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**Technical Support to the
Solvent Refined Coal (SRC)
Demonstration Projects: Assessment
of Current Research and
Development**

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Chemical Technology Division

TECHNICAL SUPPORT TO THE SOLVENT REFINED COAL (SRC) DEMONSTRATION
PROJECTS: ASSESSMENT OF CURRENT RESEARCH AND DEVELOPMENT

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Prepared for the
Department of Energy/Oak Ridge Operations
Solvent Refined Coal Demonstration Plant Projects

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TECHNICAL SUPPORT TO THE SOLVENT REFINED COAL (SRC) DEMONSTRATION
PROJECTS: ASSESSMENT OF CURRENT RESEARCH AND DEVELOPMENT

M. S. Edwards
B. R. Rodgers

ABSTRACT

A program to demonstrate Solvent Refined Coal (SRC) technology has been initiated by the U.S. Department of Energy (DOE) in partnership with two industrial groups. Project management responsibility has been assigned to the Oak Ridge Operations Office (ORO) of DOE. ORO requested that the Oak Ridge National Laboratory assess current research and development (R&D) activities and develop recommendations for those activities that might contribute to successful completion of the SRC demonstration plant projects. The objectives of this final report are to discuss in detail the problem areas in SRC, to discuss the current and planned R&D investigations relevant to the problems identified, and to suggest appropriate R&D activities in support of designs for the SRC demonstration plants. Four types of R&D activities are suggested: continuation of present and planned activities; coordination of activities and results, present and proposed; extension/redirection of activities not involving major equipment purchase or modifications; and new activities. Important examples of the first type of activity include continuation of fired heater, slurry rheology, and slurry mixing studies at Ft. Lewis. Among the second type of activity, coordination of data acquisition and interpretation is recommended in the areas of heat transfer, vapor/liquid equilibria, and physical properties. Principal examples of recommendations for extension/redirection include screening studies at laboratory scale on the use of carbonaceous precoat (e.g., anthracite) in filtration, and 15- to 30-day continuous tests of the Texaco gasifier at the Texaco Montebello facility (using SRC residues). An example of the final type of activity is the recommendation for a new dissolver exit heat exchanger at Ft. Lewis in support of SRC-I Demonstration Plant design.

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1. INTRODUCTION

A program to demonstrate Solvent Refined Coal (SRC) technology has been initiated by the U.S. Department of Energy (DOE) in partnership with two industrial groups. The SRC-I process, which produces clean,

solid boiler fuel in a one-stage reaction, will be demonstrated by a group presently represented by Southern Company Services, Inc., under the auspices of the International Coal Refinery Company (consisting of Air Products and Chemicals, Inc., and Wheelabrator-Frye); the SRC-II process, which produces a distilled liquid boiler fuel, will be demonstrated under the leadership of Gulf Oil Corporation. Project management responsibility is assigned to the Oak Ridge Operations Office (ORO) of DOE.

The SRC-I and SRC-II processes are emerging technologies that are the focus of a number of current research and development (R&D) activities — including a pilot plant at Ft. Lewis, Washington, a large process development unit at Wilsonville, Alabama, and numerous smaller scale efforts within industry, at universities, and at government laboratories. In addition, other R&D activities not directly related to SRC technology are providing results that are applicable to the SRC Demonstration Projects. ORO requested that the Oak Ridge National Laboratory (ORNL) assess current R&D activities and develop recommendations consistent with technical requirements and project schedules for those activities that might contribute to successful SRC demonstration plant designs.

During the first phase of its effort, ORNL briefly assessed the R&D needs of the SRC program. The initial results included definition of the relevant problems identified by ORNL, preliminary recommendations in order of importance, and a summary of R&D activities that have potential relevance to solution of the problems identified.

Following the completion of the Phase I R&D assessment in mid-February 1979, efforts were concentrated on a more detailed review of R&D activities in the United States. A draft interim report (completed in June 1979) was distributed widely to the coal liquefaction R&D institutions identified during the assessment. Comments received from these groups have been incorporated in this final report. The objective of this report is to provide, for ORO consideration, recommendations for R&D activities in direct support of SRC demonstration plant designs. The recommendations include scoping estimates of the funding and scheduling needed to carry out the SRC programs.

The industrial groups involved in SRC-I and SRC-II have proposed and are conducting R&D activities to fulfill data needs. If an R&D activity was compatible with ORNL recommendations, discussion of the proposed or existing approach was included and compared with the approach recommended by ORNL. Recommendations were ranked qualitatively by importance assigned by SRC developers and others in the coal liquefaction field; potential operability and/or economic benefit resulting from successful solutions; and extent of direct applicability to the designs. The last criterion was particularly important. The need for process data relevant to design conflicted in some instances with the need for long-term, comprehensive data acquisition and interpretation. Several recommendations were, therefore, more applicable to other existing programs in DOE and elsewhere rather than to the SRC demonstration projects.

This final report is intended to provide specific details of the recommended R&D activities. In particular, the recommendations have been updated according to the Phase 0 demonstration plant designs released in July 1979; the recommendations incorporate design changes occurring in Bridging Tasks if these have been identified.

2. SUMMARY OF RESULTS

2.1 Problem Areas Reviewed

The Oak Ridge National Laboratory (ORNL) Solvent Refined Coal—research and development (SRC — R&D) Assessment Team visited numerous institutions to determine what gaps exist in the data base for SRC demonstration plant designs and the capabilities (and plans) of various institutions to supply the needed data. Most of the institutions visited are listed in Table 2.1. Discussions with the SRC demonstration plant industrial groups were also held, primarily with the SRC Task Force (SRC-I) and Gulf Mineral Resources Company (SRC-II).

An important result of the institutional contacts is a general understanding of the accomplishments, capabilities, and plans of the institutions and their relationships to the needs of the SRC Demonstration Plants Projects (SRCDPP). Specific institutions are discussed in the Appendix; however, the inclusion of specific institutions is not intended to imply that these are the preferred locations for the needed R&D. We contacted as many institutions as possible (within the time constraints upon us) whose programs appeared representative of the kinds of approaches being pursued in the important problem areas. Other institutions not discussed are also involved in activities potentially relevant to the SRCDPP and may possess equal capabilities to obtain data needed for the demonstration plant designs.

The primary emphasis of this assessment focuses on R&D areas in which successful programs have high potential for improving the technical/economic basis for the demonstration plant designs. The areas receiving detailed investigation include the following:

1. Reactors/dissolvers
2. Fired heaters for slurries
3. Solids/liquid separations
4. Vapor/liquid and liquid/liquid separations
5. Gasification of SRC residues
6. Slurry mixing
7. Heat exchangers

Table 2.1. Solvent Refined Coal Research and Development
assessment contacts

Problem area	Institution visited
Fired heater	Heat Research Corporation Kinetics Technology International Corporation
Fired heaters, physical properties, instrumentation	Argonne National Laboratory
Fired heaters, physical property correlations	Institute of Gas Technology
Solids/liquid separations	University of Houston Illinois Institute of Technology Johns-Manville University of Michigan Pittsburgh Energy Technology Center Kerr-McGee
Separations, components	Wilsonville, SRC-I Process Development Unit
Vapor/liquid separations	Rice University University of California at Berkeley Purdue University Oklahoma State University
Thermal properties, vapor/liquid separations	Bartlesville Energy Technology Center
Coal liquefaction kinetics, erosion studies, instrumentation	Lawrence Berkeley Laboratory
Coal liquefaction kinetics	Stanford Research Institute
Asphaltenes characterization	University of Southern California
Coal liquefaction kinetics, mechanisms, enthalpy	Colorado School of Mines
Valves	Morgantown Energy Technology Center Fisher Controls, Inc. Willis Oil Tool Company
Heat exchangers	Heat Transfer Research, Inc.
Instrumentation	Cresap Pilot Plant Sandia Brookhaven National Laboratory
Oxygen compressor	Sulzer Brothers Mannesmann Demag Corporation
General liquefaction	Exxon; Exxon Donor Solvent Process (Baytown and Florham Park)
General liquefaction, gasification R&D	Electric Power Research Institute
SRC technology	Ft. Lewis Pilot Plant
SRC-II technology	Harmarville Process Development Unit

8. Physical properties (including thermal properties and those properties relevant to other areas, such as fired heaters)

9. Fluid-handling components

Slurry pumps

Oxygen compressors

Valves

Slurry flowmeters

The areas are not listed in priority order, but all areas with high or moderately high technical/economic relevance to the demonstration plants are listed. The areas chosen by ORNL for further investigation parallel closely those subsequently indentified by the SRC demonstration plant industrial participants in the Bridging Tasks proposals and Phase 0 documents. Such areas are described in Sects. 3.2 and 3.3 for SRC-I and SRC-II respectively. The initial list of problem areas defined by ORNL is provided in Sect. 3.1.

2.2 Summary of Recommendations

A number of programs are suggested in Sect. 4 to support the SRC DPP, subsequent demonstration plant operations, and coal liquefaction commercialization efforts. Specific recommendations relative to the SRC DPP (given in Sect. 4.1) are limited to activities that might provide information to support current demonstration designs. Funding levels estimated in this summary are only for the initial design support portion of the recommendations. Recommendations extending beyond FY 1980 would also support subsequent plant operations and are indicated, where appropriate, in Sect. 4.1.

The recommendations are listed in descending order of importance based on an assessment of technical and economic significance and potential for timely acquisition of information. Few recommendations involving the installation or modification of experimental equipment can be considered because of the time required for the design effort. The recommendations fall into four classes: continuation of current and planned activities; coordination of current and proposed activities

(including data interpretation and correlation); extension or redirection of activities with existing equipment; and institution of new efforts. Related activities and plans are detailed in Sect. 4.1.

Recommendations within each problem area are numbered sequentially throughout Sect. 4. For example, the first recommendation cited for fired heaters (in Sect. 4.1) is numbered 1; the last recommendation for this problem area (in Sect. 4.3) is numbered 7. The numbers shown in this summary correspond to those of Sect. 4.

Fired Heaters for Slurries

1. Continuation and coordination

The existing programs at the pilot plants, particularly Ft. Lewis, should be continued in support of design efforts. Special attention should be devoted to technical coordination of these various projects, extending to establishment of a central data interpretation/correlation effort in conjunction with the coordination effort discussed under "Physical Properties."

2. Slurry rheology data

Acquisition of slurry rheology data should be continued, especially at slurry temperatures above 480 K (400°F) and at wall shear rates in the range of 10 to 1000 s^{-1} . A moderate expansion of current programs is desirable. An expansion of about \$300,000 is indicated, primarily for existing small-scale projects (excluding Exxon and existing pilot plant programs).

Slurry Mixing

1. Pilot plant mixing studies

The pug mill and vortex mixer projects at Ft. Lewis should be expedited. High-temperature operation of the mix tank should be carried out when process operations permit. Tests of mixing conditions and equipment identified by other projects should be instituted as a cooperative effort. Mixing efforts [Exxon

Donor Solvent (EDS) and H-Coal, for example] at other pilot plants and their supporting laboratories should be coordinated with the Ft. Lewis effort, without increases in Ft. Lewis funding.

Solids/Liquid Separations

Assuming that existing and planned research projects at the major SRC support facilities are funded at anticipated levels, the following project is recommended to provide additional support for the SRC DPP.

1. Carbonaceous precoat project

In an attempt to reduce the high costs of precoating, a program should be initiated to assess the feasibility of using a carbonaceous precoat that would have a recoverable heating value in a gasifier. We support the proposed program at Wilsonville, which entails studying the effects of sized anthracite precoat on filtration rate and equipment performance. Carbonaceous precoat screening tests should also be run simultaneously at bench scale in a laboratory equipped for this purpose. The screening tests could be accomplished in one year at laboratories with in-place filters; the cost would be about \$200,000.

Vapor/Liquid and Liquid/Liquid Separations

1. Coordination project

To ensure successful coordination of data acquisition and correlation, it is proposed that a working group be formed on determination and correlation of vapor/liquid equilibria for coal-derived liquids. Representatives from industry, government laboratories, and universities would form the group. Because data for the SRC demonstration plant is the most critical, it should receive priority in the direction of the research. This effort could be part of the coordination effort discussed under "Physical Properties;" the estimated overall cost is about \$100,000 per year.

Gasification of SRC Residues

1. We recommend single, continuous runs of 15 to 30 days at Texaco's Montebello Research Laboratory to develop realistic information on the refractory resistance of Texaco gasifiers with respect to the high molten slag loadings. The cost of such test runs, which is higher than the expense associated with Type 3 tests performed under the current DOE-Texaco contract, is the subject of ongoing negotiations. The increased duration of the proposed tests will result in increased costs. Further recommendations in this area are not possible without a site visit to the Texaco Montebello facility; U.S. Department of Energy (DOE) approval has been requested for such a visit.

Reactors/Dissolvers

1. Solids in the reaction system (pilot plant scale)

The Wilsonville SRC pilot plant should be used to explore the effect of solids accumulation as a function of superficial velocity within the dissolver for a given particle size distribution of coal. A nuclear level gauge is in operation to measure the solids withdrawal rate. Particle size distributions obtained from particular coals under reaction conditions could be determined at laboratory scale and compared with spot samples withdrawn from the Wilsonville reactor. Auburn University, for example, has the capability for making particle size distribution measurements at the laboratory scale.

The catalytic effect of mineral matter buildup on conversion as a function of solids accumulation in the reactor for a particular coal feed should also be done at Wilsonville because of its in-place solids blowdown system. The effect of low solids accumulations on operability and coking in the reactor should be examined. The overall cost is estimated as \$600,000.

Heat Exchangers

1. Correlation of data

Increased emphasis should be placed on the correlation of data available on the design of heat exchangers for conditions representative of SRC demonstration plant operation. The correlation of data could be part of the coordination effort discussed under "Physical Properties."

Physical Properties

1. Coordination of physical and transport data acquisitions

A great need exists for overall coordination of research on physical property measurements in support of coal conversion activities. A multifunctional research location, such as a national laboratory or an energy technology center, could play a coordinating role in this area. Immediate coordination is essential if data are to be made available in time to aid in the design and operation of the SRC demonstration plants. The cost of coordinating physical property measurements would be approximately \$50,000 per year, excluding the vapor/liquid equilibria coordination project. Coordination of physical property measurements should be considered in conjunction with similar recommendations for overall coordination and data interpretation in the areas of heat transfer (fired heaters and heat exchangers) and vapor/liquid separations, for a total overall cost of about \$100,000 per year.

2. Heats of vaporization, sublimation, fusion, and reaction

Existing data on coal-derived liquids from SRC process streams are inadequate. When measured, the data are correlated by use of relations obtained for petroleum liquids. The inadequacies of petroleum-derived correlations for such applications should be identified and corrected, where possible, based on new data from this project. The institutions currently involved in these measurements (Bartlesville Energy Technology Center and Colorado School of Mines, for example) should play a major role. An increase in funding of \$100,000 to \$200,000 more per year would be needed to develop usable early data.

3. Heat transfer data

Heat transfer/rheology studies should be investigated in small-scale preheaters such as the ones available at Argonne National Laboratory (ANL), ORNL, and the Institute of Gas Technology. The cost of these studies would be about \$200,000 per year.

Heat Exchangers

2. Dissolver exit heat exchanger

A well-instrumented shell-and-tube or double-pipe heat exchanger should be installed on the dissolver outlet stream at the Ft. Lewis pilot plant. The data, if acquired in time, would be relevant to the SRC-I demonstration plant design, in which a heat transfer fluid (such as Dowtherm A) is used to recover heat for use elsewhere in the process. A vertical orientation capable of both upflow and downflow operation appears to be preferable. For a 92.9-m² (1000-ft²) stainless steel unit, the approximate cost of the heat exchanger for the relevant temperature and pressure conditions is \$55,000. The total cost, including installation and operation for several months, is about \$100,000.

Slurry Pumps

1. Pump evaluation

It is recommended that testing of state-of-the-art centrifugal slurry pumps be expedited, particularly at Ft. Lewis. This testing should require about \$100,000 in FY 1980.

Fired Heaters for Slurries

3. Flow regime verification

Verification of two-phase flow-regime maps using more recent and more fundamental two-phase flow-regime correlations is needed — especially with respect to tube inside diameter, flow angle (from the horizontal), and physical properties.

Verification would determine whether surface tension is needed for such correlations. The verification of flow-regime maps for fired-heaters and slurry-phase heat exchangers is roughly estimated to require about \$200,000 per year and would be most effectively conducted by an expert in the field.

Solids/Liquid Separations

2. Drying techniques project

The proposed program at Wilsonville to test an 0.46- to 0.65-m² (5- to 7-ft²) skid-mounted candle or leaf filter, using either steam or hot N₂ as a stripping agent to dry the filter cake, should utilize both steam and hot N₂ in a candle filter. Both alternatives to vacuum drying thus can be assessed simultaneously with the assessment of the technical feasibility of a new type of filter. Although screen blinding may not be affected by the drying method, the data will still be useful in determining the optimum technical and economic drying technique.

Valves

1. Valve evaluation

Communication should be maintained among personnel working on DOE-funded projects to ensure that the latest equipment developments are made known to decision makers as they occur and that promising industrial valve designs are tested under large-scale conditions resembling those of the SRC process. The H-Coal and EDS pilot plant programs provide the next level of assurance beyond current SRC pilot plants; the component sizes in these projects are of adequate scale to clearly indicate trends for the future. Evaluation of state-of-the-art and prototype valves requires the following levels of funding (approximately): \$50,000 in FY 1980 and \$100,000 in FY 1981.

Solids/Liquid Separations

3. Filtration support studies

A laboratory-scale screening project pertaining to materials, screen weaves, and related topics should be instituted in support of the Wilsonville studies. Also, the construction of large leaves should be evaluated because warping of the large filter (U.S. brand) leaves was encountered during tests at Wilsonville. A filter or screen manufacturer with a well-equipped laboratory could perform these tests within a year, at a cost of about \$100,000. Drain/fill time and other cyclic operational characteristics that can increase total filtration rates should also be examined at laboratory scale; the cost is estimated at about \$100,000.

Fired Heaters for Slurries

4. Erosion studies

Acquisition of erosion-rate data for coal/oil slurries, as exemplified by A. V. Levy's program at Lawrence Berkeley Laboratory, should be continued. The data should include measurements for longer radius return bends. About \$150,000 per year is required for this work.

Slurry Flowmeters

1. Recommendations for current projects

We strongly recommend continuation of the efforts to develop slurry-metering instrumentation at the Ft. Lewis and Wilsonville pilot plants. Instruments that show promise in the small-scale test stands should be tested at full scale in the pilot plants. Provision should also be made for testing these instruments in large-diameter pipes and reasonable flow rates. Current efforts in the pilot plants have included testing of conventional meters such as orifices and venturis. Additional studies may be initiated for developing and testing the following advanced type of meters in the pilot plants:

- a. Acoustic/ultrasonic
  - b. Capacitive cross-correlation
  - c. Gamma-ray cross-correlation
  - d. Neutron activation
2. Extension and/or redirection of current projects

Current efforts to develop advanced instrumentation, for example, at ANL, should be continued in and beyond FY 1980. However, a significant fraction of the efforts should be redirected toward developing slurry-metering instruments for SRC application. Specific attention should be paid to the following:

- a. High-temperature operation, to 700 K (800°F)
- b. High-pressure operation, to 13.8 MPa (2000 psi)
- c. High solids concentrations, to 50 wt % solids
- d. Effects of increased pipe diameter on the measurement of slurry flow rates

In addition, promising candidate meters developed at ANL and elsewhere should be tested in the pilot plant loops under actual SRC operating conditions on an accelerated schedule to meet start-up requirements for the demonstration plants.

The cost of continuing the instrumentation program for slurry meters is estimated at about \$600,000 per year, and prototype instruments will probably cost about \$200,000 each. Consequently, a projected cost per year would be about \$800,000, assuming that one prototype instrument is purchased each year.

Fired Heaters for Slurries

5. Slurry/gas mixers

Procedures should be established for design of effective slurry/gas mixers. The design of the mixers could affect significantly the initial flow-pattern development in the fired heater. A one-year program should be sufficient for this study; the cost would be about \$150,000.

Heat Exchangers

3. Flow-regime verification

Additional verification of existing multiphase flow-regime maps for vertical and horizontal tube flow is needed. (This was discussed under "Fired Heaters for Slurries," along with the approximate cost.)

Physical Properties

4. Physical and transport properties

Physical property data (viscosity, density, thermal conductivity, specific heat) of coal slurries are inadequate for design and scale-up of SRC units, particularly in the front end of the process. These properties should be measured under process conditions for a range of operating parameters. Institutions other than pilot plants should also be involved in such measurements because the data obtained from pilot plants are not always suitable for developing correlations, primarily because of interaction among system parameters. Thus the effort on physical property measurements on SRC materials in flow systems should immediately be extended and redirected to facilitate demonstration plant design and operation. Although the work in pilot plants could produce some data, the ability to obtain data for developing correlations is limited by the relative inflexibility of pilot plants as compared with institutions conducting bench-scale experiments; nevertheless, pilot plants should play an important role in testing the data and correlations from the bench-scale efforts. A funding level of \$400,000 per year should be sufficient.

2.3 Conclusions

General conclusions reached in the ORNL assessment of R&D activities potentially relevant to SRC Demonstration Plants Projects have been reinforced by subsequent investigation. Because it is outside the scope of this assessment, no mechanism has been devised to ensure that design-

related data needs can be pursued without interfering with other possibly conflicting and equally valid goals. DOE is devising procedures to avoid or control such conflicts. The following general conclusions resulting from the ORNL assessment are, therefore, potentially subject to certain limitations.

Critical problem areas require that major actions be undertaken at the pilot plants (Ft. Lewis, Wilsonville, Texaco Montebello). These problems areas are related to evaluation of the gasification process as a whole, to fired heater operation, and to equipment testing. Although numerous other investigations have been identified as possibly relevant to the problem areas, anticipated results are not directly useful for SRC demonstration plant designs. Feedback and close contact between R&D investigators and process developers, particularly at the pilot plants, will facilitate future application of the results of other investigations.

Coordination of related activities in the areas of physical and thermodynamic properties, heat transfer, materials R&D, and reactor studies will provide additional support for the SRC program.

Several potential problem areas have not yet been sufficiently defined to allow an assessment of their importance to the overall success of the SRC projects. Wastewater control is the principal example. The waste streams expected for the demonstration plants are not fully characterized, and the performance of existing control technologies on such waste streams is inadequately defined at this time. The amount of development required for wastewater control technologies to ensure alleviation of environmental concerns and the eventual cost of such controls should be determined during detailed design efforts. Several potential problems, such as control of polynuclear aromatics and removal of cyanides, are a priori concerns. Here, again, coordination and possible redirection of current activities are advisable to promote applicability to the SRC DPP.

3. IDENTIFICATION OF PROBLEM AREAS

Problem areas identified by the Oak Ridge National Laboratory (ORNL) through discussions with the Solvent Refined Coal (SRC), SRC-I and SRC-II, industrial groups and others involved in coal liquefaction (the Electric Power Research Institute, for example) are given in Sect. 3.1. Many of these problem areas are currently under active investigation at various installations, whereas others are included in current program plans (indicated in Sect. 4 where appropriate).

