase in maintenance costs and/or downtime. The importance of applying properly designed filters to minimize these excess control actions was shown. It was noted that special care needs to be taken when using digital filters to ensure aliasing of higher frequency components into lower frequency components does not occur. Results of this work were published in a paper and it is included in this final report. The paper was presented at the 26th IEEE Southeastern Symposium on System Theory held at Ohio University on March 20-22, 1994.

Trainee: Daniel L. Brooks, B.S.E.E.

Male, Caucasian

No practicum

Research products: 1 thesis, 1 publication, 1 presentation

Completed M.S.E.E., December 1994

Presently employed at Electrotek Concepts, Inc., Knoxville, TN

Mr. Brooks' research focused on the impact of non-utility generators (NUGs) on the stability of the system. At the present time, NUGs present few system-wide control and stability concerns due to the relatively small number of these facilities in operation. However, the steady-state and transient response of a local power system area may be markedly affected by the presence of significant amount of NUG. If present trends continue, non-utility generation is expected to make up ten percent of the total United States capacity in the near future. This increase coupled with current utility operating practices that favor operating closer to stability margins could lead to undesirable operating conditions.

In order to perform stability analyses, computer models must be developed that accurately represent NUG units. The problem is that the classical generation system computer models and assumptions which represent the traditional bulk power generating units may not be accurate for smaller NUG units. Consequently, any extensions to the classical models that might be needed to represent NUG are determined by work done using very detailed models to predict the short-term transient response of systems containing significant amounts of NUG. As a result, the models investigated in this research are valid only for short-term stability analyses and should be applied in other circumstances with care.

The machine models constructed as a part of this research were designed to meet two criteria: (1) provide an accurate electrical response of a synchronous machine along with its controls (2) provide these results faster than the models used in most readily available simulation software packages. The two control systems used in this work are the excitation and speed control systems. The excitation control system of a generating unit is responsible for assisting the machine in maintaining a specified terminal voltage. The speed control system is responsible for adjusting the mechanical power input to the generator as the load being served by the generator changes. In other words, this system helps to maintain system frequency.

The models developed as a part of this research were verified using the industry-standard software package EMTP. The verification of each of the synchronous generating unit components by comparison to EMTP proved that the models and algorithms constructed in this work were accurate. Furthermore, the simulation code implementing the models constructed in this work performed eight times faster than EMTP for the cases studied. Thus, the synchronous generating unit developed for this project accomplished the purpose for which it was intended. Results of this work were published in a paper and it is included in this final report. The paper was presented at the 26th IEEE Southeastern Symposium on System Theory held at Ohio University on March 20-22, 1994. A copy of the thesis documenting this work is also included.

Trainee: Minh-Luan Ngo, B.S.E.E.

Male, Asian (Vietnamese)

No practicum

Research products: 1 publication, 1 presentation

Anticipated completion of M.S.E.E., December 1995

The tie-line frequency bias of an interconnected power system can be obtained through estimation of the natural response of the system. The mismatch between an area's natural frequency response characteristic and the tie-line frequency bias setting used in AGC is a major cause of inadvertent energy interchange, system time deviation, fighting among system control areas, and unnecessary control actions. Therefore a better way for approximating the B coefficient is needed to reduce the mismatch between B and β . Also, with the introduction of Independent Power Producers (IPP) selling power to utilities, existing utilities have to be more concerned with the mismatch between B and β . As more IPPs appear in the generation mix, utilities inadvertently using power from them may find that paying power back to the grid will no longer be an acceptable operational technique. The IPPs may very likely ask the utility to pay back them back with money instead of power, since the IPP does not have any native load.