A specified task in the Phase 0 contracts with the SRC-I¹ and SRC-II² industrial groups requires evaluation of the adequacy of the data base, identification of additional data requirements, and recommendations on how to acquire such data. The additional data needs identified for SRC-I and SRC-II are provided in Sects. 3.2 and 3.3, respectively, in terms of the problem areas referenced. Some interpretation and expansion of these problem areas have been added by ORNL in an effort to compensate for the lack of clarity resulting from removing the problem identifications from their original context. The approaches recommended by the industrial groups are discussed, if applicable, in Sect. 4 in the context of ORNL recommendations.

It should be recognized that differences in extent and type between the problem areas defined by Pittsburgh & Midway Coal Mining Company (P&M) and Air Products and Chemicals, Inc. (APCI) relate at least as much to the engineering philosophies and criteria used by these groups as they do to any inherent process differences. In fact, pursuit of solutions to the problems identified by either group will frequently benefit both processes with only minor corrections or variations. Conclusions about the degree of technical risk involved in either process design should not be based on the problem areas identified by the demonstration groups. Major differences between the demonstration groups and ORNL (as to problems identified and solutions proposed) are related to the criterion established by the Oak Ridge Operations Office for ORNL recommendations, namely, the extent of direct impact on SRC demonstration plant designs. Solutions proposed by the demonstration

groups are discussed in Sect. 4; the ORNL listing appears in Sect. 3.1. Many of the areas identified in Sects. 3.2 and 3.3 result from perceived impacts on subsequent plant operations and commercialization. The more narrow ORNL charter produces a near-term focus on problem areas in which solutions can potentially influence demonstration plant designs in a timely fashion. The distinction is clearly seen in the presentation of recommendations in Sect. 4.

3.1 Problem Areas Identified by ORNL

The problem areas were identified by ORNL in the initial phase of the assessment through discussions with SRC process developers and others involved with coal liquefaction development. Subsequent discussions and meetings with various groups involved in coal liquefaction have changed the relative importance we attach to the problems but have not substantially altered the problem definitions. The problem areas are listed in Table 3.1; revised problem definitions are given below. Areas receiving detailed assessment in this report are noted in Table 3.1.

1. Slurry mixing. Demonstration plant designs call for elevated temperatures to reduce heat duties on heat exchangers and significantly increase the overall process thermal efficiency. The slurry mixer must take coal of a certain size, blend with process recycle solvent or slurry in fairly precise proportions, and feed this mixture to a preheater (through a heat exchanger in the SRC-I design). The process recycle stream must be cooled to the mixer temperature and the mixer effluent stream should be heated to provide feed to the slurry preheater at elevated temperatures. Thus, if the mixing temperature can be raised to about 505 K (450°F), the heat savings is considerable. As temperatures are increased, the slurry goes through a plastic stage with a sharp rise in viscosity; this creates a serious problem for the mixing and pumping equipment and can cause plugging of lines and valves. Viscosity vs temperature vs residence time data are needed to predict and avoid the plastic stage. The type of mixing device has also not been adequately demonstrated for blending wet, crushed coal with recycle solvent or slurry at high temperature.
2. Slurry flowmeters. Reliable flow-metering instruments are required for the coal-recycle slurry mixture and vacuum-tower bottom streams in the SRC-II process and for the coal-recycle solvent mixture in SRC-I. Pilot plant tests of quadrant-edge orifice plates and target meters have had limited success. Problems with changes in the orifice dimensions because of erosion and coking, calibration of the system for slurry flow, and plugging of the pressure taps with solids have been recurrent.

Table 3.1. Problem areas investigated by ORNL^a

Slurry mixing ^b
Slurry flowmeters ^b
Heat exchangers ^b
Fired heaters for slurries ^b
Reactors/dissolvers ^b
Pressure vessels and piping
Block valves ^b
Slurry letdown valves ^b
Vapor/liquid, liquid/liquid separations ^b
SRC-I solids/liquid separation ^b
SRC-II vacuum-flash system ^c
Centrifugal slurry pumps ^b
Oxygen compressors ^b
Gasification of SRC residues ^b
Gasifier slag, solid waste disposal
Acid-gas removal
Cryogenic separations
Physical properties ^b
Materials erosion/corrosion
Process modeling
Wastewater control technology
Plant area monitoring

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^aNot in order of priority.^bArea assessed in this report.^cConsidered in area 9 (vapor/liquid and liquid/liquid separations).

In the SRC demonstration plants, reliable continuous slurry-metering instruments will be required to control the vacuum-tower bottom feed to the gasifier and flows to preheater tube banks if they are split. Accurate metering of the former stream will be necessary to maintain a constant ratio of fuel/oxygen flow to the gasifier and to prevent significant changes in the reducing atmosphere.

3. Heat exchangers. There is a need for correlated design data on physical properties, heat-transfer coefficients, and pressure drops. Rates of fouling, erosion, and corrosion for specific SRC demonstration plant conditions are also required. The data base in this area is inadequate.
4. Fired heaters for slurries. There is a need for relevant property data, especially viscosity; verified data for flow regimes, heat transfer, and pressure drops; rates of erosion, corrosion, fouling, and coking; and delineation of the peak-viscosity region. There is currently no design basis for fired heaters in coal liquefaction service at turbulent Reynolds numbers and heat fluxes above $31,500 \text{ W/m}^2$ ($10,000 \text{ Btu/hr}\cdot\text{ft}^2$).
5. Reactors/dissolvers. The reactors in the pilot plants were considerably oversized and are operated often at one-half volume. The demonstration plant will provide for less overdesign and will require higher superficial velocities. A number of potential problems associated with operation closer to minimum cost design need, therefore, to be examined. Better temperature control is needed, leading to a requirement for information on exothermic/endothermic reactions, heat capacities, heats of formation, and reactor kinetics. The level of solids accumulation directly affects design. Excessive accumulation of solids demonstrates a need for a solids blowdown system. Too little accumulation may adversely affect conversion kinetics, fluid dynamics, and other parameters. The accumulation of solids depends on the velocity in the reactor, which is related to residence time and kinetics of the reaction. The catalytic effect of the accumulated solids also affects reaction kinetics and, consequently, design parameters.
6. Pressure vessels and piping. Pressure vessels and piping in some applications in SRC systems will be subjected to high pressures and possibly, under off-design conditions, to high temperatures. In addition, these pressures and temperatures will be coupled with hostile environments due to the contained media. Consequently, particular attention is required in the design and fabrication of piping and pressure vessel components to achieve long-term, reliable service under both constant and fluctuating thermal and mechanical loading conditions. Fatigue will be an important source of failure.
7. Block (shutoff) valves. Failure of block valves is considered by pilot plant personnel to be one of the more critical, unsolved problem areas because it prevents repair of installed equipment while

switching to spares. Erosion of seals by solids prevents tight initial shutoff, and subsequent leakage accelerates erosion. Shutoff valves are subject to sticking in the normal operating position because of solids accumulation between sliding surfaces. Leakage to the environment of noxious and/or flammable process constituents must also be controlled; thus, sealing of high pressure and temperature slurries is important.

8. Slurry letdown valves. Experience at SRC-related plants has shown that standard industry-supplied valves, even when designed for the temperatures and pressures encountered in the SRC letdown process, will not last long under erosive conditions. Trim wear is excessive and stem breakage has occurred. Violent flashing and cavitation aggravate erosion so that new or modified designs are needed.
9. Vapor/liquid, liquid/liquid separation. The design of the slurry letdown system calls for the use of vapor/liquid equilibria data, which are currently very limited. Thus, there is a need for this type of data. With reliable vapor/liquid equilibria data, cost savings for the slurry letdown design might be significant. In other areas, these data would have a much greater impact. The H_2 , CO , and other solubility data affect the sizing of the H_2 purification system. The vapor/liquid equilibria of light and heavy oils affect the sizing of the separation equipment, which may have an impact on the coking tendencies of the heavy liquid streams. The liquid/liquid equilibria of light oil and water affect (and perhaps control) emulsion tendencies in the solvent decanter.

Although not pertinent to this problem area, the vapor/liquid equilibria data would also be applicable to the dissolver design. Currently, it is thought that as much as 30% of the solvent is in the vapor phase in the dissolver, which reduces the amount of solvent available as a hydrogen donor. Adequate vapor/liquid equilibria data could optimize the cut used as a solvent and thus minimize such vaporization.

10. SRC-I solids/liquid separation. This is one of the more critical problem areas for SRC-I. Many technical and operational problems remain, especially in regard to maintaining acceptable throughput and demonstrating equipment reliability. This separation step is conservatively estimated to account for 10% of the product cost when precoat filtration is used. Thus, any improvements in the separation step have the potential of significantly lowering product cost and increasing plant reliability.
11. SRC-II vacuum-flash system. Although the use of a vacuum tower as the separation unit for SRC-II changes the nature of the concerns expressed for SRC-I, the concerns are nonetheless significant. To a great extent, the tower controls the amount of fuel oil product recovered; thus the unit's optimization could significantly decrease the product cost. Throughput could be a problem because of the possibility of the vacuum bottoms plugging. Plugging problems could

be caused by coking or an excessive solid/liquid ratio. Adequate vapor/liquid separation, without entrainment, at demonstration plant design conditions remains to be demonstrated.

12. Centrifugal slurry pumps. Centrifugal pumps are required for several high-volume, low-to-intermediate head applications in coal conversion plants. The principal problems that have been encountered are erosion of parts in contact with high-velocity slurry and leakage through shaft seals or packing. Extensive research on materials has been done, and materials having acceptable erosion resistance have been identified. Slurry pump manufacturers claim to offer pumps that will give satisfactory (about one year) service life, but pumps of this quality have not been installed and operated at design conditions in the pilot plants for sufficient time to verify the claims. In particular, operation of a pump to remove the vacuum bottoms slurry from the SRC-II vacuum-flash tower is unproven.
13. Oxygen compressors. The gasification pressures selected for the SRC demonstration plants could require oxygen delivery pressures of up to 10.4 MPa (1500 psig). The pressure selected should represent a design decision reflecting the need for high-pressure composition H_2 in the liquefaction loop. The optimal gasification pressure specified by the SRC design teams should be based on oxygen, steam, and residue pumping costs; economics of higher gas throughput at higher pressure; costs of compressing high-purity H_2 ; and potential equipment and process limitations. In this context, centrifugal oxygen compressors of the capacity required in the demonstration plants have not been operated above 7.0 MPa (1000 psig). Although there is confidence in the compressor industry that such compressors could be designed, built, and operated successfully at pressures approaching 10.4 MPa, the actual operation of a high-pressure, high-capacity oxygen compressor should be demonstrated before a final decision specifies high-pressure oxygen delivery.
14. Gasification of SRC residues. Long-term operability and performance characteristics with various design feed streams are needed, especially with regard to refractory, burner, feed pump, and valve lifetimes associated with the slag pit.
15. Gasifier slag, solid waste disposal. Ash or slag from the residue gasifier should be landfilled. This ash will contain a large fraction of the inorganic materials present in the coal feed and may contain up to 5% organic material. The leaching potential of this material is undoubtedly of critical importance, and various Environmental Protection Agency and Resource Conservation and Recovery Act regulations will require testing and special disposal methods if the material is found to be hazardous.

16. Acid-gas removal. The actual amounts, effects, and ultimate disposition of trace components in the gas streams are, in general, unknown. These data are needed particularly for non-H₂S sulfur compounds (COS, CS₂, and mercaptans, for example). Data on the partitioning of minor and trace compounds such as phenols, oils, NH₃, and HCN between treated gas, regenerated offgas, and any liquid blowdown streams are needed to determine the potential impact on downstream processes (e.g., cryogenic separation); environmental control processes (e.g., sulfur recovery and solvent blowdown treatment or disposal); and the environment in general (e.g., CO₂ vent streams, sulfur recovery tail gas, and fuel gas combustion).
17. Cryogenic separations. As in the problem area of acid-gas removal, the principal concern is characterization of the gas stream to include trace compounds. Trouble in the cold box is avoided by adequate pretreatment of the feed gas (e.g., liquid wash, removal of water vapor by adsorption or absorption, molecular sieves, etc.). However, without detailed characterization of the feed gas, particularly with respect to the range of likely variations, extensive and redundant cleanup systems are necessary to ensure proper operation of the cryogenic separator. Such overdesign represents an economic penalty to the demonstration plant.
18. Physical properties. Very few basic property data relevant to design are available on coal conversion process streams, particularly for coal-derived liquids. The American Petroleum Institute *Technical Data Book* and the *Coal Tar Data Book* supplemented by limited actual data, are the primary tools used by present designers. The former reference deals with petroleum-derived materials that, in many cases, differ greatly from coal-derived materials. The latter publication is very limited in content. The result is obvious: designers must incorporate factors indicative of low confidence levels, which can result in considerable overdesign and unnecessary expense, or they must accept the risk of operating difficulties. The pilot plants were designed to demonstrate advanced technology and, in many cases, are either not capable of providing adequate basic property data or have not been used to develop such data. The limited amount of data available is being used by designers because it pertains to process conditions, but accuracy considerations remain. Most of the data available from other researchers is at conditions far removed from the SRC process design, and correlations to extend the data to this range are inadequate or nonexistent.

Obtaining basic properties and developing correlations useful at other conditions will result in cost savings and increased confidence in the demonstration plant design. Use of petroleum correlations could result in serious errors, which can produce large liabilities for coal conversion facilities as large as 5.4 Ggd (6000 tons/d). Data most needed are viscosities, specific heat capacities, densities, thermal conductivities, and heating values. These data are

particularly important for the critical fired heaters and heat exchangers being designed for demonstration plants. Solubilities of coal-derived materials in water and of salts in water/oil mixtures are also of considerable importance.

19. Materials erosion/corrosion. A number of specific problems within the general area of materials have been identified. In several cases, these overlap other problem areas (such as valves), which should be reviewed concurrently. The specific problems are defined separately below.
  - a. Erosion of pumps and transport lines for cold-slurry handling. The hard abrasive particles in the cold-slurry portions of SRC-I and SRC-II erode centrifugal pumps and transport lines, leading to unscheduled downtime while failed components are replaced.
  - b. Erosion/corrosion of hot-slurry-handling pumps. The erosive/corrosive nature of the hot slurry in SRC plants leads to excessive wear and high failure rates in hot-slurry-handling pumps.
  - c. Erosion/corrosion of trim sets for letdown valves, letdown of solids from dissolvers, and durability of seats for shutoff valves. The SRC pilot plants have demonstrated that letdown valves are one of the more difficult problems. The most common problem is that the tungsten carbide trim drops off the stem. The erosive nature of the slurry also limits the service life of the trim to about three months. It is desirable to extend this service life to one year.
  - d. Identification of corrodents. Because of the complex and continually changing chemistry of coal liquefaction processes, it is difficult to identify the species that cause corrosion in the system. Methods must be developed for grab sampling process-stream material and quick quenching to identify real process conditions for correlation with materials data. The allowable levels of moisture and air must be determined, and chemical changes during shutdown (for example, generation of polythionic acid, which may be a cause of cracking) must be examined.
  - e. Gasifier materials of construction. There have been reports of problems with refractory life, burner-nozzle erosion, and erosion of the slag-removal valve in the Texaco gasifier.
  - f. Hydrogen attack of 2-1/4-Cr-1-Mo steels and weld overlay materials used for pressure vessels. The hydrogen present in coal liquefaction systems may lead to deterioration of the properties of 2-1/4-Cr-1-Mo steels and weld overlay materials used for pressure vessels in SRC liquefaction systems.

- g. Nondestructive examination (NDE) methods development. NDE methods should be developed for areas of the plant where continuing or periodic surveillance is desirable for safety purposes. Sample NDE methods include cladding inspection, rotating equipment inspection, detection of stress-corrosion cracking, and bend-wall thickness examination.
  - h. Stress-corrosion cracking of vapor/liquid separator because of salt buildup. There is concern that the buildup of NH_4Cl in the vapor/liquid separator will lead to stress-corrosion cracking.
  - i. Corrosion of fractionation column. The SRC-II Pilot Plant at Ft. Lewis has experienced severe general corrosion in the fractionation column.
  - j. Materials design review for SRC demonstration plant. Concurrently with the detailed design of the SRC demonstration plant, a materials design review should be performed to evaluate the adequacy of materials selected for use in the demonstration plant and to make recommendations for materials of construction to improve plant reliability.
20. Process modeling. Two basic categories of process modeling that are strongly desirable for the SRC programs are simulation/operation models and process cost models. Although steady-state simulation models are highly useful for sizing reactors, estimating the overall effects of process changes, etc., dynamic operation models are equally important. Such transient modeling, of a reactor system specifically, and the process in general, permits the development of reasonable plans of action for control of the process during start-up or shutdown conditions. Problems encountered in heat-exchange coupled areas (feed-effluent exchanger problems, for instance) can also be investigated. The need to develop simulation/operation models has been expressed elsewhere; physical properties, reaction kinetics, correlations for heat transfer, etc., are necessary input and, in many cases, remain to be developed.

The category of cost models derives importance from the designers' need for trade-off and optimization studies to ensure that demonstration plant designs represent an economically preferable route to clean fuels from coal via SRC. The unknowns listed above also affect the development of adequate cost models. Design uncertainties can be evaluated with cost models to determine economic penalties associated with currently necessary overdesign, thus providing the justification for additional R&D. For example, application of a cost model to data uncertainties in fired heater design could demonstrate the economic incentive for significant data acquisition in this area in support of SRC Demonstration Plants Projects.

21. Wastewater control technology. To determine the environmental impact of SRC wastewater, it is important that a complete characterization of the wastewater be undertaken. This characterization should be conducted with wastewater from the SRC pilot plants and should include the amount of biologically active materials as a function of operating conditions and coal types. Of obvious importance will be the usefulness of these data in the design of wastewater control systems.

Solvent extraction systems have been used for phenol recovery in a number of industrial applications, but the economic viability of such systems is still undetermined. Whether solvent extraction will be used or not is obviously important to the design of any downstream water treatment processes.

Although numerous groups are investigating specific control technologies and/or integrated control schemes for coal conversion processes, none is looking at processes specifically designed for SRC.

Simple and inexpensive biological tests should be incorporated in the demonstration program plan to test the toxicity (both acute and chronic) mutagenicity, and carcinogenicity of releases.

22. Plant area monitoring. Coal conversion plants are likely to have numerous surfaces contaminated with hazardous compounds. Several classes of these compounds, the polynuclear aromatic hydrocarbons and the nitrogen heterocyclics, have been identified in coal conversion products and have been characterized as particularly carcinogenic. Detection of these materials in the work place is a necessary adjunct to proper industrial safety/hygiene programs.

3.2 Problem Areas Identified by SRC-I Demonstration Group

Air Products and Chemicals, Inc. (APCI), identified a number of experimental programs that could improve the engineering data base for SRC-I and potentially affect the Phase I detailed design work.³ Some of the proposed programs involve in-house APCI activity; others require outside institutions to perform the indicated work [under subcontract with APCI or under other U.S. Department of Energy (DOE) contracts]. The data needs will be briefly discussed in this section; the status and plans for obtaining the necessary data are discussed, where applicable, in our recommendations in Sect. 4.

The proposed programs are divided into the following seven areas:

1. Reactors/dissolvers
2. Fired heaters

3. Solids/liquid separations
4. Residue gasification
5. Slurry mixing
6. Heat exchangers
7. Coal liquids stability

A discussion of each area follows.

1. Reactors/Dissolvers

- a. Fluid mechanics

The demonstration plant dissolver design represents a significant scale-up over the Wilsonville and Ft. Lewis dissolvers. The demonstration-scale dissolver will require slurry and superficial gas velocities up to tenfold greater than those used at Wilsonville. The corresponding gas void volumes will also be increased substantially. The effect of these higher velocities on solids suspension and movement through the reactor is not well understood. Experimental measurements are needed to determine the effect of superficial gas and liquid velocities on mixing, bubble size, interfacial contact, solids retention, solids particle size [especially if 841- μm (20-mesh) coal is to be considered as a feed], and to optimize the fluid injection equipment design. The latter includes the need to determine distribution plate design and location.

- b. Performance characterization

Uncertainty exists as to the quantity and characteristics of solids that will accumulate in the commercial reactor. On scale-up of the dissolver to demonstration capacity, solids accumulation may be low because of the higher gas and slurry velocities through the reactor. The effect of low solids accumulation on the process is uncertain. However, solids are known to provide some catalytic effects on all coal liquefaction processes. Another factor that creates some uncertainty is the behavior of 841- μm (20-mesh) coal particles in the dissolver. If used, the larger initial particle sizes could result

in larger sized accumulated particles within the dissolver. For optimized reactor configuration, data are needed to understand the effect of:

- reactor solids on conversion, yield structure, solvent rehydrogenation, and desulfurization;
- relative concentration of solids on the various reactions, on hydrogen consumption, and on the reduction in actual residence time because of solids displacement of liquid phase.

c. Kinetics

A kinetic model and correlation are needed to interpret the effects of operation at the demonstration plant scale. Development of the correlation from laboratory-scale kinetic data and application of the correlation to larger scale operations (to the Wilsonville unit, for example) would assist in determining effects of scale-up on reaction kinetics. The model is needed to describe how process operating conditions (retention time, temperature, pressure, etc.) can affect product yield.

d. Modeling

An important tool in the efficient design and operation of a complex plant is a system model capable of integrated heat and material balances. By looking at all unit operations simultaneously, the interaction of equipment and the effects of heat or material recycle can be established. Methods are needed for performing accurate and prompt heat and material balance calculations around the SRC-I dissolver, the associated heat exchanger and flash separators comprising the reactor system, and the hydrogen recovery portion of the SRC-I process. These methods should provide:

- a means of performing sensitivity studies for various operating conditions,
- a basic understanding of the process characteristics so that a dynamic analysis can be planned and executed.

- the ability to test a model of the process against pilot plant data.

In addition to the steady-state model, a dynamic mathematical model of the preheater and reactor would be useful for determining the temperature response of the system to variations of feed temperature, component feed rates, and pressure. The mathematical model would assist in determining system features that may be required for safe operation.

2. Fired Heater

a. Reaction kinetics

Data are needed for the effects of varying coal feed size and residence time at demonstration plant design temperature and pressure on product composition entering the dissolver. Such information can potentially have an impact on fired heater and dissolver designs and operating conditions. By obtaining data at several scales of operation, effects of scale-up could also be assessed.

b. Heat transfer and physical properties

To ensure adequate design of the demonstration plant fired heater, data are needed on pressure drops and heat transfer coefficients for coal-solvent-hydrogen mixtures at temperatures of 530 to 730 K (500 to 850°F). Laboratory-scale slurry viscosity and enthalpy measurements would aid the development of correlations suitable for use at demonstration plant design conditions.

c. Erosion

Data on tube erosion at demonstration design conditions are needed to assist in selecting appropriate materials of construction and determining allowable tube-side velocities.