Using actual frequency data for the month of July, 1994 provided by Southern Company Services for the Southern Electric System (SES), the estimated frequency responses, $10B(f_a - f)$, were calculated. For 1994, SES used a constant B term of 321 MW/0.1Hz. The calculated values are the average frequency responses over a thirty days period. The calculated values of the frequency response term of the ACE equation actually used on the SES is less than 0.5 MW. This is as expected since integral action of the controller should work to zero out the error. It was noted that 95% of the excursions for the month were within 57.6 MW (i.e., two standard deviations). However, if the B setting was 10 percent too high (and NERC prefers utilities to err towards supplying additional frequency response), then the mean would worsen by 10 percent. Also, note that two standard deviations in this case would be 63.3 MW. Conversely, if the B setting was 10 percent less than what is presently used we would see an improvement in the inadvertent energy interchange due to the system frequency contribution and an improvement in the magnitude of the excursions.

A better approximation of the B coefficient is possible, if the D coefficient from the system natural response can be approximated through load modeling. This is possible because D is not constant like the droop characteristic of the generator. Research is presently ongoing to develop an on-line method of estimating a utilities tie-line frequency bias term. Preliminary results of this work were published in a paper and it is included in this final report. The paper was presented at the 27th IEEE Southeastern Symposium on System Theory held at Mississippi State University on March 12-14, 1995.

INFORMATION

1. **NAME:** Kendall T. Harris
GENDER: Male
RACE: African American

PROGRESS IN RESEARCH: Mr. Harris has had very strong performance regarding research. He has had 2 conference papers (presentations), 3 journal articles published, has one conference paper accepted for presentation in 8/95, has one journal article submitted, and one journal article in preparation.

PROGRESS TOWARDS DEGREE: Mr. K. T. Harris has completed his M.S. in Engineering Science-Mechanical Engineering at The University of Mississippi and should complete the Ph.D. degree in 5/96.

2. **GRADUATION:** Mr. K.T. Harris completed his M.S. in Engineering Science-Mechanical Engineering in 8/93; he should complete the Ph.D. by 5/96.
3. **PUBLICATIONS:**

A) JOURNAL ARTICLES

"Transient Conductive, Radiative Heat Transfer Coupled with Moisture Transport in Attic Insulations," R. Gorthala, K.T. Harris, J.A. Roux, T. A. McCarty, Journal of Thermophysics and Heat Transfer, Vol. 8, No. 1, Jan. - March 1994, pp. 125-132.

"Experimental and Computational Tests Involving R-11, R-19, and R-30 Fiberglass Insulations," K.T. Harris, T. A. McCarty, J. A. Roux, Journal of Thermal Insulation and Building Envelopes, Vol. 17, January 1994, pp. 197-218.

"Substrate Barrier Effects for a R-19 Fibrous Insulation Batt," K. T. Harris, T. A. McCarty, and J. A. Roux, accepted (3/95) for publication in the Journal of Thermal Insulation and Building Envelopes. (Reprints available 7/95)

"Substrate Barrier Effects for a R-30 Fibrous Insulation Batt," K. T. Harris, J. A. Roux, and T. A. McCarty, submitted to the International Journal of Heat and Mass Transfer (4/95).

4. **PRESENTATIONS:**

CONFERENCE PROCEEDING

"Total Heat Transfer Due to the Variation in Fiberglass Insulation Thickness in Attics," K. T. Harris, T. A. McCarty, J. A. Roux, and R. Gorthala, ASME 29th National Heat Transfer Conference, August 8-11, 1993, Atlanta, GA., Radiative Heat Transfer-Theory and Applications, ASME HTD - Vol. 244, Ed. A. M. Smith and J. H. Chan, pp. 1-10.

"Experimental and Computational Tests Involving R-11, R-19, and R-30 Fiberglass Insulations," K. T. Harris, T. A. McCarty, and J. A. Roux, AIAA/ASME 6th Joint Thermophysics and Heat Transfer Conference, Colorado Springs, CO. June 20-23, 1994, General Papers in Heat and Mass Transfer, Insulation, and Turbomachinery, ASME HTD - Vol. 271, June 1994, pp. 135-144.