3. Solids/Liquid Separations

a. Kerr-McGee Critical Solvent Deashing

To establish the optimum design basis for the Kerr-McGee process, the following data are desired:

- performance of primary phase separator with varying phase compositions and velocities,
- separation efficiency of large-scale separator and laboratory-scale equilibrium at same conditions,
- viscosity of Kerr-McGee residue and critical solvent at conditions of primary separator exit.

b. U.S. brand filter

To determine the optimum design of the U.S. brand filter in the demonstration plant, information on the following filter aspects is needed:

- quantity and type of precoat;
- filter screen — mesh size, weave, blinding;
- drying of the filter cake;
- filter cake thickness and relationship to throughput;
- cycle time.

4. Residue Gasification

a. Texaco gasifier

To determine the critical aspects of Texaco gasification using SRC-I mineral-ash-residue (MAR) design feed, acquisition of the following data is necessary:

- measurements of flow characteristics and stability of feed slurry,
- measurement of refractory condition before and after tests,
- analyses of product slag for carbon content and environmentally undesirable constituents,
- analyses of product gas for major and trace impurities,
- carbon conversion efficiency,
- SRC-I MAR feedstock reactivity.

b. Koppers-Totzek

To assess the potential of using an alternative gasifier in the demonstration plant design, data on yields and

data on oxygen and steam consumptions for MAR gasification in a Koppers-Totzek gasifier are desired.

5. Slurry Mixing

Design of the slurry mix tank for operation at temperatures above 400 K (250°F) would reduce the amount of slurry heating required and also reduce the extent of cooling of recycle solvent necessary. To ensure operability at design temperatures above 400 K, data are required on design slurry viscosity and stability in the temperature range of interest, 360 to 450 K (200 to 350°F).

6. Heat Exchangers

Data on tube erosion at demonstration plant design conditions are needed to assist in selecting appropriate materials of construction and in determining allowable tube-side velocities.

7. Coal Liquids Stability

Instability of liquefaction product can have an effect on the handling of products downstream from the reaction vessels. The holdup of products at different temperatures in various pieces of equipment can affect the degree of repolymerization. A design option for the demonstration plant involving gas/liquid phase separation at temperatures near the dissolver outlet temperature offers considerable energy savings. The increase in efficiency results from reduction of the fired heater demand on the vacuum distillation column. High-temperature instability has been qualitatively identified at Wilsonville, but few quantitative measurements are available. Information is needed to determine the feasibility of operating the separator at temperatures close to 700 K (800°F). The effect of time at temperature and the effect of hydrogen partial pressure under these conditions are also needed to confirm the concept feasibility and to allow vessel sizing. Information is also needed on the behavior of these products in the absence of hydrogen, such as is experienced in the vacuum tower reboiler.

3.3 Problem Areas Identified by SRC-II Demonstration Group

The Pittsburgh and Midway Coal Mining Company (P&M) identified areas considered critical to the success of the SRC-II Demonstration Plant.⁴ Over two-thirds of the individual activities listed by P&M as important to the SRC-II Demonstration Plant Project were cited as being funded by DOE, principally at the Ft. Lewis SRC Pilot Plant and at the SRC Process Development Unit at Harmarville. We report the activities listed by P&M under the following categories:

1. Reaction system
2. Separation systems
3. Residue gasification
4. Heat exchange
5. Coal liquids characterization
6. Physical properties
7. Other activities

No distinction is made in this section between currently funded and proposed work; this distinction is more appropriately made in Sect. 4 as part of the discussion of preferred approaches to obtaining necessary data.

1. Reaction System

a. Preheater exit analyses

The extent of reaction occurring in the slurry preheater before entry into the reactor is an important factor in the design of the reactor. To assist in scale-up of the reactor/dissolver from the pilot plant to the demonstration plant, data are needed on preheater outlet composition in comparison with analyses of dissolver exit composition.

b. Kinetics

Laboratory-scale kinetic data on effects of solvent quality, temperature, pressure, residence time, preheater configuration, mineral residue size and composition, and variations of ash recycle amount and composition are important for improvement of existing reactor models, which are used to scale up reactor design to demonstration

conditions. Data on inherent reactivity of recycle (middle, heavy distillate, and SRC) in the absence of coal also contribute to the formulation and evaluation of the reaction area demonstration design basis.

c. Reactor modeling

Improvement of the current steady-state model, development of a transient model, and additional confirmation of the fluid dynamics relationships now used would assist in specifying the demonstration design basis, evaluating alternative reaction conditions, evaluating alternative reactor designs, specifying important scale-up factors, studying alternative quench locations and media, evaluating stability and safety, and developing reactor control procedures and hardware.

d. Characterization of catalytic mineral matter

Characterization of the minerals present in the feed coal and the changes that occur during liquefaction is expected to lead to better selection of demonstration plant feed coals and a better understanding of the catalytic effects of the mineral components. Such understanding could also help optimize the degree of coal preparation to be used in large-scale operations.

e. Heavy media separation

The extent of coal preparation can influence the ease and degree of coal liquefaction. Information from the testing of various fractions of representative coals would aid in the selection of feed coals and design of appropriate coal preparation systems.

f. Catalytic effect of mineral matter

Determining the catalytic effect of added expendable minerals on coal liquefaction should facilitate the development of inexpensive techniques of using coals that are difficult to liquefy in the demonstration plant. An alternative coal is to develop methods to liquefy coals at milder conditions than those currently planned. Attainment

of such goals should broaden the usable feedstock base or decrease expenses associated with currently identified usable feedstocks.

g. Feedstock screening methods

Measurement of feedstock reactivity currently requires expensive bench-scale testing. Development of a reliable, less expensive method would ease the selection and ranking of suitable coals for the demonstration plant.

h. Comparison of yield data

Variations in yields occurring in the Ft. Lewis, Harmarville, and Merriam facilities need to be evaluated to determine the relative contributions of analytical procedures, operational differences, and scale factors.

2. Separation Systems

a. Cryogenic separation

Design of the cryogenic separation plant depends on the amount of carbon monoxide allowable in the recycle hydrogen stream. Data on the impact of varying carbon monoxide concentration in the hydrogen feed on carbon dioxide yields and hydrogen consumption are needed for design of the cryogenic plant.

b. Coking temperatures

The proposed operating temperatures in the demonstration plant design of high-temperature separators and vacuum-flash systems are higher than those for which data are available. To minimize uncertainties with higher temperature designs, measurements of coking for heavy coal-derived liquids are needed.

c. Vapor/liquid equilibria

To increase confidence in the heat and material balances for the demonstration plant design of vapor/liquid separations, further data are needed to confirm the correlations currently used.

d. Vacuum-flash systems

Data from operating vacuum-flash systems are needed to provide demonstration design input regarding:

- vapor-liquid separation,
- solids carry-over,
- coking experience,
- instrumentation and control,
- effects of process variations.

e. Sour-water composition

Data on the partitioning of ammonia, carbon dioxide, and hydrogen sulfide between vapor and liquid phases at demonstration design conditions are needed for design of the ammonia scrubber and downstream sour-water treatment.

f. Emulsion formation

Two flash drums in the demonstration plant have possible multiphase (oil/water) separation problems. Data are needed on the potential for emulsions forming because of the small specific gravity differences between aqueous and oil phases at the conditions proposed.

g. Solvent recovery corrosion

Information on possible corrosion in the solvent recovery area is needed to assist in selecting appropriate materials of construction and corrosion control strategies for fractionation columns.

3. Residue Gasification

a. Slag/water testing

Analyses of the slag and water effluents from residue gasification are needed for two reasons – determination of subsequent treatment or disposal design and assessment of potential environmental impacts. Water analyses relate principally to the design of treatment systems; slag analyses, to ultimate disposal.

b. Gasifier charge pump

The gasifier charge pump represents a critical factor in demonstration plant design, delivering high-solids-content

vacuum tower bottoms at high temperature to the gasification plant. Because of the severity of service, actual performance information is needed to reduce current uncertainties.

4. Heat Exchange

a. Recycle slurry viscosity

To properly design the recycle slurry cooler, viscosity measurements are needed as a function of temperature [from 670 K (750°F) to 480 K (400°F)] and shear rate (to determine degree of non-Newtonian behavior).

b. Slurry-feed preheater

Heat transfer, pressure drop, and erosion data are needed at more severe conditions than those at which pilot plant fired heaters are currently operated. These data are necessary for the confident design of the demonstration-scale preheater under more severe design criteria. Use of the very conservative approach currently supported by pilot plant operation would result in a substantial economic penalty.

c. Dissolver-separator vapor cooling

Data are needed on pH, corrosivity, and composition in the liquid-water phase produced by cooling the dissolver-separator vapor from 620 to 510 K (650 to 450°F). These data would be of use in specifying downstream water treatment requirements.

d. Dissolver-separator vapor heat exchanger

Data on entrainment, corrosion, erosion, and operation of a heat exchanger cooling the separator vapor could allow design of the vapor-cooling train without use of the direct water quench currently operating at Ft. Lewis.

5. Coal Liquids Characterization

a. Distillate product cut-points

To optimize distillate product cuts, data are needed on the impact of varying naphtha and fuel oil cut points on product quality.

b. Product evaluation and utilization

To assist in determining product marketability, the following information is needed:

- identification and evaluation of techniques for suppressing NO_x emissions in boilers;
- identification and resolution of problems in storing, shipping, and handling SRC-II fuel oils and mixing of the fuel oils in customer tanks;
- development of relevant test methods for compatibility and stability;
- product evaluation for turbine fuel, diesel fuel, and chemical and other applications.

6. Physical Properties

a. Feed-slurry viscosity

To assist in the design of slurry mixing, pumping, and heating operations, data are needed on the viscosity of the feed-coal slurry as a function of temperature, shear rate, time at temperature, and coal concentration, particle size, and moisture content.

b. Recycle slurry solids

To design the high-pressure slurry charge-pump flushing system, measurements of the size distribution of solids in the recycle slurry are needed.

c. Solids settling rate

Data on the settling characteristics of solids in the feed-coal slurry are needed for design of the mix tank and slurry lines.

7. Other Activities

a. Slurry mixing

Design of the slurry mixing system requires information on various approaches to blending wet, crushed coal with recycle slurry at elevated temperatures. For economic reasons, operability needs to be assessed at the highest possible mixing temperature. Two advantages occur from

higher mixing temperatures: reduction in subsequent feed slurry preheater duty and reduction in duty or elimination of recycle slurry cooling.

b. Demonstration plant cost model

Development of an SRC-II process cost model that would express capital and operating costs of an SRC-II plant as functions of component size, operating conditions, and other factors, including calculations of discounted cash flow/return on investment as functions of process yields, prices, and cost elements, would be highly useful in subsequent engineering trade-off studies. The model would also assist in determining the economic attractiveness of various potential process improvements, thus establishing economic justification for additional research projects to pursue such improvements.

c. Control and block valves

Test data on control and block valves are needed to assist in resolving current operational problems experienced with such valves in the high-pressure reaction area. A successful resolution of this problem area would reduce the number of parallel valve assemblies envisioned for the demonstration plant and would eliminate potential downtime attributable to valve failure.

d. Slurry flowmeter

Data from slurry flowmeter tests are needed to develop flow correlation curves and to evaluate the accuracy, reliability, and wear characteristics of a variety of slurry flowmeters in various service applications. The combination of high temperature, high pressure, high solids concentration, varying viscosity, multiphase flow, corrosion, and erosion results in a very difficult service application.

e. Corrosion/erosion studies

To assist in materials selection for demonstration plant design, the following information is needed:

- evaluation of several possible alloys for service in the high-pressure gas system of the demonstration plant,
- study of the fouling potential of SRC-II naphtha,
- measurement of the corrosion/erosion resistance of Type 321 and Incoloy 800 stainless steels to various coal slurries in the process.

f. Potential health problems

Toxicological and industrial hygiene studies are needed to further investigate potential health problems associated with plant streams and products and to further evaluate measures currently being used to protect workers and the general public.

4. RECOMMENDATIONS

Oak Ridge National Laboratory (ORNL) recommendations are in three separate categories: activities that can potentially supply answers directly useful for the Solvent Refined Coal (SRC) demonstration plant designs (Sect. 4.1); activities that should provide additional understanding of SRC technology (Sect. 4.2); and activities that are generally supportive of direct coal liquefaction but would not have an impact on SRC demonstration plant designs (Sect. 4.3). Recommendations in most problem areas defined in Sect. 3 have components in more than one of these categories. For example, certain physical properties, which have a direct impact on SRC demonstration plant designs, are needed at specific design conditions (temperature, pressure, coal type, etc.). Determination of physical properties for other conditions would be supportive of SRC technology as well as aiding in subsequent plant operations and future designs. Development of reliable correlations of predicting or explaining property variations versus process conditions is a reasonable long-term goal generally supportive of coal liquefaction.

Only the first category contains direct recommendations for considerations by the Oak Ridge Operations Office (ORO) of the U.S. Department of Energy (DOE). The remaining categories are included to indicate the areas in which suggestions could be made in support of a cohesive policy for commercialization of coal liquefaction.

Within each category, we attempted to order the recommendations relative to the identified needs of SRC Demonstration Plant Projects (SRC DPP). In Sect. 4.1, this requires individual ranking of each recommendation; however, in the subsequent sections we grouped the remaining recommendations in respective problem areas and only ranked the areas. Introductory or explanatory information for the problem areas is provided when the area is initially cited. If there is current, planned, or proposed work at the various SRC installations that relates to the recommendations presented, this is indicated, particularly in Sect. 4.1. Recommendations within each problem area are numbered sequentially throughout Sect. 4. For example, the first recommendation cited for fired heaters (in Sect. 4.1) is numbered 1, and the last recommendation for this problem area (in Sect. 4.3) is numbered 7.

4.1 SRC Demonstration Plants Projects

In this section, activities that could provide significant technical input to current design efforts, subject to the current contract schedule, are offered for ORO consideration. Recommendations are provided in the approximate order of importance to SRCDPP designs. We recognize that opinions differ regarding the appropriate ranking and that institutional constraints (e.g., funding and scheduling conflicts) may heavily influence implementation of these recommendations. Many of the recommended activities listed will initially support design efforts and will also extend to the support of subsequent plant operations and commercialization. This is indicated by funding estimates that extend beyond FY 1980.

Fired Heaters for Slurries

1. Continuation and coordination

In addition to the new and/or expanded studies recommended, the existing programs at the pilot plants, particularly Ft. Lewis, should be continued to support design efforts in the SRCDPP. This recommendation is consistent with those of the SRC demonstration groups [reference problem definition 2b in Sect. 3.2 (SRC-I) and 4b in Sect. 3.3 (SRC-II)]. Existing programs are described in Sect. A.2. Special attention should be given to technical coordination of these various projects and should extend to the establishment of a central data interpretation/correlation effort, in addition to efforts at the individual sites. This effort could be part of the coordination effort discussed under "Physical Properties." The current status of fired heater correlations for non-Newtonian mixtures is reviewed in Sect. A.2.2.

2. Slurry rheology data

Acquisition of slurry rheology data should be continued, especially at slurry temperatures above 480 K (400°F) and at wall shear rates in the range of 10 to 1000 s⁻¹. A moderate

expansion of current programs is indicated. With emphasis on existing small-scale programs, \$300,000 per year for two years is indicated; this level of funding excludes Exxon and in-place pilot plant programs (see Sect. A.2). Both SRC groups also recommend acquisition of these data [SRC-I, Sect. 3.2 (2b); SRC-II, Sect. 3.3 (4a, 6a)]. We believe that work with flow systems is preferable, in combination with other physical and thermal property work. The SRC-II group suggests that this information will be obtained from planned work at Ft. Lewis on viscosity measurements (e.g., a Haake viscometer has been undergoing shakedown in recent months). We agree that this program should be continued, but we believe that additional work is warranted.

Slurry Mixing

The demonstration plant designers prefer higher effluent temperatures from the slurry mix tank to reduce heat duties on heat exchangers and to increase the overall thermal efficiency of the process. However, as temperatures are increased, the slurry goes through a plastic stage with a sharp rise in viscosity. This creates a serious problem for the pumping and mixing equipment and can cause plugging of lines and valves. Viscosity vs temperature vs residence-time data are needed to predict and to avoid the plastic stage. Also, the mixing device for blending wet, crushed coal with recycle solvent at high temperatures has been demonstrated. Current design efforts assume the blending of dry, pulverized coal at moderate temperatures, [e.g., up to about 450 K (350°F)] as a low-risk option. More efficient operation, although higher risk, is still a desirable goal.

Some mixing studies have been performed at Ft. Lewis, but equipment limitations have prevented operation at the slurry residence times and temperatures desirable in the demonstration plants. Because of the large potential for process savings and the potential for process problems, additional data are needed for SRC demonstration plant designs.

1. Pilot plant mixing studies

The pug mill and vortex mixer projects at Ft. Lewis should be expedited. High-temperature operation of the mix tank should be carried out when process operations permit. Tests of mixing conditions and equipment identified by other projects should be instituted as a cooperative effort. Mixing efforts [e.g., Exxon Donor Solvent (EDS) and H-Coal] at other pilot plants and their supporting laboratories should be coordinated with the Ft. Lewis effort. Currently, there is little information exchange. This should be accomplished within current Ft. Lewis funding, consistent with Pittsburgh and Midway (P&M) Coal Mining Company's recommendation [SRC-II, Sect. 3.3 (7a)] for obtaining data on these systems under the Ft. Lewis contract.

Solids/Liquid Separations

Solids/liquid separation remains one of the most critical research and development (R&D) problems for SRC-I. The thrust of the national R&D solids/liquid separations program, encompassing universities, technology centers, and pilot plants represents a comprehensive and cost-effective approach to the problem. We concur with Air Products and Chemicals, Inc. (APCI), [SRC-I, Sect. 3.2 (3a, 3b)] that continuation of Wilsonville's activities should be strongly supported to provide further information on the Kerr-McGee and U.S. brand filter options. Assuming that existing and planned research projects at the major SRC support facilities are funded at anticipated levels, the following is recommended for initiation to provide additional support to the SRCDPP.

1. Carbonaceous precoat project

In an attempt to reduce the high costs of precoating, at an estimated usage of 58 Mg/d (64 tons/d) for a 5.4-Gg/d (6000-tons/d) SRC-I demonstration plant, a program should be initiated to assess the feasibility of using a carbonaceous precoat that would have a recoverable heating value in a gasifier. We and APCI support the program at Wilsonville,

which entails studying the effects of sized anthracite precoat on filtration rate and equipment performance. Carbonaceous precoat screening tests should also be run simultaneously at bench scale in a laboratory equipped for this purpose. The screening test could be accomplished in one year at laboratories with in-place filters; the cost would be about \$200,000.

Vapor/Liquid and Liquid/Liquid Separations

The SRC demonstration plant design engineers currently rely on correlations for vapor/liquid equilibria developed for petroleum fractions in the early 1960s, such as the Grayson-Streed and Chao-Seader correlations, modified by a limited amount of actual process data. Even though these data are the best currently available, it has been shown by Chao at Purdue, for example, that the resulting estimates for vapor/liquid equilibria, when applied to coal liquids, are in error by as much as 40% for the standard correlations. It appears obvious that a research effort is warranted in this area.

The specific areas of the SRC process for which equilibria data are of prime importance are:

1. Reactors/dissolvers (light-gas solubility in process solvent and fraction of process solvent vaporized and therefore unavailable for hydrogen transfer)
2. Slurry letdown system (separation of light gases and coal-derived light end products from SRC and unreacted coal)
3. Vacuum fractionator (separation of heavy end products from lighter oils)
4. Solvent fractionator (final removal of other components from solvent before recycle)

The area of most concern is the impact of the slurry letdown system on the remainder of the plant. This vapor/liquid separation downstream of the dissolver dictates the quantity and composition of both the vapor stream going to purification and H₂ recycle and the liquid stream going to solvent recovery, solids/liquid separation, and product fractionation.

If the vapor/liquid equilibria correlations available are in error by even a small amount, the remainder of the plant could be oversized by a large factor, thus incurring unnecessary capital costs. Conversely, a tighter design could result in reduced capacity for the facility caused by insufficient design margin.

There are a number of experimentalists in the vapor/liquid equilibria area, as discussed in the Appendix. However, in general, there is a lack of overall coordination of the research effort, a lack of data being taken on actual hot-fresh coal liquids, and no use of a flow-generation system.

1. Coordination project

To ensure successful coordination of data acquisition and correlation, it is proposed that a working group be formed on determination and correlation of vapor/liquid equilibria for coal-derived liquids. Representatives from industry, government laboratories, and universities would form the group. Because data acquisition for the SRC demonstration plant is the most critical, it should receive priority in the direction of the research. The current SRC-II vapor/liquid equilibria calculations have been compared with data from the Harmarville vacuum tower, which is funded under the current Ft. Lewis contract [SRC-II, Sect. 3.3 (2c)]. We recommend a broader approach, including comparisons with Ft. Lewis data, for this coordination project so that both SRC-I and SRC-II design correlations receive the benefit of such confirmation. This effort could be part of the coordination effort discussed under "Physical Properties." The estimated overall cost is about \$100,000 per yr.

Gasification of SRC Residues

1. The maximum test duration in the Texaco Montebello gasifier, based on maximum charge and minimum feed rate, is only 5.6 days (Type 3) in the recent DOE Texaco contract. This contract, which is currently being renegotiated for extended testing,

has resulted in valuable preliminary results. The new contract is expected to allow for 7- to 10-day runs. We recommend future single continuous runs of at least 15 days and preferably 30 days at Texaco Montebello Research Laboratory in order to develop realistic information on gasifier refractory resistance to the high molten slag loadings involved. Longer runs would also provide the data and/or samples desired by P&M [SRC-II, Sect. 3.3 (3a)] and APCI [SRC-I, Sect. 3.2 (4a)]. The cost of such runs is properly the subject of ongoing negotiations. The cost would undoubtedly be higher than that associated with Type 3 tests performed under the previous contract, because of the increased duration recommended. Further recommendations in this area are not possible without a site visit to the Texaco Montebello facility.

Reactors/Dissolvers

The reactor design for the demonstration plant will provide for less overdesign, requiring higher superficial velocities. Therefore a number of potential problems associated with operation closer to minimum cost design need to be examined. Adequate temperature control is needed, which requires information on exothermic/endothermic reactions, heat capacities, heats of formation, and reactor kinetics. The level of solids accumulation also affects design. Excessive accumulation would evidence a need for a solids blowdown system, whereas low concentrations may adversely affect conversion kinetics and fluid dynamics.

1. Solids in the reaction system (pilot plant scale)

The effect of solids accumulation vs superficial velocity for a given feed-coal particle size distribution should be explored at the Wilsonville SRC pilot plant because there is a nuclear-level gauge in operation there to measure the solids withdrawal rate. Particle size distributions obtained from particular coals under reaction conditions could be determined at laboratory scale (for example, Auburn University has the

technology in place) and compared with spot samples withdrawn from the Wilsonville reactor.

The catalytic effect of mineral matter buildup on conversion vs solids concentration in the reactor for a particular coal feed should also be determined at Wilsonville because of the in-place solids blowdown system. The effects of low solids concentrations on operability and coking in the reactor should be examined. This effort should consider concerns raised by APCI [SRC-I, Sect. 3.2 (1b)] and P&M [SRC-II, Sect. 3.3 (1d)]. The overall cost would be about \$6000,000.

Heat Exchangers

Recommendations in several other areas also support the heat exchanger area, for example, "Physical Properties" and "Fired Heaters."

1. Correlation of data

Increased emphasis should be placed on correlation of data available at conditions representative of SRC demonstration design heat exchangers from both current and future programs. This effort could be part of the coordination effort discussed under "Physical Properties."

Physical Properties

Although many aspects of the SRC process are being verified at the pilot plants, there are a number of uncertainties regarding the design, scale-up, and operation of individual units. These uncertainties stem mostly from the lack of basic property data on SRC process streams. Most available data outside the pilot plants are at conditions far removed from SRC process design conditions, and correlations to extend the data to this range are either nonexistent or are based on petroleum-derived materials. The direct applicability of petroleum correlations to coal-derived materials has been shown to be inadequate in many cases. Physical property data, if obtained within the relevant time frame, could lead to significant improvements in the design of SRC process units, reduce operational problems, and provide the necessary data base for the development of reliable engineering correlations for scale-up.

1. Coordination of physical and transport data acquisition

There is a strong need for overall coordination of research on physical property measurements in support of coal conversion activities. A multifunctional research location, such as a national laboratory or an energy technology center, should play a coordinating role in this area, primarily because of broad technical expertise in various areas of coal conversion and independence from project bias. Immediate coordination is essential if data are to be made available in time to aid the design and operation of the SRC demonstration plant. The cost would be about \$50,000 per year, excluding the vapor/liquid equilibria coordination project. This effort should be considered in conjunction with similar recommendations for overall coordination and data interpretation in the areas of heat transfer (fired heaters and heat exchangers) and vapor/liquid separations, for a total overall cost of about \$100,000 per year.

Efforts should be directed at specific data needs of the SRC DPP [SRC-I, Sect. 3.2 (2b, 5); SRC-II, Sect. 3.3 (2b, 2d, 2e, 4a, 4b, 4c, 6a, 6b, 6c)]; at coordinating the resources available at Ft. Lewis, Wilsonville, Harmorville, and the other facilities discussed in Sect. A.8, and at continuance of ongoing activities.

2. Heats of vaporization, sublimation, fusion, and reaction

Data on coal-derived liquids from SRC process streams are inadequate. When measured, the data are correlated by use of relations obtained for petroleum liquids. The inadequacies of petroleum-derived correlations for such applications should be identified and corrected, where possible, based on new data from this project. The institutions currently involved in these measurements (e.g., Bartlesville Energy Technology Center and Colorado School of Mines) should play a major role. A funding increase of \$100,000 to \$200,000 per year would be needed to develop early usable data. This

work should deal with the concerns of APCI [SRC-I, Sect. 3.2 (2b, enthalpy measurements; 7)] and P&M [SRC-II, Sect. 3.3 (2b)], which have already received some attention in connection with reactor modeling work.

3. Heat transfer data

Heat transfer/rheology studies should be investigated in small-scale preheaters such as the ones available at Argonne National Laboratory (ANL), ORNL, and Institute of Gas Technology (IGT). This information is important to the SRCDPP and should provide input desired by APCI [SRC-I, Sect. 3.2 (2b)] and P&M [SRC-II, Sect. 3.3 (4b, 6a)]. The cost would be about \$200,000 per year. Such data would supplement the major effort at Ft. Lewis, which is scheduled to start up early in FY 1980.

Heat Exchangers

2. Dissolver exit heat exchanger

A well-instrumented shell-and-tube or double-pipe heat exchanger should be installed on the dissolver outlet stream at the Ft. Lewis pilot plant. The data, if acquired in time, would be relevant to the SRC-I demonstration plant design, in which a heat transfer fluid (such as Dowtherm A) is used to recover heat for use elsewhere in the process. A vertical orientation capable of both upflow and downflow operation appears to be preferable. For a 92.9-m² (1000-ft²) stainless steel unit, the approximate cost of the heat exchanger for the relevant temperature and pressure conditions is estimated at \$55,000. The total cost could be about \$100,000, including installation and operation for several months. This program would principally support SRC-I design; a similar effort has been suggested by P&M on the effluent vapor phase [SRC-II, Sect. 3.3 (4d)], but it seems unlikely that it will provide data required for design under the current schedule. We agree that the P&M program is reasonable, both for support of subsequent demonstration plant operation and for verification of current demonstration plant design assumptions.

Slurry Pumps

Most of the slurry pumping requirements in the SRCDPP can be met with state-of-the-art pumps. Some of these pumps are currently in use in small pilot plants, and more are being installed in the large EDS and H-Coal pilot plants that will begin operation in early 1980. Operation in the pilot plants should be monitored to determine whether or not these pumps perform as well as expected.

1. Pump evaluation

We recommended that testing of state-of-the-art centrifugal slurry pumps be expedited, particularly at Ft. Lewis. Testing will require about \$100,000 in FY 1980. We agree with the recommendation by P&M [SRC-II, Sect. 3.3 (3b)] that a gasifier charge pump should be tested at Ft. Lewis, with the concurrent testing of a slurry pump for the vacuum-tower bottoms. Engineering of a vacuum-bottoms test loop at Ft. Lewis is under way.

Fired Heaters for Slurries

3. Flow-regime verification

Verification of two-phase flow-regime maps using more recent and more fundamental two-phase flow-regime correlations is needed — especially with respect to tube inside diameter, flow angle (from the horizontal), and physical properties. Verification would determine whether surface tension is needed for such correlations. The verification of flow-regime maps for fired heaters and slurry-phase heat exchangers is roughly estimated to require about \$200,000 per year for two years and would be most effectively conducted by an expert in the field. A. E. Dukler, at the University of Houston, is the type of investigator envisioned because of his many years of work in the field. P&M has proposed flow-regime studies at Harmarville, and APCI is also interested in such information.

Solids/Liquid Separations

2. Drying techniques project

The program at Wilsonville to test an 0.46- to 0.65-m² (5- to 7-ft²) skid-mounted candle or leaf filter, using either steam or hot N₂ as a stripping agent to dry the filter cake, should utilize both steam and hot N₂ in a candle filter. Both alternatives to vacuum drying thus can be assessed simultaneously with the assessment of the technical feasibility of a new type of filter. Although screen blinding may not be affected by the drying method, the data will still be useful in determining the optimum technical and economic drying technique. APCI also supports this and other filtration work at Wilsonville.

Valves

Continued pilot plant operation, up to the time of finalization of specifications for demonstration plant equipment, will make possible continued improvement in small valves. If suspected improvements in scale-up are assumed, reasonable standards of acceptability established, and sufficient redundancy incorporated in the demonstration plant, the urgency for valve research and development will be somewhat reduced. Even with this optimistic approach, valves will be so numerous and expensive in the proposed commercial-scale operation that investments in improving reliability will ultimately be reflected in reduced maintenance costs and possibly increased plant availability. In light of currently proposed operating schedules for the larger H-Coal and EDS liquefaction pilot plants, it might be reasonable to postpone decisions on valves for demonstration projects until early results of these pilot plant projects become available.

1. Valve evaluation

Communication should be maintained among DOE-funded projects to ensure that the latest equipment developments are made known to decision makers as they occur and that promising industrial valve designs are tested under large-scale conditions

resembling those of the SRC process. The H-Coal and EDS pilot plant programs provide the next level of assurance beyond current SRC pilot plants; the component sizes in these projects are of adequate scale to clearly indicate trends for the future. Evaluation of state-of-the-art and prototype valves requires the following levels of funding (approximately): \$50,000 in FY 1980, \$100,000 in FY 1981, \$200,000 FY 1982, and \$100,000 in FY 1983. Such testing has also been recommended by P&M [SRC-II, Sect. 3.3 (7c)] at Ft. Lewis under the current contract.

Solids/Liquid Separations

3. Filtration support studies

A laboratory-scale screening project pertaining to materials, screen weaves, and related topics should be instituted in support of the Wilsonville studies. Also, the construction of large leaves should be evaluated because warping of the large U.S. brand filter leaves was encountered during tests at Wilsonville. A filter or screen manufacturer with a well-equipped laboratory could perform these tests within a year, at a cost of about \$100,000. Drain/fill time and other cyclic operational characteristics that can increase total filtration rates should also be examined at laboratory scale; the cost is estimated at about \$100,000.

Fired Heaters for Slurries

4. Erosion studies

Acquisition of erosion-rate data for coal/oil slurries, as exemplified by A. V. Levy's program at Lawrence Berkeley Laboratory, should be continued. Data acquisition should include measurements for longer radius return bends, which was also recommended by APCI. About \$150,000 per year through CY 1982 is indicated. Continued work, particularly at Ft. Lewis, is also recommended by APCI [SRC-I, Sect. 3.2 (2c, 6)] and P&M [SRC-II, Sect. 3.3 (4b, 7e)].

Slurry Flowmeters

1. Recommendations for current projects

We strongly recommend continuation of the efforts to develop slurry-metering instrumentation at the Ft. Lewis and Wilsonville pilot plants. This is consistent with P&M's recommendation for continued work at Ft. Lewis [SRC-II, Sect. 3.3 (7d)] using a skid-mounted slurry flowmeter test stand. Instruments that show promise in the small-scale test stands should be tested at full scale in the pilot plants. Provisions should also be made for testing these instruments in large-diameter pipes and at reasonable flow rates. Current efforts in the pilot plants have included testing of conventional meters such as orifices and venturis. Additional studies may be initiated for developing and testing the following advanced types of meters in the pilot plants:

- a. Acoustic/ultrasonic
- b. Capacitive cross-correlation
- c. Gamma-ray cross-correlation
- d. Neutron activation

The installation of an ANL-developed ultrasonic flowmeter in the feed line to the new slurry fired heater at Ft. Lewis, scheduled for September 1979, is consistent with our recommendations.

2. Extension and/or redirection of current projects

Current efforts to develop advanced instrumentation, for example, at ANL, should be continued in and beyond FY 1980. However, a significant fraction of the efforts should be redirected toward developing slurry-metering instruments for SRC application. Specific attention should be paid to the following:

- a. High-temperature operation, to 700 K (800 °K)
- b. High-pressure operation, to 13.8 MPa (2000 psi)
- c. High solids concentrations, to 50 wt % solids
- d. Effects of increased pipe diameter on the measurement of slurry flow rates

In addition, testing of promising candidate meters developed at ANL and elsewhere should continue in the pilot plant loops under actual SRC operating conditions on a schedule accelerated to meet start-up requirements for the demonstration plants.

The cost of continuing the instrumentation program for slurry meters is estimated at about \$600,000 per year for three years. Prototype instruments will probably cost about \$200,000 each. Therefore, a projected cost per year would be about \$800,000, assuming that one prototype instrument is purchased each year.

Fired Heaters for Slurries

5. Slurry/gas mixers

Procedures should be established for design of effective slurry/gas mixers. This area has not been suitably covered but could affect significantly the initial flow pattern development in the fired heater. A one-year program should be sufficient for this study; the cost would be about \$150,000.

Heat Exchangers

3. Flow-regime verification

Additional verification of existing multiphase flow-regime maps for vertical and horizontal tube flow is needed. (This was discussed under "Fired Heaters for Slurries," along with approximate cost.)

Physical Properties

4. Physical and transport properties

Physical property data (viscosity, density, thermal conductivity, specific heat) of coal slurries are inadequate for design and scale-up of SRC units, particularly in the front end of the process. These properties should be measured under process conditions for a range of operating parameters. Institutions other than pilot plants should also be involved

in such measurements because the data obtained from pilot plants are not always adequate for developing correlations, primarily because of interaction among system parameters. Thus the effort on physical property measurements on SRC materials in flow systems should immediately be extended and redirected to facilitate demonstration plant design and operation. Although the work in pilot plants is producing some data, the ability to obtain data for developing correlations is limited by the relative inflexibility of pilot plants as compared with institutions conducting bench-scale experiments; nevertheless, pilot plants should play an important role in testing the data and correlations from the bench-scale efforts. A three-year program at \$400,000 per year, excluding efforts funded separately in the pilot plants, would be effective. Many of the recommendations by the SRC groups could be considered under this program [SRC-I, Sect. 3.2 (2b, 5); SRC-II, Sect. 3.3 (2b, 2d, 4a, 4b, 5a, 6a)] to supplement current efforts at the pilot plants.

4.2 SRC Technology

Activities that would potentially assist in demonstration plant operations and commercialization efforts, but which lack near-term design impact, are identified as optional activities in this section. Follow up activities in many of the areas identified in Sect. 4.1 also qualify as optional efforts. If funding beyond FY 1980 is suggested for an activity in Sect. 4.1, this activity is assumed to carry over into the optional category. We have not ranked each activity individually in this section; a qualitative order of importance is indicated by the ranking shown within each problem area.

Fired Heaters for Slurries

6. Heat transfer data correlation

Laminar and turbulent-slurry heat transfer data for homogeneous velocities to ~ 10 m/s (~ 30 fps) should be compared with available correlations for power-law fluids

(e.g., Charm and Merrill for laminar flow, Metzner and Friend for turbulent flow) in a manner similar to IGT's efforts in the Coal Conversion Systems Technical Data Book Project. Supplemental data from heat transfer studies previously recommended would support this analytical effort, because the current data base is unlikely to be sufficient. This correlation of data would involve a two-year program costing about \$200,000 per year.

Vapor/Liquid and Liquid/Liquid Separations

2. Emulsion studies

Very stable emulsions are formed in the vapor/liquid separators at Ft. Lewis because of the presence of water, surfactants, and coal liquids of similar densities. The problem is primarily with oil-in-water emulsions, which because of high emulsion stability, resist separation in the effluent treatment facilities. A similar situation exists for the Harmarville Process Development Unit (PDU); the stability of the PDU emulsions is less severe because Harmarville does not use the same system of direct water quench to cool reactor effluent as does the Ft. Lewis operation. Instead, a much smaller amount of water is injected to flush out salts from exchanger tubes. Correlations currently available are inadequate to determine the extent of emulsion problems likely under demonstration plant conditions. To experimentally assess this problem, a project should be initiated to (1) prevent emulsion formation by, for example, proper selection of flash temperatures and pressures or (2) break such emulsions. The first project has to some extent been undertaken at the SRC Harmarville PDU. The second project should be performed at a university with expertise in emulsions, such as the Illinois Institute of Technology or West Virginia University. Additional funding needed to pursue the first project at Harmarville would be about \$100,000 per year. A university

effort at \$75,000 per year for two years should be adequate to perform the second task. P&M has been performing similar work at Harmarville [SRC-II, Sect. 3.3 (2f)] under current contract funding.

3. Reactor flow vapor/liquid equilibria

A program to form the basis for vapor liquid equilibria research for the developing coal conversion industry, and specifically for the SRC demonstration plant, should be considered. Ongoing research (described in Sect. A.4) in the feed-pot flow, batch, and classical batch systems should be coordinated to rapidly produce SRC data. The second facet of the proposed work is the experimental determination of K-values in a reactor flow vapor/liquid equilibrium system (i.e., the coal liquid is generated in situ). Equipment components in this system include (1) a liquid or coal/solvent slurry feed tank and delivery system, (2) a gas supply system, (3) a reactor/dissolver, (4) vapor/liquid separators, (5) sample receivers, (6) on-line analytical equipment, and (7) analytical equipment for support purposes. Important advantages of the reactor flow system are minimization of cracking and repolymerization, plus reduction of sampling and aging effects caused by taking a pilot plant sample, cooling it, and then reheating it at the experimental site.

We recommend that the proposed experiment be considered for a national laboratory or an energy technology center. The reason for this is threefold. First, national laboratories and energy technology centers encompass large and diversified areas of experimental expertise. Second, to form a coherent base for an experimental program it is beneficial to have a long-term continuity in manpower that universities often do not have. Third, sound quantitative data can best be obtained from small-scale experiments in which conditions can be precisely controlled, rather than in pilot plants. However, pilot plants should be used to verify any correlations obtained.

P&M is doing this now [SRC-II, Sect. 3.3 (2c)] by checking current design correlations against Harmarville data. APCI has performed similar checks against Wilsonville data.

The funding for this program should be about \$750,000 per year for three to five years.

Solids/Liquid Separations

4. Deashing project

Experiments on both the Lummus and Kerr-McGee deashing processes indicate that stability of the feed is imperative. Currently, there is a very limited program to study vacuum-bottoms stability to obtain data suitable for scale-up. We proposed, therefore, that a new program be initiated to investigate vacuum distillation of SRC reactor effluent. This project would correlate feed and yield parameters; these parameters would then be related to the parameters to which the deashing units are known to be sensitive (e.g., preasphaltene content).

The project should also:

- a. Determine the effects of feed properties, residence time, and solvent vaporization on coking in the vacuum preheater
- b. Determine the effects of feed properties, flash-zone residence time, pressure, and temperature on solvent recovery, thermal cracking, repolymerization, bottoms viscosity, and bottoms stability

APCI's recommendation [SRC-I, Sect. 3.2 (3a)] represents a near-term approach to acquisition of some of this information. The project also addresses other concerns of APCI [SRC-I, Sect. 3.2 (7)] and P&M [SRC-II, Sect. 3.3 (2b, 2d)], some of which have been empirically addressed in sufficient detail to answer the near-term questions. The program would involve a three-year effort costing about \$500,000 per year. The necessary equipment would include a feed source, gas/liquid

separator, vacuum preheater, vacuum column, and various analytical devices; the scoping cost assumes that an experimental rig, which could be used with only minor modifications, is already in place. Examples of institutions with at least some of the appropriate equipment in place include ANL, Pittsburgh Energy Technology Center (PETC), and several other groups involved in coal liquefaction, such as P&M in Merriam. A new installation would delay this effort by at least one year and would entail capital equipment costs of greater than \$1 million.

Reactors/Dissolvers

2. Solids in the reaction system (bench scale)

The cooperative efforts at Sandia, PETC, Pennsylvania State and Gulf (discussed in Sect. A.1.4) should be continued. If possible, this program should use SRC materials and be coordinated with the major pilot plant effort outlined previously.

At high solids levels, coking reactions are facilitated by long residence times, whereas at low solids concentrations, solvent quality (a measure of the solvent's capability as a hydrogen donor) is an important factor in coking. The presence of a small amount of solids retards coking under the latter conditions, and the amount, type, and mechanisms for this retardation are factors important to reactor operations. The SRC processes may be capable of operating with very low solids accumulation, thereby allowing for a smaller (and less expensive) reactor; however, coking and operability under these conditions must be determined. A new project should investigate the effect of solids on operability and coking in the SRC reaction system. Preliminary work could be done at a university equipped for this type of investigation (Pennsylvania State, for example, has the expertise and equipment). The preliminary work could then be verified at Wilsonville or Ft. Lewis. The cost for the bench-scale efforts would be about \$200,000 per year for two years.

3. Reaction mechanisms

The endothermic/exothermic reactions in the dissolver determine the heat load, the reactor control characteristics, and the distribution of components in the effluent (which determines the feed to the rest of the process); the endothermic and exothermic reactions also have a major effect on solvent quality. Continuing efforts (e.g., at Auburn University) should be directed toward reaction mechanisms using demonstration plant feeds and operating conditions when possible. The SRC pilot plants and PDUs should increase their efforts in this area. The additional expense would be about \$300,000 per year. The program should also seek to provide the kinetic information desired by APCI [SRC-I, Sect. 3.2 (1c, 2a)] and P&M [SRC-II, Sect. 3.3 (1a, 1b)], although much of this work has already been accomplished in conjunction with reactor and yield modeling studies.

4. Fluid dynamics vs residence time

Studies to determine fluid dynamics and property changes with residence time should be funded at an institution with in-place capabilities. ORNL's physical properties loop, which determines rheological and transport properties at process conditions, could be adapted for such studies.

Slurry Pumps

2. Prototype pump testing

Currently available pumps as well as similar pumps that may become available from other suppliers should be purchased, installed, run, and evaluated, principally at Ft. Lewis. When other manufacturers offer pumps designed for high-pressure hot-slurry service, prototypes of these pumps should be bought and tested. Funding at a level of \$250,000 per year should provide for the procurement and installation of two pumping units. Continuation at this level of funding over the next three years is desirable. The pumps acquired would be installed as

replacements in existing pilot plants, and test and evaluation costs would be absorbed in the pilot plant operating and maintenance costs.

Oxygen Compressors

1. Manufacturers of centrifugal oxygen compressors seem confident that they can supply the equipment needed, although it may be beyond the present state of the art. A test to confirm operation of three-stage centrifugal compressors has been proposed and is in the process of being arranged. A compressor manufacturer, Mannesmann Demag Corporation, has offered to supply the high-pressure section of a centrifugal oxygen compressor to DOE, and testing of this compressor at the Engineering Technology Center (operated by the Atomics International Division of Rockwell International) is proposed. Funding for the test has been allocated, and a contract to do the work is being negotiated by the San Francisco Operations Office of DOE. It is expected to take at least a year to manufacture the compressor and to modify the test loop to accommodate it; thus, testing could not begin before the late summer of 1980. The overall program is expected to require 18 months. Funding for the proposed program is \$1.8 million. The testing program should be expedited if the demonstration design continues to require high-pressure centrifugal compression. At best, however, even preliminary results would not be available before the late summer of 1980.

Two design options are available if this program is not funded:

- a. Use state-of-the-art design for high-pressure oxygen compression, represented by one- or two-stage centrifugal compression followed by reciprocating compressors, thereby incurring additional capital and operating expenses (APCI approach in Phase 0 design)
- b. Reduce the oxygen delivery pressure at design conditions to the maximum level currently proven in two-stage centrifugal oxygen compressors [about 6.5 MPa (940 psia)], thereby

potentially increasing other associated expenses (approach recommended to P&M for design in Phase I)

Neither of these options carries the technical risk of designing for three-stage centrifugal oxygen compression without prototype testing.

Slurry Mixing

2. Mixing device project

In addition to the pug mill, inductors, and vortex mixer studies at Ft. Lewis, a new bench-scale project should be instituted to examine a number of commercial-type mixing devices. This project could be accomplished most expeditiously at the industrial laboratory of a mixing technology vendor. The estimated funding level is \$125,000 for one year.

Valves

2. Valve modeling

Development, by the demonstration plant designers or the valve industry, of a computer model capable of predicting flow fields within various geometries would be a useful tool during the early design stages of valves and many other fluid-handling components. Such a model could also serve to screen prototype designs and to aid in sizing valves. Cold flow-model testing and visualization of flow could provide valuable supplemental information as reinforcement to, or as a check on, analytical determinations. Such studies should cost about \$100,000 to \$150,000 per year over the next three years.

4.3 Coal Liquefaction Technology

Along with many of the previously recommended activities that are also generally supportive of coal liquefaction technology, the following efforts would be useful. Note that previous recommendations tied to long-term funding estimates could be included in this category in later

years. A tentative list of priorities is provided by the ranking of the problem areas in this section.