"Substrate Barrier Effects for a R-19 Fibrous Insulation Batt," K. T. Harris, T. A. McCarty, and J. A. Roux, accepted for presentation at the ASME 1995 National Heat Transfer Conference, August 5-9, 1995, Portland, OR. (Reprints available 8/95)

5. **CO-OP EXPERIENCE:** None.

6. **REPORT OF ACCOMPLISHMENTS:**

The DOE-EPSCOR Graduate Traineeship has been of great value in supporting the graduate education of Mr. Kendall T. Harris toward his Ph.D. degree in the Mechanical Engineering Department at The University of Mississippi. Mr. Harris has been able to travel to present 2 conference papers and is scheduled to present one more paper this August (1995). He has had 3 journal articles published, has one journal article submitted, and is in the process of preparing another journal article. The experience of working in a team environment with a balance between experimental data and numerical modeling have been over invaluable experience for him. His research has been extremely fruitful and would have not been possible without the support of the DOE-EPSCOR Graduate Traineeship.

DOE EPSCoR Traineeship Final Report
Department of Mechanical Engineering
Mississippi State University

Name of Trainee: Carl A. James

Time Employed on Project: January 1992 - September 1994

Gender and Race: Male, Caucasian

Graduation: Master of Science, May 1993
PhD expected December 1995

Practicums:

Year 1:	HeatCraft, Inc., Granada, MS (manufacturing and design)
Year 2:	Attended ASME Fluids Engr. Conf. Attended ASME Nat. Heat Trans. Conf. (research and networking)
Year 3:	Site visits with MSU Energy Analysis and Diagnostic Center (applications)

Summary of Research Progress:

The research of Carl A. James has been concerned with heat exchanger technology. He has worked on two aspects of the heat exchanger engineering: (1) development and validation of a model for two-dimensional rib roughness in heat exchanger tubes and (2) the use of uncertainty analysis in the analysis and design of cross-flow heat exchangers.

Carl has been very successfully in both of these aspects. The enclosed papers attest to that success. One of the papers, "A Validated Procedure for the Prediction of Fully-Developed Nusselt Numbers and Friction Factors in Tubes with Two-Dimensional Rib Roughness," *J. Enhanced Heat Transfer*, Vol. 1., No. 2, 1994, was selected by the Southeastern Section of American Society of Engineering Education (ASEE) as the Outstanding Contribution to Research for 1994. This is a very prestigious award as all refereed articles from ASEE-member engineering faculty in the southeast are eligible.

The paper "The Application of Uncertainty Analysis to Cross-Flow Heat Exchanger Performance Predictions" resulted from the PhD research proposal of Carl James and was accepted for presentation at the ASME/JSME conference in Hawaii. All conference papers were refereed, and the location resulted in a low rate of acceptance. The fact that a PhD research proposal was accepted for this conference is testimony to the ability of Mr. James. His research is progressing satisfactorily, and I expect him to complete his degree by December 1995. The DOE EPSCoR Traineeship markedly enhanced the graduate experience of Carl James.

**FINAL DOE-EPSCoR
Graduate Trainee Report**

May 15, 1995

Preceptor: Gwang S. Jung
Department of Computer Science
Jackson State University
Jackson, MS 39217

1. Graduate Trainees Supported

Name: Mr. John Lowther, Jr
Period: January 1, 1992 -- January 31, 1993
Gender: Male
Race: Caucasian

Mr. John Lowther held the position from January 1992 to January 1993. He left the position to join a computer company in Jackson, Mississippi. He completed Master Thesis entitled "Adaptive Image Retrieval by Inductive Learning Algorithm Based on Rough Set Theory Using User Relevance Feedback" in December, 1993.

Name: Miss Mildred O. Leonard
Period: March 1, 1993 -- August 31, 1993
Gender: Female
Race: African American

Miss Mildred O. Leonard had been supported by this project from March 1993 to August 1993. She left the position after the graduation. She completed her Master Project titled "Pattern Matching in a Philological Engine."