Fired Heaters for Slurries

7. Flow effects of bends

An investigation of flow/heat transfer in long-radius heated return bends and the effects of bend flow on the downstream straight-section flow pattern is needed. A two-year, \$200,000 per year program is indicated.

Reactors/Dissolvers

5. Short-residence-time studies

Short-residence-time studies should be funded because success with this process configuration could entirely eliminate the dissolver (all reactions to heavy product can be accomplished in the preheater). Installations with capabilities in this area include Sandia, Kerr-McGee and Wilsonville.

6. Reactor mixing studies

Vapor/liquid/solid distribution in the reactor is of importance to modeling efforts. Dispersion studies on three-phase mixing in bubble columns under reaction conditions would be beneficial, particularly in view of the more stringent operating conditions proposed for the demonstration reactor. A three-year program at a national laboratory, energy technology center, or university could perform such a task for \$200,000 per year.

Heat Exchangers

4. Laminar-flow heat transfer

A modest effort to extend small-scale work on enhancing laminar-flow heat transfer rates to industrial-scale equipment conditions seems warranted. This work would apply primarily to tube side flows of relatively clean but viscous liquids such as recycle solvent coolers. A one-year program might

reasonably be expected to cost about \$100,000 to \$150,000

5. Evaluation of rod-baffle exchangers

Tests should be conducted to determine shell-side fouling rates in rod-baffle exchangers relative to the rate of fouling in standard segmentally baffled units. Indirect (field) data indicate that such rates are lower in rod-baffle exchangers. Areas of application would encompass all dirty shell-side flows with the exception of concentrated liquid/solid mixtures. Such dirty flows will be common in coal liquefaction plants. Two small heat exchangers — one with standard segmental baffles and the other with rod baffles, each with about 15 m² (160 ft²) of stainless steel surface area and designed with a temperature-pressure tolerance of 530 K/2.2 MPa (500°F/300 psig) — would cost about \$25,000. A one-year test program conducted, for example, by Phillips Petroleum Co. (Bartlesville, Okla.) or by Heat Transfer Research Institute (Alhambra, Calif.) might involve a total cost (including the exchangers) of about \$125,000.

Slurry Flowmeters

3. Recommendation for new projects

A facility is needed for testing slurry flowmeters under actual SRC plant conditions and at a scale that would provide realistic information for the demonstration plants. The largest systems that will be available for such tests will be available for such tests will be the H-Coal pilot plant at Catlettsburg, Kentucky [at up to 540 Mg/d (600 tons/day) of coal], and the EDS pilot plant at Baytown, Texas [at 225 Mg/d (250 tons/d) of coal]. These tests could involve \$800,000 initially in FY 1981 and \$120,000 per year for two subsequent years.

If testing cannot be accomplished in the H-Coal pilot plant (preferably operating in the boiler fuel mode) or the EDS pilot plant, we recommend development of a loop with adequate capacity for testing slurry meters in pipes of

sufficient diameter to simulate demonstration plant operations. Coal liquids from the SRC-I and SRC-II plants could be circulated in this loop to simulate plant operations. The cost of this loop would be high. For example, the cost of a slurry loop for testing pumps over a range of 0.03 to 0.15 m³/s (500 to 2500 gpm) was estimated to be \$23 million in 1976. Therefore, it is recommended that efforts be made to use pilot plants such as the H-Coal plant for testing large slurry-metering instruments.

Slurry Mixing

3. Mixing data project

We recommend a new project to obtain mixing data on mixtures of SRC-process recycle solvent, SRC-slurry recycle solvent, and demonstration design coals. Initial efforts should be directed toward obtaining data on:

- a. Slurry properties for 841- μ m (20-mesh) and larger coal sizes
- b. Viscosity changes with temperature
- c. Viscosity changes with residence time
- d. Gelling tendencies as a function of time and temperature

This effort is similar to that recommended by APCI [SRC-I, Sect. 3.2 (5)] to establish the maximum operating temperature for the mixing system. Questions affecting demonstration operations that could be answered within two to three years are:

- a. Effects of changes in feed coal and solvent characteristics
- b. Effects of solvent/coal ratio
- c. Effects of agitation rate

The project could be done by an industrial, national, or energy technology center laboratory. The cost would be about \$200,000 per year for three years.

Solids/Liquid Separations

5. Rotary drum filter project

A program to test the rotary pressure precoat filter (designed by Johns-Manville) at Ft. Lewis should be considered by DOE. The filter is installed and could be operated cost effectively; it could be operated alternatively with the Lummus deashing unit when the plant operates in the SRC-I mode.

REFERENCES

1. Letter Contract No. ET-78-C-01-3054, Southern Company Services, Inc., and the U.S. Department of Energy, July 10, 1978.
2. Letter Contract No. ET-78-C-01-3055, Pittsburg & Midway Coal Mining Co. and U.S. Department of Energy, July 10, 1978.
3. Air Products and Chemicals, Inc., Allentown, Pa., *Phase I Bridging Tasks*, DOE Contract No. ET-78-C-01-3054, submitted to DOE/ORO, Mar. 7, 1979.
4. Pittsburg & Midway Coal Mining Co., Denver, Colo., *SRC-II Demonstration Project, Technical/Cost Proposal Phase Zero & Bridging Tasks*, DOE Contract No. ET-78-C-01-3055, submitted to DOE/ORO, Apr. 11, 1979.

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Appendix

DETAILED ASSESSMENT OF R&D PROBLEM AREAS RELEVANT TO
SOLVENT REFINED COAL DEMONSTRATION PLANTS PROJECTS

- A.1 Reactors/Dissolvers
- A.2 Fired Heaters for Slurries
- A.3 Solids/Liquid Separations
- A.4 Vapor/Liquid and Liquid/Liquid Separations
- A.5 Gasification of SRC Residues
- A.6 Slurry Mixing
- A.7 Heat Exchangers
- A.8 Physical Properties
- A.9 Fluid-Handling Components (including slurry pumps, oxygen compressors, valves, and slurry flowmeters)

Purpose of Conclusions and Recommendations for
the Institutions Visited

The institutions visited were diverse in their programs, expertise, and facilities. Their funding structure and program guidance were derived from many sources, including a number of divisions and departments within the U.S. Department of Energy (DOE). They were chosen because it was believed that they had something to offer in support of the Solvent Refined Coal (SRC) Demonstration Plants Projects (SRCDPP), either now or in the future. Consequently, conclusions and recommendations concerning a given program made after a visit by the assessment team are made from the viewpoint of the SRCDPP and are intended solely to inform the SRC-DOE group of changes in those programs that could lend more productive support to the SRCDPP. The program managers responsible for these individual programs have other goals that are equally valid. Only to the extent that they wish to consider redirection toward more SRCDPP support will this information be useful to them.

A.1 Reactors/Dissolvers

A.1.1 Introduction

The overdesign of dissolvers is not detrimental to their performance, as has been adequately demonstrated in the pilot plants. However, superficial velocities much greater than those in the pilot plants are planned in the Solvent Refined Coal (SRC) demonstration plant designs. Higher velocities result in significant savings but require better reactor control. Better reactor control requires significant knowledge of the characteristics of the reaction itself. Basically, very little is known about the mechanisms of coal liquefaction, such as kinetics, change of physical and thermal properties during reactions, and thermodynamics. A further complication is the effect of ash buildup on reactor operation and kinetics; control of ash buildup has not been satisfactorily demonstrated at demonstration design conditions. The difference in cost between an overdesigned unit similar to the pilot plant reactors (in which performance has been demonstrated) and a minimum-cost design (in which performance has not been demonstrated) is significant. Funding to resolve some of these problems can produce a more cost-effective design for the demonstration plants.

A.1.2 Relevant research and development (R&D) programs

Questions concerning the extent of gas mixing, slugging, degree of backmixing, and amount of solids buildup in upflow reactors are under investigation. Uncertainties regarding the effects of these phenomena on reaction kinetics and yield structures would result in the design of larger than necessary reactor sizes and in increased costs. Kinetic and fluid dynamic data at conditions closer to the proposed design would help to verify operability at the planned higher superficial gas/liquid velocities.

SRC-I pilot plant. The Wilsonville, Alabama, SRC pilot plant generally operated at only one-half the dissolver volume because of overdesign. Using the Kerr-McGee Critical Solvent Deashing (CSD) process, operation at half volume resulted in only 18% recovery of SRC product at a space velocity

of $0.22 \text{ kg/s}\cdot\text{m}^3$ ($50 \text{ lb/hr}\cdot\text{ft}^3$). Operation at higher space velocities to reduce dissolver size (and costs) is desirable, but product loss should be minimized. Wilsonville will continue to experiment with space velocity and reactor volume vs conversion and should produce some useful data. Raising the pressure from 11.8 to 14.6 MPa (1700 to 2100 psig) at 75% reactor volume results in a superficial space velocity of $0.17 \text{ kg/s}\cdot\text{m}^3$ ($38 \text{ lb/hr}\cdot\text{ft}^3$) and yields 84% recovery of SRC product from the Kerr-McGee CSD unit.

Other factors being studied at Wilsonville include solids accumulation in the reactor and its effect on operations. A solids-withdrawal system installed at Wilsonville had operated continuously for about two months as of June 1979. The solids are withdrawn through a valve programmed to open into a holding tank according to a preset time sequence. Thus the solids accumulation rate in the reactor is relatively constant. A nuclear density gauge monitors the holding tank level and dumps the contents periodically. The system has worked well.

The catalytic effect of solids on conversion to product has been shown to be particularly important for SRC-II. Some Wilsonville data indicate that the additional conversion is to liquids (benzene solubles) and not solvent refined coal. Because the solids have little effect on solvent refined coal production in the SRC-I process, maintenance of a minimum solids inventory in the reactor appears desirable (the accumulation of solids in the Wilsonville operation increases the required reactor size). Although a minimum solids inventory is desirable at low space velocities, a minimum inventory is not necessarily desirable at high space velocities. A small quantity of solids has an effect on the solvent quality that seems to retard coking at higher space velocities. This aspect needs to be explored further at Wilsonville and in the laboratory.

A current trend at Wilsonville is toward development of a two-stage liquefaction process that imposes very mild conversion conditions in the reactor (one mode eliminates the need for the reactor entirely). This type of process, currently funded by the Electric Power Research Institute (EPRI), sometimes has been called the SRC-1/2 process. Use of the pilot plant in the SRC-1/2 program process reduces its usefulness to the SRC-I process as currently designed.

SRC-II pilot plant. Accumulation of solids is being studied at Ft. Lewis as well. To determine solids concentrations, samples are withdrawn from various locations within the reactor. Conversion and solids accumulation are being related to coal feed size. However, Ft. Lewis does not have a solids-withdrawal system. In the SRC-II mode, samples at reactor exit and at an intermediate position have measured solids content equal to about 10% of the content at the reactor inlet, if 74- μm (200-M) coal is used. Conversely, 74- μm (200-M) feed coal in the SRC-I mode did show measurable solids accumulation. The effect of 595- μm (30-M) feed coal on solids accumulation in the SRC-II mode has not been determined yet. The results of a recent study in which a radioactive-tracer technique was used to measure solids inventory were inconclusive.

Other related studies. Gulf researchers at Harnmarville, Pennsylvania, are studying dissolver hydrodynamics in glass models. They are also developing empirical models of hydrodynamics and kinetics.

The Pittsburgh Energy Technology Center (PETC) has conducted several studies of the coal liquefaction process that, if redirected, might be useful to demonstration plant programs. Examples are studies of the mechanisms of coal liquefaction and studies of certain catalysts. Currently, the SRC effort at PETC is minimal.

Some other programs potentially applicable to SRC demonstration plants are discussed in Sect. A.1.4.

A.1.3 Conclusions and recommendations

A number of potential problems associated with plant operation at superficial velocities lower than those used for the demonstration plant design need to be examined. Because temperature must be controlled, information on exothermic/endothermic reactions, heat capacities, heats of formation, and reactor kinetics is essential. The level of solids accumulation directly affects design. Excessive solids accumulation indicates a need for a solids blowdown system. Too little accumulation may adversely affect conversion kinetics, fluid dynamics, etc. Solids accumulation depends on the velocity in the reactor, which is related to residence time and to the kinetics of the reaction. The catalytic effect of the accumulated solids also affects reaction kinetics and has a consequent impact on plant design parameters.

A summary of conclusions and recommendations follows:

1. Data indicate that dissolver design is adequate for operation at lower superficial velocities.
2. Operation at higher superficial velocities than previously demonstrated will require additional R&D in the following areas:
  - a. Solids accumulation vs superficial velocities for specific feed-coal particle size distributions;
  - b. Catalytic effects on conversion vs solids concentrations for particular particle size distributions and coal feeds;
  - c. Thermodynamic and kinetic properties, particularly heats of reaction and yield distributions;
  - d. Gas-liquid-solids mixing in bubble columns.

These data should be accumulated from pilot plant operations to the extent possible, particularly so that data from concurrent laboratory or bench-scale studies can be verified.

Laboratory or bench-scale supporting studies for the first two items could result in greater versatility in the selection of pilot plant experiments and in accompanying savings. Much of the work in fluid dynamics has already been initiated and, in some cases, completed, in the laboratories of the industrial participants. Laboratory-scale studies of thermodynamic and kinetic properties and mixing could be designed to ensure relevance to plant-scale operations by using a flow system and by avoiding wall effects, particularly in studies on mixing.

We recommend or suggest that:

1. The Wilsonville SRC pilot plant be used to explore the effect of solids accumulation vs superficial velocity for given particle size distributions, because the facility is equipped with an operable nuclear level gauge. Particle size distributions of coals determined in the laboratory under reaction conditions could be compared with those of spot samples withdrawn from the Wilsonville reactor (Auburn University is equipped to perform this comparison). The applicability of the Ft. Lewis reactor for this study, however, is believed to be limited.

2. The catalytic effect of mineral matter on conversion vs solids concentration in the reactor for a particular coal feed be studied at Wilsonville in conjunction with the solids accumulation study (because of the in-place solids blowdown system). Some of this work could be done at Ft. Lewis, where inlet, outlet, and one intermediate sample can be withdrawn, but solids-level control system is available. Use of Harmarville P-99 to check on major effects is another option for SRC-II support.
3. The cooperative effort discussed in Sect. A.1.4 (involving Sandia, PETC, Pennsylvania State, and Gulf) be continued at the current level of funding. The cooperative work should be tied to a major pilot plant scale effort as outlined above.
4. The effect on coking of solids in the reactor should be explored. At high solids levels, coking reactions seem to be favored; at low concentrations the solvent quality (a measure of hydrogen donor capability) seems to be the controlling factor in coking. The presence of a small amount of solids may retard coking under the latter conditions, and the amount, type, and mechanisms for this retardation are factors important to reactor operations. The SRC process could operate at a very low solids accumulation, which might allow for a smaller (and less expensive) reactor, but coking and operability under these conditions must be determined. Preliminary work should be done at Pennsylvania State (or at an institution equivalent in expertise and equipment) and verified at Wilsonville or Ft. Lewis.
5. There are not enough kinetic rate and heat-of-reaction data to permit a nonempirical approach to coal dissolution. The endothermic/exothermic reactions in the dissolver determine the heat load, reactor control characteristics, and component distributions in the effluent (which determines the feed to the rest of the process) and have a major effect on solvent quality. The efforts at Auburn and Pennsylvania State University should be continued and directed toward reaction mechanisms that have demonstration plant application. These studies should concentrate on practical relationships applicable to

demonstration design to supplement the empirical modeling already under way by industrial participants. Another institution experienced in reactions of heavy organic systems (e.g., the University of California at Berkeley) should be funded for more fundamental, basic engineering studies to increase the understanding of coal liquefaction reactions.

6. Studies to determine fluid dynamics and property changes with residence time be funded. The Oak Ridge National Laboratory's (ORNL) physical properties loop, which is producing rheological and transport properties data at process conditions, could be adapted for these determinations. Investigation of property changes with residence time could supplement empirical, broad-scale evidence obtained from pilot plants.
7. Short-residence-time studies be funded as long-range research. Success with this process configuration would entirely eliminate the dissolver because all reactions to heavy product can be accomplished in the preheater. Sandia, Kerr-McGee, and Wilsonville are capable of this type of study.
8. Vapor/liquid distribution in the reactor is also an area of concern. Dispersion studies on three-phase mixing in bubble columns under reaction conditions would be beneficial long-range research.

A.1.4. R&D efforts listed by institution

Category: Reactors/Dissolvers

Institution: Auburn University

Funding agency: U.S. Department of Energy (DOE), \$718,000 for 3 years;
EPRI, \$40,000 per year.

Principal investigators: J. A. Guin and R. A. Tarrer, 205/826-4827

Location: Chemical Engineering Department; Auburn, Ala. 36830

Schedule: Current DOE work begun November 1978; EPRI work begun March 1979.

Objectives:

DOE project — To characterize the effects of process conditions, equipment, and nature of raw materials on kinetics, mechanisms, degree of coal dissolution, and heteroatom removal.

EPRI project — To evaluate the feasibility of a two-stage process for low-sulfur solvent refined coal that meets the new point-source standards proposed by the U.S. Environmental Protection Agency (EPA). Included are process reaction conditions, hydrotreatment of recycle streams, and catalytic desulfurization of product.

Accomplishments:

DOE project — This is a continuation of a previous research effort that can claim the following accomplishments:

1. Development of a sulfur removal process for the dissolver (i.e., add iron and other materials to react with the sulfur);
2. Use of Kerr-McGee residue as a catalyst (add to slurry mix tank);
3. Examination of viscosity effects under preheater conditions (autoclave studies);
4. Development of a solvent quality index; ongoing — research that used H-NMR, IR, elemental analysis and mass spectrometry;
5. Development of a Coulter-counter technique for particle size distribution;
6. Development of an x-ray fluorescence technique for residue analyses;

7. Determination of H_2 solubility in creosote oil and recycle solvent at process conditions;
8. Study of the kinetics of desulfurization, hydrogenation, and dissolution [reaction rate constants and Arrhenius parameters using SRC materials (autoclave studies)].

EPRI project — New; too early to report results.

Category: Reactors/Dissolvers

Institution: Pennsylvania State University

Funding agency: DOE

Principal investigator: P. H. Given, 814/865-1662

Location: Department of Material Science and Engineering; University Park, Pa. 16802

Funding: 3/76 to 3/79, \$1,051,000; 3/79 to 3/80, \$282,000.

Objectives:

To reach an understanding of the dependence of the various aspects of liquefaction behavior on coal characteristics and to express this understanding quantitatively for prediction of coal reaction behavior.

To establish a collection of coal samples having an adequate range of all significant properties (129 different coals).

To determine the liquefaction behavior of subsets of the above samples under catalytic and noncatalytic conditions, but with emphasis on naturally occurring catalysts.

To develop methods to determine the mechanisms of coal liquefaction, organic structures in coals, and the distribution of minerals in coals.

Accomplishments:

1. Samples of 111 well-characterized bituminous and subbituminous coals from three major geological provinces have been assembled, along with 18 lignites, for use in this project.
2. A versatile liquefaction reactor system designed around tubing bombs was built and commissioned in late 1976.
3. The liquefaction reactor system was used in determining duplicate conversions of coal in coal-tetralin mixtures for 104 coals. Detailed statistical analyses, involving analysis of principal components, cluster analysis, multiple regression analyses of conversion data, and 14 coal properties, have been completed.
4. Data from 47 runs on 27 coals in the Gulf continuous flow reactor have been obtained. The data include conversion, yield of vacuum distillable material, saturate-aromatic-aliphatic analysis (SARA), gas yields, gas composition, and material balances.

5. A start has been made with the comparison of behavior of coals in batch reactors (a) when hydrogen is added and (b) when hydrogen and a coal mineral or mineral mixture is added. A study of the effect of agitation rate on conversion when these conditions prevail in batch reactors was begun.
6. Phenolic hydroxyl contents of 52 coals have been determined and statistical analyses have been made of the relation of the data to (a) other coal properties and (b) conversion in liquefaction.
7. The magnitude of the biases (as a result of oxidation of forms of sulfur) in mineral matter content, determined by low-temperature ashing, has been explored for 20 coals.
8. The nature of the primary separation of coal liquids, achieved on a molecular sieve gel, has been investigated as a function of residence time and temperature. Elemental analyses have produced evidence of degradation reactions at the longer residence times.
9. A complex mixture of by-products formed from tetralin under liquefaction conditions has been analyzed by gas chromatography-mass spectrometry.
10. The loss of total oxygen and oxygen as OH and C=O has been monitored in the early stages of coal liquefaction (at five temperatures and five residence times) with conclusions that bear on the mechanisms of coal conversion.
11. Conversions of three coals with tetralin to pyridine-soluble materials, plus gases, have been performed at five residence times and five temperatures [610 to 670 K (340 to 400°C)]. A new theoretical analysis of the kinetics of the conversion of mixed starting materials (formed by a number of parallel reactions) to aggregates of the products has been developed and applied to experimental data to derive activation energies. The analysis has also been applied to literature, and data have been extrapolated to demonstrate the utility of the analysis in testing mechanistic models for coal liquefaction in batch and continuous flow reactors.
12. Detailed petrographic studies have been made of the residues from liquefaction of one type of coal with tetralin at two temperatures and eight residence times and of two other coals to better understand agglomeration phenomena during liquefaction and to characterize the behavior of different macerals (fragments of plant debris in coal).
13. Measurements of the extent and rates of adsorption of six organic adsorbates (including methanol, benzene and tetralin) have been made on ten coals at three temperatures - 303, 323, and 343 K (30, 50, and 70°C). The degree of swelling by the organic molecules has also been studied and the extent of absorption investigated. The penetration of donor solvent into the interior of

coal particles, which is of vital importance in determining product selectivity in coal liquefaction, has been carefully analyzed in the light of the data.

14. A reinvestigation of the products of previously established oxidative reactions followed by reduction of coals has, to some extent, confirmed and extended previous findings of chemical fossils of lignin in coals. Benzene/ethanol extracts of the coals were also studied.
15. Heats of interaction of hydrogen [at 723 K, 5.5 MPa (842°F, 800 psi)] have been determined for 25 coals and correlated with conversion in liquefaction.
16. A geochemical study of 30 major, minor, and trace elements in four lignite drill cores and a channel sample of a lignite has been completed.
17. A qualitative and quantitative procedure for the analysis of minerals in coals, based on Fourier transform infrared spectroscopy has been set up and partially validated.

Category: Reactors/Dissolvers

Institution: Pennsylvania State University

Funding agency: EPRI

Principal investigator: P. L. Walker, Jr., 814/865-6511

Location: Department of Material Science and Engineering; University
Park, Pa. 16802

Funding: \$500,000 for the period January 1975 to June 1979. No indications that the project will be continued.

Objectives: To correlate liquefaction phenomena with mineral matter or organic material in feed coals and to determine coking properties of SRC and its fractions.

Accomplishments:

1. Determined coking tendencies of SRC product from the SRC-I process at Wilsonville, Alabama.
2. Determined coking tendencies of SRC from Wilsonville obtained by bypassing the dissolver (short residence time configuration).
3. Determined that some organic compounds reduced coking tendencies (donor solvents) and some increased coking tendencies (hydrogen scavengers).
4. Proposed a mechanism of coking under SRC reactor conditions that includes hydrogen transfer reactions.