Name: Mr. Eric Peters
Period: January, 1994 -- Present
Gender: Male
Race: African American
Progress Towards Degree:

Mr. Eric Peters has been supported by this grant since January 1994. He has been developing an artificial neural network program based on Kohonen's Feature Map Method. In this summer, he plans to complete his master project on applying Kohonen's algorithm to classify image segments for geographic information system.

2. Graduation

Mr. John Lowther: M.S. in Computer Science, December, 1993.

Miss Mildred Leonard: M.S. in Computer Science, August, 1993.

Mr. Eric Peters: M.S. in Computer Science, Expected in Summer of 1995.

3. Publications

N/A

4. Presentations

N/A

5. Co-op Experience

N/A

6. Accomplishments, status

Most of the students aim to obtain programming and software development experience through the master program in computer science at JSU. I therefore tried/emphasized to train them to get *useful working knowledge in programming and software development rather than in research oriented topics*.

Mr. Lowther obtained knowledge in advanced programming and software development under Microsoft windows environment, and got a job offer from a software development company located in Jackson, MS. His experience in window programming through this project quite impressed the senior members of the company's software development team. His work for the master thesis was valuable resource for the preceptor's recent research in "distributed adaptive content-based image retrieval" supported by ARPA.

Miss Mildred Leonard also obtained good working knowledge in LISP programming and X window management. Since her graduation, she has been working with Psychology department at JSU for administering various computing facilities. She is also responsible for programming for the researchers in the department. Her work for the master project was also valuable resource for the preceptor's recent research investigation in "distributed information retrieval and filtering" partly supported by ARPA.

Mr. Eric Peters has programmed for implementing an artificial neural network algorithm based on Kohonen's Feature Map Method. He obtained advanced working knowledge in C and UNIX programming. He completed around 2,000 lines of coding in C under AIX Operating System in IBM RISC6000/970. This program will be helpful for the preceptor's recent research investigation, partly supported by ARPA, for identifying semantic textures

in geographic images delivered by UARS Satellites. I believe that he has acquired good working knowledge in developing non-trivial software in scientific applications.

There have been discontinuities in training students throughout this project. The preceptor had difficulty in finding a minority full-time graduate student with US citizenship for the position about 5 months in 1993. Since JSU is located in metropolitan area, most of the students qualified for the position requirement had full-time/part-time jobs off campus at that time. It was also difficult to send students to the co-op since there were not co-op opportunities for the graduate students. Moreover, students preferred working for the project to joining co-op programs.

DOE-EPSCoR GRADUATE TRAINEESHIP PRECEPTOR FINAL REPORT

Kenneth A. Mauritz
Department of Polymer Science
University of Southern Mississippi

Name of Trainee: Stephen V. Davis

Time on Project: September 1990- December 1994

Gender and Race: Male, Caucasian

Graduation: December 1995, expected

Practicum: The mission of this practicum was to learn details of the operation of desulfurizer equipment installed by ABB Environmental Systems in a coal-fired electrical energy plant in Taiwan (Taiwan Power Co.). Time of residence in Taiwan: Jul 12-20, 1994. Specific objective: learn key facts regarding nature and fate of fossil fuel combustion products that impact on the future use of membrane technology applied toward pollutant and useful product removal from waste gas streams. Upon his return to the U.S., S.V. Davis formally presented information gleaned from his trip, to the Mauritz research group. Group members were informed of salient issues and a discussion of the use of our membranes, in this industrial context, was conducted. The overall benefit gained by the student was an experience-based appreciation of present technology and the possible role and limitations of laboratory science in the improvement of this technology.

Publications:

1. S.V. Davis, with K.A. Mauritz, I.D. Stefanithis, R.W. Scheetz, R.K. Pope, G.L. Wilkes, and H-H. Huang, "Microstructural Evolution of a Silicon Oxide Phase in a Perfluorosulfonic Acid Ionomer by an *in situ* Sol-Gel Reaction," *J. Appl. Polym. Sci.*, **1995**, 55, 181.
2. S.V. Davis, with K.A. Mauritz and R.B. Moore, "Monitoring of pH as a Control in the Initialization of Perfluorosulfonate Ionomer Membranes," *Am. Chem. Soc. Polymer Prepr.*, **35**(1), 419 (1994).
3. S.V. Davis, with K.A. Mauritz, C. Bordayo, and R.B. Moore, "Oriented [Perfluorosulfonate Ionomer]/[Silicon Oxide] Nanocomposite Membranes," *Am. Chem. Soc. PMSE Div. Prepr.*, **70**, 230 (1994)
4. S.V. Davis, with K.A. Mauritz "Gas Permeability of Metal Ion-PFSI-SiO₂ Nanocomposite Membranes," *Am. Chem. Soc. Polymer Preprints*, **34** (1), 608 (1993).
5. S.V. Davis, with K.A. Mauritz, "Macro- and Microstructural Characterization of Silicon Oxide-Filled, Metal Ion Exchanged Perfluorosulfonate Membranes," *Am. Chem. Soc. Polymer Preprints*, **33**(2), 363 (1992).

6. Numerous papers based on the PhD Dissertation of S.V. Davis are in preparation.

Presentations:

1. S.V. Davis, with K.A. Mauritz, "Gas Permeability of Ion-Exchanged and Silicon Oxide-Filled Perfluorosulfonate Membranes," Poster, *Gordon Research Conference on Ion Containing Polymers*, New London, NH, 9-14 Jul 95.
2. S.V. Davis, with K.A. Mauritz, K.A. Bair, and R.B. Moore, "Oriented [Perfluorosulfonate Ionomer]/[Silicon Oxide] Nanocomposite Membranes," *Am. Chem. Soc. 46th Southeast Reg. Meet. (Abstr. #465, p210)*, Oct. 16-19, 1994, Birmingham, AL.
3. S.V. Davis, with T. Lewis and K.A. Mauritz, "TGA of SiO₂/Nafion Nanocomposite Membranes", *3rd National Conference on Diversity in the Scientific and Technological Workforce (NSF)*, Washington, D.C., 29 Sep - 1 Oct, 1994.
4. S.V. Davis, with K.A. Mauritz, Q. Deng, P. Shao, C. Bordayo, and R.B. Moore, "[Perfluorosulfonate Ionomer]/[Silicon Oxide] Nanocomposites via *in situ* Sol-Gel Reactions for Inorganic Alkoxides," *Materials Research Society Spring Meet., 1994*, San Francisco, *MRS Proc. Abstr. V5.2*, p 441, invited,
5. S.V. Davis, with K.A. Mauritz, C. Bordayo, and R.B. Moore, "Oriented [Perfluorosulfonate Ionomer]/[Silicon Oxide] Nanocomposite Membranes," *Am. Chem. Soc. Spring Meet.*, San Diego, 1994, invited.
6. S.V. Davis, with K.A. Mauritz and R.B. Moore, "Monitoring of pH as a Control in the Initialization of Perfluorosulfonate Ionomer Membranes," *Am. Chem. Soc. Spring Meet.*, San Diego, 1994.
7. S.V. Davis, with K.A. Mauritz, C. Bordayo, and R.B. Moore, "Oriented [Perfluorosulfonate Ionomer]/[Silicon Oxide] Nanocomposite Membranes," *Mississippi Acad. Sci. 59th Ann. Meet.*, 17, 18 Feb 94, Abstr. Vol. XXXIX, Issue 1, p 45.
8. S.V. Davis, with T. Lewis and K.A. Mauritz, "Formulation of Oriented Nanocomposites with Silicon Oxide Particles in Clusters of Perfluorosulfonate Membranes. Evaluation of Gas Permeability," *Mississippi Acad. Sci. 59th Ann. Meet.*, 17, 18 Feb 94, Abstr. Vol. XXXIX, Issue 1, p 44.
9. S.V. Davis, with K.A. Mauritz, "Gas Permeability of Nanocomposite Membranes Formed via an *in situ* Sol-Gel Reaction within Metal Ion - Perfluorosulfonate Ionomers," *Southeastern Graduate Polymer Conference*, 21-23 Apr 93, Georgia Inst. Tech., Abstr. Vol. I, Paper No. 19.
10. S.V. Davis, with K.A. Mauritz, "Gas Permeability of Metal Ion - PFSI - SiO₂ Nanocomposite Membranes," *205th Am. Chem. Soc. Nat. Meet.*, Denver, 1993.
11. S.V. Davis, with K.A. Mauritz, "Gas Permeation Studies of Nafion®/SiO₂/Metal Nanocomposite Membranes," *Asilomar Conference: Recent Developments in Ionomers*, May 10-14, 1992, invited.
12. S.V. Davis, with K.A. Mauritz, "Macro- and Microstructural Characterization of Silicon Oxide-Filled, Metal Ion Exchanged Perfluorosulfonate Membranes," *Am. Chem. Soc. Fall Meet.*, Washington, DC, 1992.