Category: Reactors/Dissolvers

Institution: Sandia Laboratory

Funding agency: DOE

Principal investigators: Barry Granoff for mineral matter studies and
R. K. Traeger for short-time reaction studies,
505/264-2155

Location: Dept. 4730, Albuquerque, N.M. 87185

Funding: Mineral matter studies — \$280,000 in 1979; \$350,000 in 1980.
Short-time reactions — \$400,000 in 1979; \$450,000 in 1980.

Objectives:

Mineral matter studies — To identify the catalytic behavior of minerals and to evaluate their use as potential disposable catalysts.

Short-time reactions — To define the chemistry and kinetics of short-residence-time reactions.

Accomplishments:

Mineral matter studies — 1. Confirmed mineral matter effects;
2. Demonstrated the effectiveness of pyrite in converting SRC to distillates.

Short-time reactions — 1. Developed revised reaction schemes;
2. Developed a preliminary model for physical changes during reaction;
3. Developed instrumental methods to measure reactor residence times (pulse studies based on magnetic or conductive properties).

Relation of project to other programs:

The coordinated (DOE) effort involves:

1. PETC for screening mineral and disposable catalysts;
2. Sandia for research on chemical and process effects and identification of active species;
3. Pennsylvania State University for chemical analyses and maintenance of a coal data base;
4. Gulf (Harmarville and Merriam) for runs in continuous flow units to confirm autoclave studies.

A.2 Fired Heaters for Slurries

A.2.1 Introduction

In the SRC demonstration plant designs, fired heaters are specified for one or more slurry services: SRC-I requires a fired heater to increase the feed slurry (including some hydrogen) temperature from about 530 K (500°F) to about 700 K (800°F); SRC-II heats a similar slurry-hydrogen stream over a slightly lower temperature range, 480 to 670 K (400 to 750°F). A second application of the slurry in SRC-II is the preheating of the heavy feed to the vacuum tower, where residue-containing solids is separated from distillate product. The most demanding service is preheating mixed-phase hydrogen and slurry to the reactor. Design difficulties relate to several factors: rapid viscosity changes resulting from initial coal dissolution, which produces swelling and a viscosity peak at a temperature of about 590 K (600°F); endothermic and exothermic chemical reactions at higher temperatures; non-Newtonian flow characteristics; high potential for erosion and corrosion; variations in flow regime and homogeneity caused by physical and chemical changes; fouling and possible coking at the higher temperatures; and the effect of all these factors on the overall heat transfer and pressure drop characteristics in the fired heater tubes. Current design efforts rely on data from pilot plant fired heaters taken at conditions not representative of, and possibly not scalable to, demonstration plant design. Pilot plant heaters are operated typically at lower slurry velocities and heat fluxes than those under consideration for the demonstration plants. Predictive capabilities for flow regimes at demonstration process conditions are not adequate currently, as discussed in the following section.

A.2.2 Comments on coal/oil slurry preheater design aspects

Flow regime. The two-phase flow regime will affect other design aspects, including heat transfer, pressure drop, and chemical reaction. Therefore, the current state of the art in predicting flow-regime transitions in horizontal (or near horizontal) pipes was reviewed and evaluated. The sample case chosen was the Phase 0 design for the SRC-I demonstration

plant slurry preheater. Input values for the Phase 0 slurry preheater were obtained at a DOE project review meeting¹ and from other public sources. Because of Gulf proprietary claims, the SRC-II Phase 0 preheater design cannot be reviewed publicly. The coal/oil phase was treated as a homogeneous mixture, an assumption supported by preliminary calculations for 841- μm (20-mesh) particle size (Phase 0 design basis) and certainly applicable to the 74- μm (200-mesh) coal considered a design option in the Phase I effort.

Examination of a dozen or so flow-regime maps for horizontal flow of gas/liquid mixtures, the rationales underlying them, and comparisons of data with these maps led to the following conclusions:

1. The first usefully generalized flow-regime map — that of Baker,² though a pioneering effort when published in 1954 — is decidedly obsolete now. One of the coordinates of this dimensional map, for example, contains surface tension to the first power, which definitely exaggerates the influence of this property on the various transitions possible among the seven flow regimes identified on the map.

2. Many other flow-pattern predictors proposed in later years are quite similar to one another, although based on increasingly larger data sets than those available to Baker. Several maps are plotted with axis coordinates that are equivalent to the inlet volumetric fraction of gas (or liquid) a modified Froude modulus [$N_{Fr} = V_m/gD$, where V_m is the homogeneous (no-slip) mixture velocity, g is gravitational acceleration, and D is the pipe's inside diameter]. Correlations in this general category that were examined include those proposed by Griffith and Wallis,³ Beggs and Brill,⁴ Mandhane et al.,⁵ Husain et al.,⁶ Coulson and Richardson,⁷ Scott,⁸ and Schicht.⁹ The last two are essentially revised Baker maps that use the same coordinates but have somewhat shifted transition boundaries.

3. The ingenious theoretical approach used by Taitel and Dukler¹⁰ in developing their flow-regime transition model leads to a flow-pattern map much more firmly based on fundamentals of the phenomenon than any previously proposed map. Comparison of the correlation with a large data set for adiabatic flow showed agreement of the data.¹⁰ The method has been extended successfully to transient gas-liquid horizontal flow,¹¹

and simplified versions of the model/map have been proposed by Breber et al., at Heat Transfer Research, Inc. (HTRI), who compared their model with a large amount of data for diabatic (condensing vapor) flow inside horizontal tubes.¹²

In neither version of this fundamental approach to flow-regime transition prediction does surface tension enter in any way.^{10,12} In the paper from HTRI, surface tension in the data set varied over an order of magnitude (0.0058 to 0.0573 N/m) for tubes with inside diameters from 4.8 to 50.8 mm (0.19 to 2.00 in.), but no effect of surface tension is evident in the data comparison.¹² It appears that, except for the relatively unimportant transition from smooth stratified flow to wavy stratified flow and for the secondary parameters such as equilibrium bubble diameter in dispersed bubble flow, surface tension is unlikely to play a significant role in flow-regime maps for inside diameters beyond the capillary range. Kutateladze and Sorokin analyzed the critical gas velocity associated with disturbance of liquid-phase stability for thirteen diverse cases of gas-liquid system interaction.¹³ For nine cases, which include those most similar to the gas/liquid cocurrent-flow-phase transition situations, $V_{gc} \propto \sigma^{1/4}$, in which V_{gc} is the critical gas velocity and σ is the surface tension (liquid-gas). Parimi has suggested that the important transition between slug and bubble flows may be influenced, approximately, by $\sigma^{1/4}$.¹⁴ In summary, Baker's plot² certainly overstates flow-regime transition dependence on surface tension; it may be influenced by surface tension to a power as large as 1/4, but this figure has yet to be proven. Available data are best correlated by the dimensionless coordinates map proposed by Taitel and Dukler¹⁰ and a simpler version developed by Breber et al.,¹² in neither of which surface tension appears.

4. The dependence of two-phase flow regime on non-Newtonian slurry rheology will require a considerable effort to delineate. One study was found in which a good degree of agreement was reported between a modified correlation and the available data,¹⁵ but the results are limited to two-phase pressure drop and average gas volume fraction for the one case of cocurrent horizontal annular flow of a turbulent gas and laminar non-Newtonian (pseudoplastic) liquids. Additional study of non-Newtonian liquids is necessary, including determination of flow-regime transitions.

For pseudoplastic liquid (slurry) behavior, the results of Hanks and Ricks¹⁶ for friction factor as a function of a generalized Reynolds number (Re') and the flow behavior index (n) could be used to modify the Taitel-Dukler flow map, but this modification would require comparisons with relevant data.

Rheological data acquired at ORNL with 35 wt % (Illinois No. 6) coal slurries in SRC-I recycle solvent at 400 to 700 K (260 to 800°F) and at shear rates of 100 to 600 s^{-1} have been correlated by R. W. Hanks.¹⁷ Critical slurry velocities for transition from laminar flow were calculated for various pipe diameters and flow indices ($n = 0.3$ to 1.0). Of particular interest is that for 0.13- to 0.17-m ID (6- to 8-in.) preheater pipes, the critical transition velocities are ≤ 0.5 m/s (1.64 fps). Based on these data and this analysis, slurry flow in the Phase 0 SRC-I and SRC-II demonstration plant preheaters would be in the transitional or turbulent regions because the design slurry velocities significantly exceed 0.5 m/s. These results relate only to the slurry flow regime and do not address the two-phase flow pattern.

Example: The inlet flow regime for the Phase 0 SRC-I demonstration plant preheater was estimated first, based on the following information (see notation for this section, p. A24):

$$\begin{aligned} D_i &= 0.17 \text{ m [8 in. (0.568 ft)]} \\ G_t &= 1211 \text{ kg/m}^2 \cdot \text{s} \text{ (8.926 x 10}^5 \text{ lb/hr} \cdot \text{ft}^2\text{)} \\ y &= 0.0262 \\ \rho_L &= 1011.25 \text{ kg/m}^3 \text{ (63.13 lb/ft}^3\text{)} \\ \rho_g &= 17.75 \text{ kg/m}^3 \text{ (1.108 lb/ft}^3\text{)} \\ \Delta \rho &= 993.50 \text{ kg/m}^3 \text{ (62.02 lb/ft}^3\text{)} \\ \mu_L &= 0.062 \text{ Pa} \cdot \text{s (62 cP)} \\ \mu_g &= 13.2 \text{ } \mu\text{Pa} \cdot \text{s (0.0132 cP)} \end{aligned}$$

Surface tension was estimated from a value for Exxon Donor Solvent (EDS) recycle solvent (0.036 N/m at 25°C) by extrapolating to the inlet temperature of 537 K (507°F) using the parachor relationship. This procedure yielded a value of ~ 0.0186 N/m. Only the Baker map² and the directly related modifications by Scott⁸ and Schicht⁹ predict bubble flow; all the others indicate slug flow. At this point, the HTRI version¹² of

the Taitel and Dukler model¹⁰ was adopted as the most accurate simple procedure available, and the exit flow regime for the 0.17-m ID (8-in.) heater, as well as the inlet and exit regimes for a 0.13-m ID (6-in., 0.432-ft) heater, were estimated. Exit parameters were as follows:

$$G_t = 1209 \text{ kg/m}^2 \cdot \text{s} \quad (8.914 \times 10^5 \text{ lb/hr} \cdot \text{ft}^2 \text{ for 8-in. heater})$$

$$2086 \text{ kg/m}^2 \cdot \text{s} \quad (1.538 \times 10^6 \text{ lb/hr} \cdot \text{ft}^2 \text{ for 6-in. heater})$$

$$y = 0.2162$$

$$\rho_l = 802.85 \text{ kg/m}^3 \quad (50.12 \text{ lb/ft}^3)$$

$$\rho_g = 60.87 \text{ kg/m}^3 \quad (3.80 \text{ lb/ft}^3)$$

$$\Delta \rho = 741.98 \text{ kg/m}^3 \quad (46.32 \text{ lb/ft}^3)$$

$$\mu_l = 0.021 \text{ Pa} \cdot \text{s} \quad (21 \text{ cP})$$

$$\mu_g = 20 \text{ } \mu\text{Pa} \cdot \text{s} \quad (0.020 \text{ cP})$$

The outlet slurry viscosity was not stated in the available documents, and the nominal value of 0.021 Pa·s (21 cP) was taken from ranges estimated by Thorogood.¹

Two-point operating lines are plotted on the Breber map¹² in Fig. A.1 for both 0.2-m (8-in.) and 0.15-m (6-in.) pipe diameters. For each size, the two-phase flow begins in the slug regime; but whereas the outlet state is in a transition zone for the 8-in. diameter (slug to annular or slug to bubble), the outlet flow for the 6-in. diameter would be in the desirable dispersed-bubble regime. These estimates were recalculated using Taitel and Dukler's model, and the results were unchanged. Note that potentially high erosion rates in the return bends caused by local flow-pattern changes and secondary flows could be accompanied in straight runs of preheater piping by flow surging (between two flow patterns) when the nominal flow is in the transition region (Fig. A.1).

Phase interaction. The power dissipated per unit volume can be viewed as a measure of the mixing intensity (of slurry with H₂-rich gas). Using methods accepted for two-phase Newtonian flows,¹⁸⁻²⁰ the frictional pressure gradients at coil inlet and outlet for both 6- and 8-in. pipes were calculated. These values were used to calculate P_d/v for the four cases with the following results:

Size (in.)	P_d/v [kW/m ³ (fluid hp/ft ³)]	
	Inlet	Outlet
8	0.45 (0.017)	1.13 (0.043), if slug flow 7.00 (0.266), if bubble flow
6	1.63 (0.062)	39.69 (1.507)

Similarly, estimates of the average shear rates at the inlet and the outlet of the preheater can be made. At a fluid point, $P_d/v \equiv \dot{\gamma}\tau$. Over an area or within a finite volume, this is not rigorously valid but can be used to approximate the average shear rate. Using the previously calculated values for P_d/v and the relation $\bar{\dot{\gamma}} = [(P_d/v)g/\mu]^{1/2}$,

Size (in.)	Planar-average shear rate $\bar{\dot{\gamma}}$, (s ⁻¹)	
	Inlet	Outlet
8	85	233, if slug flow 580, if bubble flow
6	162	1380

For the 8-in. reference design, the average shear rate is, therefore, about 85 to 233 s⁻¹, which may be compared with Parimi's early estimates¹⁴ of about 40 to 100 s⁻¹ for the SRC-II Phase 0 demonstration plant preheater and of 70 to 230 s⁻¹ for the new (1979) Ft. Lewis preheater.

Bubble flow, if achievable without excessive erosion rates, would provide a smoother flow, less pipe vibration, and probably a longer coil life in addition to enhanced interphase mixing.

Heat transfer. From the two-phase, Newtonian viewpoint, the correlations used in the SRC-I Phase 0 design¹ (i.e., those of Chen and of Collier, Lacey, and Pulling, as described elsewhere^{19,20}) are appropriate. If emphasis is placed on the non-Newtonian aspects of the slurry flow, the correlations proposed for laminar forced flow of power-law pseudoplastics by Bassett and Welty,²¹ Charm and Merrill,²² and Mahalingam et al.²³ could be effectively used. For turbulent flow of a pseudoplastic slurry, the correlation of Clapp²⁴ should be considered.

Preliminary comparisons of available slurry heat transfer data with two-phase Newtonian correlations and with non-Newtonian relationships indicate good engineering agreement.²⁵ Each approach should be carefully compared with data obtained with the new Ft. Lewis preheater.

Other aspects. A number of additional aspects that are important, in varying degrees, to a successful, integrated slurry preheater design include the following:

1. Erosion rates and associated velocity limits
2. Fouling and coking rates
3. Chemical reaction rates and net heat of reaction
4. Phase residence times (slip ratio and void fraction)
5. Fractional solvent vaporization in the preheater
6. Effective inlet mixing techniques for the slurry and H₂-rich gas phases

Available data and known estimates are in relatively poor agreement. An integrated program involving acquisition, interpretation, and correlation of relevant data is still needed if an operable and efficient preheater design is to result.

Notation for Section A.2.2

- D_i = pipe inside diameter
 G_t = total mass velocity (all phases)
 g = gravitational acceleration
 j_g^* = Wallis's dimensionless gas velocity, $\frac{G_t y}{(D_i g \rho_g \Delta \rho)^{1/2}}$
 n = flow behavior index in power-law model
 P_d/ν = fluid power dissipation per unit fluid volume
 ΔP = axial pressure drop
 $\dot{\gamma}$ = local shear rate
 $\bar{\dot{\gamma}}$ = average shear rate
 V_{gc} = critical gas velocity
 X = Martinelli two-phase flow parameter, $\sqrt{\Delta P_l / \Delta P_g}$
 y = weight fraction gas or vapor
 $\Delta \rho$ = phase density difference, $\rho_l - \rho_g$
 μ_g = viscosity of gas
 μ_l = viscosity of liquid (slurry)

- ρ_g = density of gas
 ρ_l = density of liquid (slurry)
 σ = surface tension (liquid/gas)
 τ = local shear stress

A.2.3 Related R&D programs

Several institutions have been involved in examining various facets of the fired heater problem. Table A.1 lists many of these. The most relevant experimental program is that planned to start at Ft. Lewis in late 1979. The pilot plant studies, in order of relevance to the SRC Demonstration Plants Projects (SRCDPP), are listed below.

Ft. Lewis Measurements of ΔP and skin and slurry temperatures with the existing 89-mm-OD (3-in., sched 160) preheater. A new racetrack preheater, 48- and 60-mm OD (1-1/2-in. and 2-in. sched 160), will be tested beginning about October 1979. The initial test schedule calls for 17 runs (12 in SRC-II mode and 5 in SRC-I mode), plus 6 erosion tests of 500 h each.

Wilsonville Measurement of ΔP and skin and slurry temperatures in a 42-mm OD (1-1/4 in., sched 160) helical coil during dissolver bypass runs and preheater gas bypass tests.

Exxon Tests in Exxon's 907-kg/d (1-ton/d) pilot plant of both separate and mixed-phase preheating in tubes of about 6-mm (1/4-in.) ID to outlet slurry temperatures of 700 to 750 K (800 to 885°F), varying coal type, coal loading, and velocity.

Smaller scale studies could also provide input to the SRCDPP, particularly with respect to slurry rheology, laminar heat transfer, erosion, and fouling and coking propensities.

A.2.4 Recommendations and conclusions

Visits, discussions, and study have indicated that the following areas are of particular importance:

1. Continued acquisition of slurry rheology data in flow systems, especially at slurry temperatures above 480 K (400°F) and at wall shear rates ranging from 10 to 1000 s⁻¹. A moderate

Table A.1. Fired heaters for slurries

Contractor	General description	Other project information	Applicability
Exxon Research & Engineering Co. ^a	EDS liquefaction process development	Includes rheology of coal/solvent slurries and slurry preheater development (separate and mixed-phase operation)	Partial. SRC design coal type and recycle solvent are different from those studied for EDS
Pittsburg and Midway Coal Mining Company ^b	SRC-II process development. Contract 496 (Ft. Lewis)	Measurement of slurry viscosities and coking temperatures of heavy liquids, development of new slurry preheater design data, analysis of preheater outlet samples	Direct to SRC-II
ORNL ^b	Coal slurry rheology and preheater studies	Two- and three-phase heat transfer and ΔP data in small tubes with fine coal	Direct for rheology and laminar-flow heat transfer. Potentially for turbulent flow regime
Institute of Gas Technology ^b	Coal conversion systems technical data book	Comparisons of correlation predictions with limited data on heating of coal/oil slurries	Partial. Relates more closely to SRC-I
Gulf R&D ^b	Slurry preheaters — coking and fouling parameters and preheater testing, including a scheduled series using 50 coals	In P98 and P99 units at Harnmarville, Pa.	Direct to SRC-II
Lawrence Berkeley Lab. ^b	Alloy test program. Determine erosion/corrosion caused by coal slurry flows at 340 to 480 K (160 to 400°F) and $V \approx 3$ to 12 m/s (10 to 40 fps)	Principal investigator, A. V. Levy; Berkeley, Calif.; through 1981 at \$130,000 per year	Direct
Sandia Lab. ^b	Chemical reaction and conversion in three-phase preheaters, including kinetics, H_2 consumption, and flow regime	Principal investigator, R. K. Traeger; funded through FY 1979	General
Argonne National Lab. ^b	Slurry viscosity, heat transfer, flow regimes, and heat of reaction	Principal investigator, J. Fischer; emphasis on Synthoil; terminated September 1977	Limited (a few simulation tests)

Table A.1 (continued)

Contractor	General description	Other project information	Applicability
Auburn Univ. ^b	SRC and related coal liquefaction studies	Principal investigator, J. A. Guinn and A. R. Tarrer; some data regarding preheater coal conversion and the peak-viscosity region	Limited
Stanford Univ. ^c	Characteristics of coal slurries	Principal investigator, Dr. Acrivos; emphasis on prediction of viscosities and correlation of settling rates in inclined tubes; recently completed	Not to SRC coal/oil slurries

^aFunding agency - DOE and EPRI.

^bFunding agency - DOE.

^cFunding agency - EPRI.

expansion of current programs is indicated. Excluding Exxon and in-place pilot plant programs and with emphasis on existing small-scale programs, about \$300,000 per year for two years is recommended.

2. Verification (for SRC slurries) of the later and more fundamental two-phase flow-regime maps, especially with respect to tube inside diameter, flow angle (from the horizontal), and physical properties. This would include a determination as to whether surface tension is needed for such correlations. In conjunction with verification of such flow maps also recommended for slurry phase heat exchangers, this study would require about \$200,000 per year for two years and would be most effectively performed by an expert in the field. For purposes of illustration, A. E. Dukler, at the University of Houston, is the type of investigator envisioned because of his many years of previous work in this field.
3. Comparison of laminar and turbulent slurry heat transfer data, for homogeneous velocity to ~ 10 m/s (~ 30 fps), with available correlations for power-law fluids (e.g., Charm and Merrill for laminar flow; Metzner and Friend for turbulent flow), similar to IGT's efforts in the Coal Conversion Systems Technical Data Book Project. This would involve a two-year program at about \$200,000 per year.
4. An investigation of flow/heat transfer in long-radius heated return bends and the effects of bend flow on the downstream straight-section flow pattern. Again, a two-year, \$200,000 per year program is indicated.
5. Continued acquisition of erosion-rate data for coal/oil slurries as exemplified by A. V. Levy's program at Lawrence Berkeley Laboratory. This would include measurements for longer radius return bends. About \$150,000 per year through CY 1982 is indicated.
6. Establishing procedures for design of effective slurry/gas mixers. This area has not been suitably addressed; it could significantly affect initial flow-pattern development in the fired heater. A one-year program at about \$150,000 should be sufficient for this study.

Items 1 through 5, taken jointly, would provide information that would allow an informed trade-off between an undesirable flow pattern, pulsating flow, and poor heat transfer rates at low levels of slurry velocity and exceeding allowable limits of ΔP and/or erosion rate at high slurry velocities.

In addition to the new and/or expanded studies listed above, existing programs at the pilot plants, particularly at Ft. Lewis, should be continued to support design efforts in the SRC DPP. Special attention should be devoted to technical coordination of these various projects, extending to establishment of a central data interpretation/correlation center in addition to efforts at the individual sites.

A.2.5 Literature cited

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A.2.6 R&D efforts listed by institution contracting the Research

Category: Fired Heaters for Slurries

Contractor: Exxon Research and Engineering Co.

Funding agency: DOE and EPRI

Principal investigators: B. T. Fant and W. N. Mitchell

Location: Baytown, Tex.; Florham Park, N.J.

Schedule: Continuing.

Objectives: Support of Exxon Donor Solvent process.

Need for testing of results at larger scale: Probably none.

Applicability to demonstration project: Indirect (different coal and recycle solvent).

Relevance to SRC or coal liquefaction technology: General.

Relation of project to other programs: Broad.