Summary of Research Progress (Selected Excerpts)

Gas Permeability Studies of Ion-Exchanged and Silicon Oxide-Filled Perfluorosulfonate Ionomer Membranes

[Nafion® perfluorosulfonate ionomer]/[SiO₂(1-x/4)(OH)_x] nanocomposite membranes were formulated and characterized for microstructure and properties. Our central mission was to investigate gas transport through these membranes and correlate the results with information extracted from structure-property characterizations within the context of permselectivity in industrial separations. These membranes must be tailored in reproducible fashion and part of our efforts dealt with this important issue.

Nafion®, consisting of nanometers-in-size ionic clusters in a semicrystalline perfluorocarbon phase, was employed as a morphological template for the *in situ* sol-gel reaction of tetraethylorthosilicate (TEOS) as shown below.

Our working hypothesis was that the clusters act as favorable regions in which the sol-gel reaction initiates and silicon oxide nanoparticles ultimately reside upon sample drying.

Membrane Formulation. Basically, dry membranes for which $\text{SO}_3^-:\text{H}^+ = 1:1$ were equilibrated in alcohol/water solutions to which TEOS/alcohol solutions were then added. After sorbing TEOS for a set time, the membranes were rinsed and dried. In addition to sorption of TEOS in ionic clusters, silica surface deposits were observed. We successfully excluded these deposits by metal ion exchange prior to the *in situ* sol-gel reaction. We reevaluated the entire membrane initialization and ion exchange procedures that occur prior to nanocomposite formulation. Experimentation lead to the following improved method for membrane initialization and sample reproducibility: Reflux in concentrated HCl → reflux in DI/DS water → reflux in concentrated HNO₃ → reflux in H₂O → reflux in H₂O → reflux in H₂O → reflux in MeOH → repeat steps until pH_{H₂O} > 6.5.

We established that TEOS pre-hydrolysis in external bulk solutions is crucial for effective permeation of this monomer and optimal condensation in pre-swollen/hydrated membranes.

To achieve compositionally-homogeneous hybrid membranes, the condensation step of the sol-gel reaction was slowed in relation to monomer diffusion by adding the inhibitor THF which complexes with Si-OH. Using SEM-EDAX, we demonstrated reduced concentration gradients across the thickness of the membrane.

Membranes were placed in a liquid (H₂O/alcohol, TEOS/alcohol solutions) reaction vessel while they were mechanically oriented by an Instron. After *in situ* sol-gel reactions an oven enclosed the sample for drying-annealing while maintaining tension. These in-

house-constructed attachments were used to produce oriented Nafion® templates for the purpose of formulating anisotropic nanocomposites. These samples showed SAXS and mechanical anisotropy.