Category: Fired Heaters for Slurries

Contractor: Gulf Science and Technology Co.

Funding agency: DOE

Principal investigators: K. Parimi, 412/665-5626 and F. E. Wynne,
412/665-6413

Location: Pittsburgh, Pa.

Schedule: Funded through December 1979.

Objectives: Heat transfer, fouling, and slurry reactions for SRC-II preheaters.

Accomplishments: Early results reported for tube ID of 3 to 6 mm (1/8 to 1/4 in.), wall temperatures to 805 K (990°F), and heat fluxes to 7090 W/m² (2250 Btu/hr·ft²).

Need for testing of results at larger scale: Desirable.

Applicability to demonstration project: Direct to SRC-II.

Relevance to SRC or coal liquefaction technology: General.

Relation of project to other programs: Part of Gulf's overall program.

Program plans: To extend the thermal and flow conditions.

Category: Fired Heaters for Slurries

Contractor: Institute of Gas Technology (IGT)

Funding agency: DOE

Principal investigator: A. L. Talwalkar, 312/567-3755

Location: Chicago, Ill.

Funding level: \$570,000 per year, through October 1980.

Schedule: Compilation continuation.

Objective: Preparation of *Coal Conversion Systems Technical Data Book*.

Accomplishments: Task IV includes coal-solvent slurry heating; comparisons of measured properties, heat transfer, and ΔP have been made for SRC-I and SRC-II preheaters with values from various correlations.

Applicability to demonstration project: Broad.

Relevance to SRC or coal liquefaction technology: Broad.

Relation of project to other programs: Considerable.

Category: Fired Heaters for Slurries

Contractor: Lawrence Berkeley Laboratory (LBL)

Funding agency: DOE

Principal investigator: A. V. Levy, 415/486-5822

Location: Berkeley, Calif.

Funding level: \$130,000 per year through CY 1981.

Schedule: 340 K (160°F) operation now, to 480 K (400°F) in Fall 1979.

Objectives: Determine erosion/corrosion of various metals caused by coal slurry flows at 3 to 12 m/s (10 to 40 fps) and 340 to 480 K (160 to 400°F) in a 50-mm-diam (2-in.) pipe loop and ancillary experiments.

Accomplishments: First firm results just becoming available.

Applicability to demonstration project: Direct.

Relevance to SRC or coal liquefaction technology: Direct.

Relation of project to other programs: Complementary.

Program plans: To continue with various combinations of metals and stream compositions.

Impact of redirection/extensions: Continuation/expansion desirable; include longer radius return bends in the program.

Category: Fired Heaters for Slurries

Contractor: Oak Ridge National Laboratory

Funding agency: DOE

Principal investigator: E. L. Youngblood, 615/574-6814

Location: Oak Ridge, Tenn.

Funding level: \$250,000 for FY 1979.

Schedule: Combined with physical properties program at ORNL (see Sect. A.8.4) in FY 1980.

Objectives: Coal slurry rheology and preheater studies.

Accomplishments: Acquisition of rheological and heat transfer data for coal/oil slurries over a range of temperatures and shear rates.

Need for testing of results at larger scale: Desirable for heat transfer results.

Applicability to demonstration project: Direct.

Relevance to SRC or coal liquefaction technology: Direct.

Relation of project to other programs: Complementary.

Category: Fired Heaters for Slurries

Contractor: Pittsburgh & Midway Coal Mining Co. (P&M)

Funding agency: DOE

Principal investigator: C. H. Wright, 913/722-3200

Location: Merriam, Kan.

Schedule: Through FY 1979.

Objectives: Measurement of thermal, chemical, and physical behavior of SRC-I and SRC-II preheaters.

Accomplishments: Values obtained for viscosities, conversions, approximate heats of reaction, and product compositions for various coals, coal loadings, and temperatures. Both batch autoclave and small, 2.8-mm-ID (0.11-in.) coiled preheaters.

Need for testing of results at larger scale: Desirable.

Applicability to demonstration project: Limited.

Relevance to SRC or coal liquefaction technology: General.

Relation of project to other programs: Part of Gulf's overall program.

Program plans: Extended studies of high-temperature, short-residence-time SRC reactions. Merriam efforts directed toward longer range program goals not directly supportive of SRC demonstration plant.

Category: Fired Heaters for Slurries

Contractor: Sandia Laboratories

Funding agency: DOE

Principal investigator: R. K. Traeger, 505/264-2155

Location: Albuquerque, N.M.

Funding level: \$400,000 in FY 1979; \$450,000 in FY 1980.

Schedule: Funded through FY 1980.

Objectives: Study of the physics, chemistry, and kinetics of SRC-II preheaters.

Accomplishments: Conversions, kinetics, and product compositions determined for Illinois No. 6 coal in a heavy distillate oil to 730 K (850°F) with 13.8 MPa (2000 psi) H₂ in a 3.1-mm-ID (0.122-in.) helical coil.

Need for testing of results at larger scale: Desirable.

Applicability to demonstration project: Direct.

Relevance to SRC or coal liquefaction technology: General.

Relation of project to other programs: Complementary.

Program plans: Extend studies of kinetics, flow regime, residence time, and characterization of high-viscosity region by using a larger coil [5.16-mm ID (0.203-in.)].

A.3 Solids/Liquid Separations

A.3.1 Introduction

One of the most critical R&D problems in the SRC-I process is solids/liquid separation. The major effort in solids/liquid separation for this process has been pressure precoat filtration. Filtration of coal liquids, often described as a mechanical nightmare, is characterized by high capital costs, high operating costs, low filtration rates, screen blinding, and a multitude of mechanical/operational problems. These problems are interrelated and a solution for one may help solve several of the others. For example, a nonmechanical means of increasing filtration rates would lessen the required filter area and the amount of precoat used, thus decreasing both capital and operational costs. Therefore, the thrust of research in this area should be to determine the best filter type via optimization of several candidates such as vertical leaf, horizontal leaf, rotary drum, and candle filters and to determine alternatives to filtration. These experiments should be performed primarily at the pilot plant level, particularly at Wilsonville.

A.3.2 Relevant R&D programs

Applicable research projects in this area are shown in Table A.2. As the primary source of SRC-I data, the Wilsonville pilot plant has become the primary source of solids/liquid separation data. In the past, Wilsonville has concentrated on optimizing and determining the operability of a FUNDA filter and a U.S. brand vertical leaf filter. No optimal solution has yet been discovered to eliminate the problems of low filtration rates and filter screen blinding. The solution to mechanical problems is proceeding well in the areas of screen supports, seals, and the sluicing system. Operation of the U.S. brand filter is continuing, as well as several important new areas of exploration:

1. Sized anthracite is being used as a carbonaceous precoat, thus making the discharged precoat material a source of high-carbon-content feed to the gasifier;

Table A.2. Summary of solids/liquid separation research projects

Researcher	Location	Project
W. L. Utnage	Kerr-McGee Corp., Oklahoma City, Okla.	Critical Solvent Deashing
Gordon R. S. Smith	Johns-Manville Sales Corp., Denver, Colo.	Rotary drum filtration
J. G. Walters	PETC, Pittsburgh, Pa.	Low temperature carbonization
D. E. Briggs	Univ. of Michigan, Ann Arbor	Fundamental filtra- tion studies
	Ft. Lewis Pilot Plant, Tacoma, Wash.	SRC Pilot Plant
Everett Huffman	Wilsonville Pilot Plant, Wilsonville, Ala.	SRC Pilot Plant
F. M. Tiller	Univ. of Houston, Houston, Texas	Fundamental filtra- tion studies
R. H. Long	Lummus Company, New Brunswick, N.J.	Lummus antisolvent deashing process
J. D. Henry	West Virginia Univ., Morgantown	Solids/liquid sepa- ration via aqueous extraction
D. T. Wasan	Illinois Institute of Technology, Chicago	Solids/liquid sepa- ration via elec- trofiltration; emulsion studies ^a

^aInstitution capabilities regarding both solids/liquid separation and emulsion studies are described in Sect. A.8.4.

2. The filter is being operated without a precoat and a modified sluicing system is being used to potentially eliminate the cost of precoating;
3. Skid-mounted $0.46\text{--}0.65\text{-m}^2$ (5- to 7-ft²) candle or leaf filters are being operated using either steam or heated N₂ as stripping agents to dry the filter cake on the filter screen.

It has been hypothesized that one possible cause of screen blinding is the drying of the filter cake in place via vacuum. Thus, candle filter studies can not only assess the feasibility of the filter type but will also explore the effects of alternative filter cake drying techniques.

The rotary-drum pressure precoat filter (RPPF) developed by the Johns-Manville Corporation was tested at the Ft. Lewis pilot plant. The 4.6-m^2 (50-ft²) unit was plagued by incorrect installation, process upsets generated in other areas of the plant, and mechanical problems. The unit did exhibit filtration rates in excess of $0.27\text{ kg/s}\cdot\text{m}^2$ ($200\text{ lb/hr}\cdot\text{ft}^2$) prior to shutdown; this is a marked improvement over the typical filtration rates of other units, which are consistently below $0.14\text{ kg/s}\cdot\text{m}^2$ ($100\text{ lb/hr}\cdot\text{ft}^2$). Rates as high as $0.35\text{ kg/s}\cdot\text{m}^2$ ($260\text{ lb/hr}\cdot\text{ft}^2$) were obtained under conditions not optimized for high rates (e.g., at only one-half the design drum rotation speed).

Alternatives to filtration are being pursued at Wilsonville, Ft. Lewis, Kerr-McGee, and Lummus. The Kerr-McGee CSD process is being tested and modified at both Kerr-McGee and Wilsonville. Variables of interest in the study include (1) solids discharge, (2) effect of feed conditions on operability, (3) operability, (4) product recovery, and (5) solvent recovery and durability. The unit at Wilsonville has been operated continuously for as long as 20 days. The Lummus antisolvent deashing process is being developed at Lummus and tested at Ft. Lewis. Operation of the unit at Ft. Lewis was delayed because of faulty welds during installation and equipment plugging during initial start-up attempts. Subsequent testing has been performed, and several periods of acceptable overflow product quality were reported in August 1979. Variables of interest in the program include antisolvent-to-feed ratio, feed rate, temperature of operation, and the effect of these on the ash

level in the stripped overflow; and overall SRC recovery, solvent losses, and operability of settler-underflow flow-control systems. Specific results obtained in both programs are proprietary and cannot be discussed in this report. General discussions of results can be found in the periodic reports of the pilot plants.

Studies on fundamental agglomeration techniques and their subsequent effect on filtration have been performed at ORNL, West Virginia University, the University of Michigan, the University of Houston, Johns-Manville, and elsewhere. The studies have concentrated on the effects on filtration of solvent/antisolvent addition, body feeds, and precoat types but have produced no economical means of greatly increasing filtration rates. Of these programs, the only one still funded to any significant degree is at the University of Michigan under Dr. Dale Briggs. His work concentrates on identifying the fractions of coal liquids that are primarily responsible for the low filtration rates. Once they are identified, he will explore possible solutions to the problem. Future plans include an exhaustive look at carbonaceous precoat materials.

A.3.3 Conclusions and recommendations

Because solids/liquid separation remains one of the most critical R&D problems for SRC-I, the emphasis in this area of research should be directed toward the SRC-I process. The thrust of the national R&D solids/liquid separation program encompasses universities, technology centers, and pilot plants and represents a comprehensive and cost-effective approach to the problem. Assuming that existing and planned research projects at the major facilities are funded at anticipated levels, only a few major projects remain to be initiated.

Several general conclusions are drawn from the completed assessments:

1. Pilot plants are best used for testing and optimizing large-scale equipment and obtaining qualitative information;
2. National laboratories and energy technology centers are best used for generating quantitative data, particularly for long-term (greater than two or three years) projects;

3. Universities are best used for generating quantitative data for short-term projects, particularly those with general applications.

The universities visited expressed little desire to generate data for a particular coal liquefaction process; they preferred to use coal liquefaction studies primarily as a tool for training students.

The primary source of separation data is, and should remain, the pilot plants. Wilsonville is the main source of data because of its emphasis on SRC-I. The Wilsonville approach is twofold: (1) optimize pressure precoat filtration via a U.S. brand vertical leaf filter and (2) optimize an alternative to filtration (i.e., the Kerr-McGee CSD process). The goal of the filtration work is to optimize the operation of the unit and to demonstrate its long-term reliability. Key variables in this study are the resulting specific filter cake resistance and compressibility.

We encourage funding for the proposed projects at Wilsonville because they attempt to solve some of the major problem areas of pressure precoat filtration. One major problem is operating cost, due largely to the expense of filter precoat. Wilsonville proposes to study the use of sized anthracite as a precoat. Although it is not anticipated that this material will greatly reduce the volume of discharged filter precoat, it will produce a solids residue that has a positive rather than a negative cost value; the discharged carbonaceous precoat will have a heating value that can be recovered via gasification. Another major problem associated with filtration is screen and precoat blinding; this is because of the nature of the solids in coal slurries (e.g., a major portion of the solids are in the 1- to 10- μ m range). Wilsonville proposes to approach the problem in two ways: (1) try to eliminate the use of precoat via a modified sluicing system and (2) study alternative filter cake drying methods. The elimination of the use of precoat would, of course, eliminate a major operating expense, but it would enhance the possibility of screen blinding (thereby lowering filtration rates). It has not been demonstrated that the sluicing system can be modified to offer a feasible technical and economic solution to the problem. The technique of vacuum drying the filter cake directly on the filter screen

has been hypothesized as a major cause of screen blinding. Thus, Wilsonville is proposing to operate a skid-mounted candle filter or a leaf filter to test either steam or hot N_2 gas as a stripping agent for drying the filter cake in place. Another alternative would be to assess the impact of not drying the filter cake in place but removing it wet and performing the drying operation external to the filter.

PETC has existing wet filter cake processing units (i.e., a calcinator and a fluidized bed) that are currently uncommitted. As a long-range investigation, a coordinated effort between Wilsonville and PETC could be initiated, resulting in a program to

1. Assess the effects on filtration rate and screen blinding of wet filter cake removal
2. Assess the economic and technical feasibility of low-temperature carbonization as a means of solvent recovery from filter cake discharge.

The most promising feed to such a unit would be from filtration units using carbonaceous precoat.

In conjunction with the filtration work at Wilsonville, Johns-Manville (with EPRI funding) constructed an improved rotary-drum filter that was given to DOE and subsequently tested at the Ft. Lewis pilot plant. The original tests at Ft. Lewis resulted in identification of several mechanical problems and produced filtration rates of $>0.27 \text{ kg/s} \cdot \text{m}^2$ ($>200 \text{ lb/hr} \cdot \text{ft}^2$), which are substantially higher than those exhibited by other filtration devices. Johns-Manville has proposed a program to correct the mechanical problems and retest the filter. The two main objectives of the proposed program are to optimize the unit and to demonstrate its long-term operability. This program could be initiated as long-range research because the rotary-drum filter is likely to be the most economical of the pressure precoat techniques, if it can be operated as predicted. Any effort in this area is unlikely to affect decisions on SRC-I demonstration plant design, however.

The Lummus antisolvent deashing process, which is a possible alternative to filtration, is being tested at Ft. Lewis. Operation of the unit had been delayed until recently because of faulty welds during installation and system plugging during initial start-up attempts. Thus, of the

two primary alternatives to filtration, the Kerr-McGee CSD process is currently the frontrunner in the SRC-I design because of demonstrated operability (continuous operation of the unit at Wilsonville for as long as 20 days). Because specific information is proprietary, it cannot be discussed in this report. However, both programs should be supported as comprehensive and cost-effective approaches to answering key questions about the processes, including operability, solvent and product recovery, and effects of feed conditions.

Bench-scale data obtained at Kerr-McGee and Lummus, as well as Wilsonville data, indicate that stability of the feed to the processes is necessary. Specifically, for the Kerr-McGee process, the asphaltene content of the feed is a key variable. In an attempt to stabilize the asphaltene content in the CSD feed, Wilsonville will attempt to operate without a vacuum column preheater. It is hoped that this project will result in less repolymerization (because of shorter residence time) of the feed to asphaltenes/preasphaltenes and in the elimination of an expensive piece of equipment. A comprehensive study of the vacuum column and preheater is needed. Pilot plant personnel have indicated that this study is needed not only to determine the chemical stability of the vacuum bottoms (i.e., Kerr-McGee and Lummus feed stream), but also to determine the operating conditions that maximize product recovery and still maintain fluidity of the process stream. The very nature of the study will likely result in equipment plugging, which is undesirable in pilot plant operation. The proposed study could be performed on a bench scale at an appropriate laboratory. The objectives of the program would be to

1. Determine the effects of feed properties, residence time, and solvent vaporization on coking in the preheater;
2. Determine the effects of feed properties, flash zone residence time, pressure, and temperature on solvent recovery, thermal cracking, repolymerization, bottoms viscosity, and bottoms stability.

Equipment needed for this project would include a feed source, vapor/liquid separator, vacuum preheater, vacuum column, and various analytical

devices. The anticipated cost of the three year program is \$500,000 per year, assuming that an experimental rig is already in place and would require only minor modifications.

Currently, there is a minor effort at universities in support of solids/liquid separation research. The universities are directing their efforts toward a more fundamental approach to the problem of filtration or separation. A continuation of this emphasis is the most effective use of their expertise. Of the university work, the primary source of data is the University of Michigan. Dr. Dale Briggs at the University of Michigan proposes to study the causes of micelle formation in coal liquids and the effect of solid extenders and liquid additives (including process-derived light ends), as well as carbonaceous precoat, on filtration rates. The current anticipated funding level (~\$120,000 per year) is the maximum that can be effectively utilized at the university. However, redirecting the program's priorities to emphasize the study of process-derived liquid additives and carbonaceous precoat would increase its applicability to the demonstration plant.

In summary, it is recommended that:

1. Several projects at the Wilsonville SRC pilot plant should be supported. These projects are viable attempts at solving two primary problems associated with filtration: high precoat costs and screen blinding.
  - a. High precoat cost. To reduce high precoat costs, a program to assess the feasibility of a carbonaceous precoat that would have a recoverable heating value in a gasifier should be supported. The program at Wilsonville entails studying the effects of a sized anthracite precoat on filtration rate and equipment performance. Carbonaceous precoat screening tests should be run simultaneously at bench scale in a laboratory equipped for this purpose.
  - b. Screen blinding. The program at Wilsonville to test a 0.46- to 0.65-m² (5- to 7-ft²) skid-mounted candle or leaf filter, using either steam or hot N₂ as a stripping agent

to dry the filter cake, should utilize both steam and hot N_2 in a candle filter. Two alternatives to vacuum drying thus can be assessed simultaneously with the assessment of the technical feasibility of a new type of filter. Although screen blinding may not be affected by the drying method, the data will still be useful in determining the optimum technical and economic drying technique.

2. A program to test the Johns-Manville rotary-drum filter at the Ft. Lewis SRC pilot plant should be considered by DOE as long-range research. Costs could be reduced by operating the unit when the Lummus antisolvent deashing process is offstream.
3. The University of Michigan program should be supported as proposed — a three-year extension at \$120,000 per year. With minor administrative redirection, this program could be made more beneficial to the SRC DPP. Two of the program objectives should be given priority: (1) determination of the effects of process-derived light ends on filtration and (2) determination of the effects of carbonaceous precoating. Promising results should be verified on either the U.S. brand filter or the skid-mounted filter at Wilsonville.
4. Experiments on both the Lummus and Kerr-McGee deashing processes indicate that stability of the feed is imperative. Currently, there is a very limited program to study vacuum-bottoms stability or to obtain data suitable for scale-up. Thus, it is proposed that a new program be initiated to study vacuum distillation. The program would involve a three-year effort costing about \$500,000 per year. Equipment would include a feed source, vapor/liquid separator, vacuum preheater, vacuum column, and various analytical devices. The objectives of the program would be to (1) determine the effects of feed properties, residence time, and solvent vaporization on coking in the preheater and (2) determine the effects of feed properties, flash-zone residence time, pressure, and temperature on solvent recovery, thermal cracking, repolymerization, bottoms viscosity, and bottoms stability.

5. The U.S. brand filter studies at Wilsonville should be complemented by laboratory-scale studies to eliminate screen tearing and bottoms seal failure and to reduce drain and fill time. This program should be carried out at a filter company or comparably equipped laboratory.

A.3.4 R&D efforts listed by institution contracting the research

Category: Solids/Liquid Separations

Contractor: Johns-Manville Sales Corp.

Funding agency: DOE/Fossil Energy Division (DOE/FE), EPRI, P&M

Principal investigator: Gordon R. S. Smith, 303/979-1000

Location: Research and Development Center, Ken-Caryl Ranch,
Denver, Colo. 80217

Funding level: FY 1974 to FY 1979 overall average of \$205,000 per year for a five-year period. Total DOE/FE funding, \$290,000; EPRI, \$665,000; P&M, \$70,000.

Schedule: Program not currently active.

Objectives:

EPRI

This was a two-phase research project:

Phase I. It was the purpose of this study to design and assemble a laboratory-scale, constant-pressure test filter, develop and describe operating procedures, and use the equipment and procedures to study relationships between particle characteristics, filtration pressure, viscosity (as controlled by temperature and solvent dilution), addition of various materials to the unfiltered liquid, and pretreatment methods. The test filter was also to be practical for plant evaluation and monitoring.

Another purpose of this program was to study particle characteristics of both unfiltered and filtered materials. Unfiltered solid materials were analyzed for quantities of suspended solids and particle shape, size, distribution, composition, density, and compressibility.

Phase II. This was an equipment development project of the Fossil Fuel and Advanced Systems Division. The mechanical and operating problems of commercially available rotary precoat filters were listed, based on the contractor's practical experience in the filtration field. A conceptual design for an improved filter to alleviate these problems was prepared. A new filter was sized and built to fit the DOE Ft. Lewis SRC plant. An agreement was reached between EPRI and DOE whereby EPRI would give the filter to DOE. In return, DOE would install the equipment and test the filter in SRC-I service at Ft. Lewis.

DOE

The primary objective of DOE's program was to achieve major improvements in the filtration process and equipment used in coal liquefaction processes. Reproducible doubling of the filtration rate and reduction of filter aid use were ultimate goals. A secondary objective was the development of operating conditions for the start-up in June 1978 of a 4.6-m^2 (50-ft^2) pilot filter at Ft. Lewis.

Accomplishments:EPRIPhase I

1. Measured vapor pressure and viscosity of Ft. Lewis and Wilsonville filter feed over a wide range of conditions.
2. Did extensive characterization of the solids in coal liquids.
3. Built filter leaf, 0.0017 m^2 (0.018 ft^2), for testing filtration characteristics.
4. Measured filtration rates of coal liquids as a function of pressure, temperature, and filter aid concentrations.

Phase II

1. Designed, built, installed, and operated a 4.6-m^2 (50-ft^2) RPPF at the Ft. Lewis pilot plant.
2. Attained high overall filtration rates [$\sim 0.33\text{ kg/s}\cdot\text{m}^2$ ($\sim 240\text{ lb/ft}^2\cdot\text{hr}$)] at 0.5 rad/s (5 rpm), 590 K (600°F), and 0.8 MPa (100 psig).
3. Were unable to demonstrate long-term reliability of the unit.