Our gas transport studies were initiated with an existing steady state permeation cell console. It was discovered that these multicomponent membranes exhibit dual mode sorption: sorbed gas molecules solubilize in at least two nanoscopic regions, namely the perfluorocarbon matrix and nanoparticle-filled clusters. ^{29}Si solid state NMR spectroscopy probed the degree of molecular connectivity of the silicon oxide phase as it affects gas transport. Microstructural aspects as well as thermal transitions and thermal degradation were explored using DSC and TGA. Mechanical tensile tests revealed ductile-to-brittle transitions with increasing filler content.

A Multi-Temperature, Multi-Pressure permeability cell console (MTMP) was designed and fabricated for rapid, accurate manometric determinations of permeability (P), diffusion (D), and solubility (S) coefficients of polymer membranes. A schematic is shown below.

The increase in pressure in the low pressure compartment, given constant high pressure in the other, is measured vs time. P, D, and S are extracted from this data using the solution of a diffusion equation with these boundary conditions. After data is collected at one pressure, the high pressure is incremented and the membrane is degassed and the entire process repeats until after data is collected at the last specified pressure. The temperature increases by a user-defined interval and the pressure cycle repeats. Below is a drawing of the MTMP permeability console that we have constructed.

Mississippi State UNIVERSITY

Dean of Engineering
P.O. Drawer DE
Mississippi State, MS 39762-5635

MEMORANDUM

TO: David L. Wertz
Department of Chemistry and Biochemistry

FROM: Millie Sellars, Business Manager
College of Engineering

M. Sellars

DATE: May 15, 1995

REFERENCE: Information for Annual Report for DOE Traineeship
DOE DE-FG2-91ER75660

Gary W. Reid completed work for his PhD degree in Petroleum Engineering and was graduated on 13 May 1995. The purposes of his project were to put miscibility equipment in working condition and to take such data as were necessary to investigate the physical possibility of enhanced oil recovery by CO₂ miscible process in reservoirs located within Mississippi. Specifically, the objectives used to define this latter purpose were:

- 1) Conduct such tests as were necessary to determine if existing screening criteria were adequate and accurate to predict successful CO₂ miscible floods.
- 2) Develop and validate a relationship between simple reservoir characteristics and CO₂ miscibility.
- 3) Explore the possible economic viability of incremental oil production due to a CO₂ miscible flood in promising reservoirs in Mississippi.

The equipment used in this study was extremely sophisticated and of the highest quality to be found anywhere in the world. It was, however, necessary to make several additional pieces of equipment in order that the tests be accurately conducted. It took approximately one year of full-time work to get the system to the point of producing the high quality data required in this study. Based on the data produced in this project and analysis of same, it was concluded:

- 1) A correlation has been developed which will allow the accurate prediction of minimum miscibility pressure from the depth of the reservoir.
- 2) It was determined that minimum miscibility pressure is highly dependent on whether there are significant amounts of aromatic and/or paraffinic compounds in the oil.
- 3) Significant amounts of aromatic and/or paraffinic compounds in the oil greatly reduce the minimum miscibility pressure.
- 4) Only those reservoirs at depths less than 10,000 ft are candidates for miscible CO₂ floods, unless the oil contains significant amount of aromatic and/or paraffinic compounds in which case depths up to 15,000 ft are acceptable.
- 5) The temperatures used to obtain the minimum miscibility pressures of the laboratory samples are representative of the reservoir temperature from which the oil samples were obtained.

Additionally, the following pertinent activities are provided:

PRECEPTOR INVOLVEMENT

PRECEPTOR	DEPT	TYPE OF INVOLVEMENT
Sawyer	Petroleum Engineering MSU	1 National Technical Conference 1 Regional Regulatory Conference 3 Technical papers in progress

TRAINEE	PRECEPTOR	TYPE OF INVOLVEMENT
G. Reid	Sawyer	1 International Technical Conference 1 National Technical Conference 1 Regional Regulatory Conference 2 State-wide Technical Meetings Discussions with engineers and technicians at 5 different laboratories Numerous discussions with state officials, university professors, independent oil field operators and major oil company emgineers

DESIGN AND SYNTHESIS OF A *DE NOVO* HYDROPHOBICALLY ASSOCIATING POLYPEPTIDE FOR PHASE TRANSFER APPLICATIONS

Mark J. Logan, Gordon C. Cannon, Sabine Heinhorst, and Charles L. McCormick, Department of Polymer Science, University of Southern Mississippi, Hattiesburg, MS 39406-10076

Utilizing recombinant DNA technology a synthetic gene encoding for a *de novo* polypeptide has been synthesized. The construction of this gene, is such that polypeptides consisting of varying degrees of polymerization may be expressed. Each repeat of the synthetic gene encodes for a 29 amino acid sequence. The amino acid sequence has been designed to incorporate many of the structural attributes associated with naturally occurring lipoproteins. Lipoproteins assist in the solubilization and transport of hydrophobic lipid particles in many organisms. Many lipoproteins utilize rearrangements in secondary structure elements to fulfill their function. We are currently involved in the expression of *de novo* polypeptide designed to mimic the phase transfer properties of naturally occurring lipoproteins. Additionally, this polypeptide has been designed to incorporate pH triggers which should allow for environmentally stimulated phase transfer behavior. Applications for this polypeptide range from bioremediation to controlled drug release. Design considerations, cloning strategy and the current status of this project will be discussed.

APPENDIX II. SCIENTIFIC/TECHNICAL PUBLICATIONS.

conference papers
Reprints and Preprints removed for separate cycling. *at*

STATISTICAL ANALYSIS OF MULTICOMPONENT
INFRARED SPECTRA OF VOLATILE
ORGANIC COMPOUNDS

By

Linda Meschelle Jacobs Thomas

A Thesis
Submitted to the Faculty of
Mississippi State University
in Partial Fulfillment of the Requirements
for the Degree of Master of Science
in the Department of Chemistry

Mississippi State, Mississippi

August 1994

PREDICTION OF FRICTION FACTORS AND HEAT
TRANSFER IN RIB-ROUGHENED PIPES

By

Carl A. James

A Thesis
Submitted to the Faculty of
Mississippi State University
in Partial Fulfillment of the Requirements
for the Degree of Master of Science
in the Department of Mechanical and Nuclear Engineering

Mississippi State, Mississippi

May 1993

University of Southern Mississippi

SULFUR DETERMINATION IN COALS BY WAVELENGTH-
DISPERSIVE X-RAY FLUORESCENCE SPECTROMETRY

by

Charles Byron Smithhart

Abstract of a Dissertation
Submitted to the Graduate School
of the University of Southern Mississippi
in Partial Fulfillment of the Requirements
for the Degree of Doctor of Philosophy

May 1995

INVESTIGATION OF MISCIBLE CONDITIONS AND OIL RECOVERY
USING CARBON DIOXIDE AND OIL FROM RESERVOIRS
WITHIN THE STATE OF MISSISSIPPI

By

Gary William Reid

A Dissertation
Submitted to the Faculty of
Mississippi State University
in Partial Fulfillment of the Requirements
for the Degree of Doctor of Philosophy
in Petroleum Engineering
in the College of Engineering

Mississippi State, Mississippi
May 1995

DETAILED SYNCHRONOUS GENERATOR MODELS SUITABLE
FOR ELECTRIC POWER SYSTEM ANALYSES AND
COMPUTER-GENERATED SIMULATIONS

By

Daniel Lee Brooks

A Thesis
Submitted to the Faculty of
Mississippi State University
in Partial Fulfillment of the Requirements
for the Degree of Master of Science
in the Department of Electrical and Computer Engineering

Mississippi State, Mississippi

December 1994