Problems in Phase II

1. A number of problems were associated with incorrect installation of the filter rather than with filter performance.
2. Only a four-month period was allowed for start-up and operation of the unit at Ft. Lewis.
3. Numerous mechanical problems were encountered. Most are believed to be correctable.

DOE

1. Designed and built a laboratory test leaf filter system [0.01 m^2 (0.1 ft^2)], which simulates an RPPF.
2. Operated a system to investigate the effect of temperature, pressure drop, knife advance rate, drum rotation rate, CELITE precoat, and filter feed materials (from Wilsonville and Ft. Lewis).

Need for testing of results at larger scale:

If RPPF is to be included in the list of viable alternative processes for solids/liquid separation, this apparatus should be tested further at the current scale. If, after testing the unit further, it is apparent that all the mechanical problems can be solved successfully, an economic and technical comparison should be made of the contending processes to decide which one should ultimately be chosen. Current calculations show that if the RPPF operates as predicted, it is one of the more economical filtration alternatives.

Applicability to demonstration project:

Not applicable to the SRC-I demonstration project because of the low probability of affecting design decisions. Long-term impact is possible.

Relation of project to other programs:

This project was operated in conjunction with the Ft. Lewis SRC pilot plant.

Program plans:

Presented below are proposed plans, obtained from Johns-Manville, for an operating campaign for the 4.6-m^2 (50-ft^2) RPPF installed in the Ft. Lewis SRC pilot plant.

Stearns-Roger would be contracted to find solutions in the following problem areas:

1. Modification of the drum drive to facilitate operation at drum speeds of up to 1 rad/s (10 rpm).
2. Replacement of sight glasses.
3. Replacement and/or redesign of sluicing system.
4. Replacement of screens and replacement or modification of screen supports.
5. Installation of recirculation lines to promote slurry mixing in the filter tub.

This effort should be coordinated with Wilsonville. This proposed work has been submitted to EPRI. Johns-Manville estimates installation time for the changes at about 6 man-months, with engineering costs (Stearns-Roger) totaling \$23,000, plus minor capital costs.

Category: Solids/Liquid Separations

Contractor: Kerr-McGee Corp.

Funding agency: Kerr-McGee and EPRI

Principal investigator: W. L. Utnage, 405/270-2569

Location: P.O. Box 25861, Oklahoma City, Okla. 73125;
Wilsonville Pilot Plant, Wilsonville, Ala.

Funding level: Kerr-McGee proprietary information.

Schedule: Continuing.

Objectives:

1. To obtain sufficient bench-scale and pilot plant data to allow engineering evaluation of the CSD process.
  - a. Operate on hot-fresh Wilsonville pilot plant feed.
  - b. Produce deashed solvent refined coal containing not more than 0.16% ash.
  - c. Produce ash concentrate in an easily handled powder form.
  - d. Operate with acceptably low losses of deashing solvent.
  - e. Operate continuously for prolonged periods.
  - f. Determine the effect of SRC properties on CSD performance.
  - g. Maximize SRC recovery.
  - h. Investigate maintaining long-term solvent integrity.
2. To develop the SRC-1/2 process by using the CSD technique to improve process solvent quality.
  - a. Define CSD process conditions to recover 20 to 30% of an SRC feed as light solvent refined coal for recycle to the process front end.
  - b. Define CSD process conditions to recover light solvent refined coal from an unhydrogenated, short-residence-time feed.
  - c. Demonstrate operation of CSD process as an integral part of the SRC-1/2 process. This portion of the program is in conjunction with Conoco Coal Development Company.

Accomplishments:1. Wilsonville CSD operations

- a. The 90-kg/h (200-lb/h) Wilsonville CSD unit has been operated on fresh feed from the SRC pilot plant vacuum column bottoms.
- b. Ash content of the solvent refined coal averaged 0.09% during an extended 20-day run.
- c. Dry, flowable ash concentrate was produced, conveyed, and packaged on a continuous basis.
- d. Solvent losses appear to be reasonably low; however, the minimum attainable solvent loss level is not yet defined.
- e. The unit was operated for 20 days without upset.
- f. Effects of pressure, space rate, and withdrawal of solids on the performance of the CSD unit have been investigated.

2. SRC-1/2 process

- a. Light solvent refined coal is an excellent recycle solvent component and acts as a very good hydrogen transfer agent.
- b. Light solvent refined coal in the feed solvent does not affect coal conversion but increases solvent yields from the CSD unit.
- c. A short-residence-time system was operable using hydrogenated light solvent refined coal and a low-quality recycle solvent without hydrogen addition in the front end of the process.

Need for testing of results at larger scale:

The CSD process has been successfully scaled from 180 kg/d to 2.3 Mg/d (0.2 to 2.5 tons/d). If CSD operations at Wilsonville prove technically and economically successful, further expansion is probably not needed to support the demonstration plant design.

Applicability to demonstration project:

Directly applicable for SRC-I.

Relevance to SRC or coal liquefaction:

Directly relevant to coal liquefaction solids/liquid separations.

Relation of project to other programs:

Operated in conjunction with Wilsonville pilot plant (CSD unit). SRC-1/2 operations are in conjunction with Conoco and EPRI.

Program plans:

Continued operation of the CSD unit at Wilsonville is planned to further demonstrate process operability and solvent recycle and to measure scale-up parameters.

Plans to build an advanced PDU coal liquefaction system to demonstrate SRC-1/2, SRC-I, and SRC-II liquefaction modes, CSD and hydrotreating are under consideration. Short-residence-time processes, phase equilibria, product evaluation, and hydrotreating would be investigated.

Impact of redirection/extension:

We recommend continuation of this work as planned.

Category: Solids/Liquid Separations

Contractor: University of Michigan

Funding agency: DOE

Principal investigators: Dr. Dale E. Briggs, 313/763-1331 or 764-4326 and
Dr. Peter A. S. Smith, 313/764-7355

Location: G. G. Brown Laboratory, Department of Chemical Engineering,
Ann Arbor, Mich. 48109; Chemistry Building, Department of
Chemistry, Ann Arbor, Mich. 48109.

Funding schedule: Table A.3 shows the project and sponsor breakdown.
Funding by calendar year for DOE project:

1976	\$ 55,158
1977	127,178
1978	127,704
1979	68,872

Accomplishments:

An apparatus to filter coal liquids has been constructed and is operational. Current work focuses mainly on characterizing the various components of coal slurries as to their reactivity, adsorption characteristics, and effect on filtration. The work has primarily been with extracted coal liquid fractions dissolved in pure compounds. Some of the specific results to date are:

1. Determination that massive micelle formation occurs at a threshold concentration of asphaltenes.
2. Proof that the addition of solvent (antisolvent) to coal liquids produces viscosities and specific cake resistances that are not consistent with the idealized calculation, indicating that association (or disassociation) of particles takes place.
3. Determination of the following characteristics of coal slurry fractions:

<u>Component of slurry</u>	<u>Characteristic</u>
Oils, nonaromatic	Promotes association
Oils, aromatic	Peptizes low heteroatomic asphaltenes
Low-molecular-weight, low-heteroatomic asphaltenes	Peptizes asphaltenes and pre- asphaltenes

Table A.3. University of Michigan project and sponsor breakdown

Funding agency	Amount (\$)	Period covered by award	Project title	Objectives
<i>Past</i>				
EPRI	150,000	8/1/73-3/1/74	Clean Fuels from Coal	Identify R&D needs to advance best processes for utilizing coal for electric power industry
NSF-RANN ^a	34,000	7/1/73-2/1/75	Studies on the Separation of Coal Extract from Mineral Residue in the Solvent Refining of Coal	Identify best processes for solid-liquid separation in coal liquefaction. Identify causes of low filtration rates
NSF-RANN	111,110	5/1/75-1/1/77	Studies on the Separation of Coal Extract	Extend preceding NSF-RANN project
<i>Current</i>				
DOE	378,912	10/1/76-1/1/80	Physical and Chemical Behavior of Liquefied Coal in Solids Separation	Measure interactions between solvents, mineral matter, and catalysts to provide solubility, colloidal behavior, adsorption, viscosity, and surface tension data

^aNational Science Foundation—Research Applied to National Needs.

<u>Component of slurry</u>	<u>Characteristic</u>
High-molecular-weight, high-heteroatomic asphaltenes	Self-association at moderate temperatures; peptizes preasphaltenes
Preasphaltenes	Bridging agent; self-association at high temperatures
Solids	Able to π - π and H-bond with asphaltenes and preasphaltenes

Need for testing of results at larger scale:

Previous comparisons between small-scale filters and industrial-scale filters exhibit similar qualitative characteristics. However, the best results should be verified on pilot plant filters.

Applicability to demonstration project:

Most results will probably not be available in time to affect demonstration design. Applicable to SRC process technology.

Relevance to SRC or coal liquefaction technology:

Although the work concentrates on identifying the cause of low filtration rates (and finding a solution to the problem), the work is relevant to processes other than SRC-I. The work on characterizing the various components and their interaction capability should prove useful to all processes that use a solids/liquid separation step.

Relation of project to other programs:

The work complements the work on filtration being performed at Wilsonville, Ft. Lewis, and the University of Houston. However, this work should also be considered one of the primary data sources for determining means of lowering the specific cake resistance in filtration.

Institution's capability to perform necessary demonstration support work:

The filtration equipment is high caliber and functional. The approach to the problem is quite comprehensive. The investigators are not advocates of any separation process, preferring instead to determine the cause of the problem before looking for solutions. The quantity and quality of the data generated are excellent.

Program plans:

An extension of the existing program has been proposed to DOE. Dr. Briggs feels that the three year, \$380,000 program will be funded beginning December 1, 1979. The desirable spending schedule would be:

1979	\$ 20,000
1980	130,000
1981	110,000
1982	120,000

The objectives are as follows:

1. On SRC-I and H-Coal slurries derived from Illinois No. 6 and Kentucky No. 9 coals, respectively, study the effect on filtration of (a) process-derived light end additives, (b) liquid additives and solid extenders, and (c) carbonaceous precoating materials.
2. Study the colloidal nature of coal-derived liquids.
3. Study solvent and thermal peptization of bituminous coal in model and potential recycle solvents at conditions comparable to those of the preheater.

Impact of redirection/extension:

Dr. Briggs has stated that the requested funding level is the maximum his institution can handle efficiently for this project. To make the work directly supportive of the demonstration project would only require placing priority on carbonaceous precoats and process-derived light end additives.

Category: Solids/Liquid Separations

Contractor: Pittsburgh and Midway Coal Mining Co.

Funding agency: DOE

Location: Ft. Lewis Pilot Plant, Tacoma, Wash.

Funding schedule: Jan. 1, 1979 to Dec. 31, 1980.

Objectives:

1. Operate the pilot plant to define optimum performance of both SRC-I and SRC-II process for several coals.
2. Evaluate Lummus antisolvent deashing process.
3. Obtain design and operating data leading to improved process operation and equipment design.
4. Perform an in-plant and offsite environmental monitoring program as well as a biomedical program on toxicity of SRC products.

Accomplishments:

The accomplishments are too numerous to list here and are well-documented in various reports. Delays in operation of the Lummus process have been caused by faulty welds during installation and plugging during start-up.

Need for testing of results at larger scale:

Does not apply.

Applicability to demonstration project:

A primary source of SRC-I and SRC-II demonstration plant design data.

Relevance to SRC or coal liquefaction technology:

Direct.

Relation of project to other programs:

The work is a joint effort with other pilot plants.

Institution's capability to perform necessary demonstration support work:

A primary location for support work.

Program plans:

To continue testing and evaluating Lummus antisolvent deashing system. The program will consist of three phases:

1. Initial testing to establish operability and evaluate component performance;
2. Recommendation and implementation of process and/or component modifications as may be required to improve operability and performance (the unit has been operated, although not at steady-state conditions, and has demonstrated several periods of acceptable product quality);
3. Additional testing to establish optimum performance and determine process design criteria.

The process variables to be tested include the antisolvent-to-feed ratio, the feed rate to each module, and the temperature of operation. The test results of interest include:

1. Ash level in the stripped overflow;
2. Overall SRC recovery;
3. Solvent losses;
4. Operability of settler-bottoms flow control system.

Category: Solids/Liquid Separations

Contractor: Pittsburgh Energy Technology Center (PETC)

Funding agency: DOE

Location: Pittsburgh, Pa.

Principal investigator: J. G. Walters, 412/892-2400

Funding schedule: None currently.

Objectives:

To evaluate methods for recovery of oil from primary solids/liquid separation residues.

Accomplishments:

Have a calcinator and fluidized bed installed. The calcinator has gone through shakedown.

Applicability to demonstration project:

None currently.

Relevance to SRC or coal liquefaction technology:

None currently. The facility has two low-temperature carbonization units that are potentially applicable to support of the SRC and liquefaction technology. The equipment could be used to assess the technical and economic feasibility of oil recovery from wet filter cake (Wilsonville) and vacuum bottoms (Ft. Lewis). The information could be applicable to any coal liquefaction process.

Program plans:

None currently.

Impact of redirection/extension:

As a long-range research effort, the program could be extended (i.e., reinstated) to determine the technical feasibility of oil recovery from wet filter cake (produced at Wilsonville) via low-temperature carbonization. This effort would need to be coordinated at the pilot plant with Wilsonville to determine the effect on filtration rates of wet cake removal (rather than drying of cake directly on the filter). A two-year program at PETC would be sufficient to determine the feasibility of the process.

Sufficient funding would be:

1980	\$210,000	3-1/2 man-years
1981	240,000	4 man-years

Equipment modification and shakedown could be accomplished within three months after initiation of the program. Determination of effects of wet filter cake removal on filtration rates would take about two weeks of operation at Wilsonville. This two-week period of operation would provide sufficient feed for the entire experimental program at PETC. To demonstrate the efficiency of low-temperature carbonization it would be advantageous to operate the filter using a carbonaceous precoat. Thus, this program would preferably be coordinated with the planned Wilsonville project of using sized anthracite precoat.

Category: Solids/Liquid Separations

Contractor: Southern Company Services, Inc.

Funding agency: DOE and EPRI

Principal investigator: Everett Huffman, 205/870-6324

Location: Wilsonville Pilot Plant, Wilsonville, Ala.

Objectives:

1. To operate the pilot plant to define optimum performance of the SRC-I process for several coals.
2. To evaluate the Kerr-McGee Critical Solvent Deashing process and the U.S. brand filter.
3. To obtain design and operating data leading to improved process operation and equipment design.

Accomplishments:

The accomplishments are numerous and well-documented. Accomplishments in solids/liquid separations include operation and characterization of a FUNDA horizontal leaf filter, the Kerr-McGee process, and a U.S. brand vertical leaf filter.

Applicability to demonstration project:

The primary source of SRC-I operating and design data.

Relation of project to other programs:

The work is a joint effort with other pilot plants, although this work assumes the lead role for SRC-I.

Institution's capability to perform necessary demonstration support work:

Excellent technical expertise, information transfer, and well-documented results.

Program plans:

1. To continue optimization of the Kerr-McGee process.
2. To operate without E-102 cooler (liquid will go directly from dissolver to flash drum).
3. To operate the Kerr-McGee process without vacuum column preheater.

4. To replace the gas-fired heater on vacuum bottoms with low-temperature Dowtherm system.
5. To operate the U.S. brand filter with sized anthracite as a precoat.
6. To operate the U.S. brand filter without precoat and using a modified sluicing system.

Work proposed:

7. To add gas- and liquid-withdrawal lines to the dissolver to obtain aqueous phase formation information.
8. To operate a 0.46- to 0.65-m² (5- to 7-ft²) skid-mounted candle filter or leaf industrial filter. Either steam or heated N₂ will be used as a stripping agent. This project will not only test a new filter type but will explore alternatives to vacuum drying filter cake on the filter.

Impact of redirection/extension:

It is recommended that the proposed projects (i.e., items 7 and 8) be funded. It is further recommended that item 7 be amended to include the capability of measuring *K*-values as well as aqueous phase information (see vapor/liquid separations section).

Category: Physical Properties

Institution: Illinois Institute of Technology (IIT)

Funding agency: DOE and NSF

Principal investigators: Dr. D. T. Wasan, Dr. D. Gidaspo, 312/567-3009

Location: Department of Chemical Engineering, Illinois Institute of Technology, Chicago, Ill.

Schedule:

DOE: August 1977 - July 1978, \$56,316

NSF: August 1978 - July 1979, \$62,000

Objectives:

The objective of the program is to determine the feasibility of separating micron-size particles from synthetic oil mixtures and coal-derived liquids by electrokinetic separation techniques. Of more relevance to SRC is the effort in emulsion characterization.

Accomplishments:

1. Bench-scale experiments have been conducted on emulsion characterization and foam stability. Professor Wasan is one of the few individuals in the country who is looking at emulsion characteristics. Most of his work is on petroleum crude, which is extremely viscous and resembles asphalt. The objectives of his experiments are to predict the conditions of stable emulsion formation and how to break these emulsions. Facilities to measure surface shear viscosity (as opposed to bulk viscosity), surface tension, and foam size are also available in his laboratory. With some modifications, most of these units could be used with coal slurries because of their similarity to petroleum crude. An instrument that can measure bubble growth and also the actual bursting of the bubble in an emulsion has been recently acquired in his laboratory. The data generated are fundamental but could be useful for SRC technology.
2. Fundamental settling (sedimentation) data for synthetic oil mixtures (alumina-xylene, alumina-tetralin, alumina-tetralin-xylene), both in the presence of a high voltage electric field and without the electric field, have been obtained as a function of particle size, dielectric constant and zeta potential. Preliminary settling experiments were conducted on coal liquid samples from pilot plants, viz., SRC, H-Coal, Synthoil, and Amoco. The high viscosity of the coal liquids caused serious problems in the experiments. No settling was achieved at room temperatures even with the application of high electric fields.

3. Based on surface charge characteristics of coal-derived particles, a technique called cross-flow electrofiltration has been developed at IIT. The experimental set-up at IIT has been used on H-Coal liquids and synthetic liquids and the rates of filtration have been compared with those obtained from conventional rotary drum pressure filters. The maximum rate of filtration from the rotary filter for the H-Coal liquid at 480 K (400°F) is about 20% higher than that for the electrofilter at room temperature. At higher temperatures, a higher filtration rate can be expected in the cross-flow electrofilter due to reduced viscosity. The device was intended to demonstrate the principle, and with sufficient modification the filtration rates can be significantly improved.

A configurational modification of the electrofilter is being worked on at the present, based on the dictates of a mathematical model which has been developed for settling behavior of nonaqueous slurries.

4. Sedimentation experiments on a synthetic liquid (alumina/tetralin) are being carried out in an experimental unit. The unit consists of a cylindrical stainless steel tube (schedule 80) surrounded by a cylindrical tube made of galvanized iron. Heat transfer oil flows through the concentric tube and heats the slurry in the stainless steel tube. The entire unit is mounted on a moving platform which can be raised or lowered to permit measurement of settling rate as a function of distance along the column length. The instrumentation is not adequate for coal slurries, and substantial modifications would be needed to test the system on coal slurries.

Need for testing results at larger scale:

Definitely.

Applicability to the demonstration project:

Minimal.

Relevance to SRC or coal liquefaction technology:

The research is relevant to SRC liquefaction technology to a small extent with respect to emulsion studies.

Capability to perform demonstration support work:

The Chemical Engineering Department at IIT is not adequately equipped to conduct large-scale experiments. The capabilities and facilities are suited for basic research in solids/liquid separation and emulsion characterization.

Impact of redirection/extension:

If redirected to include coal liquids, the IIT research could contribute to liquefaction technology, particularly in surface characteristics of particles, in coal slurries, and emulsion properties of coal slurries. The data would be basic but could be useful to liquefaction process developers.

A.4 Vapor/Liquid and Liquid/Liquid Separations

A.4.1 Introduction

In all stages of the SRC process in which vapor and liquid streams are separated, the phenomenon of prime importance is the definition of the quantity of each component that will report to each phase (i.e., vapor/liquid and liquid/liquid equilibria). Typical unit operations involved are vapor/liquid flash (pressure or vacuum), distillation, stripping, and dissolution. The specific areas of the SRC process in which equilibria data are of prime importance are:

1. Reactors/dissolvers (light gas solubility in process solvent and fraction of process solvent vaporized and therefore unavailable for hydrogen transfer)
2. Slurry letdown system (separation of light gases and coal-derived light ends from SRC and unreacted coal)
3. Vacuum fractionator (separation of heavy ends from lighter oils)
4. Solvent fractionator (final removal of other components from solvent before recycle)

It is important to realize the significance of vapor/liquid equilibria to plant costs. The area of most concern is the impact of the slurry letdown system on the remainder of the plant. This vapor/liquid separation downstream of the dissolver dictates the quantity and composition of the vapor stream going to purification and H_2 recycle and the liquid stream going to solvent recovery, solids/liquid separation, and product fractionation. If the vapor/liquid equilibria correlations available are in error by even a small amount, the remainder of the plant could be oversized by a large factor, thus incurring unnecessary capital costs.

The SRC demonstration plant design engineers currently rely on correlations for vapor/liquid equilibria that were developed for petroleum fractions in early 1960s, such as the Grayson-Streed and Chao-Seader correlations, modified by limited actual process data. Even though these are the best currently available relations, Chao and

Greenkorn at Purdue University have shown that the resulting estimates for vapor/liquid equilibria (i.e., K -values), if applied to coal liquids, are in error by as much as 40% for the standard correlations. It appears obvious that a research effort is warranted in this area.

A.4.2 Relevant R&D programs

Current research in vapor/liquid equilibria as related to coal conversion systems can be divided into four groups: (1) feed-pot flow systems, (2) batch autoclave systems, (3) classical batch systems, and (4) reactor flow systems. The feed-pot flow system is characterized by the ability to heat a process-generated liquid sample and to contact this liquid with a gas phase at pressure and temperature for a short period of time. Subsequently, the phases are separated and measurements made on the key components in both phases. The chief advantage of this type of system is that the residence time of the process fluids at temperature and pressure is minimal, thus avoiding or greatly reducing possible coking, degradation, or repolymerization. This method also allows closer simulation of process conditions than either of the batch systems. The chief disadvantage is that samples that have been withdrawn from a process, cooled, stored, and shipped must be reheated. The most significant public research utilizing a feed-pot flow system with heavy hydrocarbons and a few coal liquids is done by Chao and Greenkorn at Purdue University. They have built and successfully operated a flow vapor/liquid equilibria apparatus to measure the solubility of H_2 , CH_4 , and CO_2 in various hydrocarbon liquids (decane, benzene, tetralin, etc.). Measurements of the solubility of light gases (H_2 , CH_4) in a few EDS and SRC-II recycle liquids have also been made.

A batch autoclave system has been used by Harrison et al. at the Bartlesville Energy Technology Center (BETC). Harrison saturates a hydrocarbon liquid (SRC-II, tetralin, etc.) with helium, hydrogen, or a synthesis gas in a stirred autoclave. The pressure and temperature are closely regulated and, after the desired residence time, samples of each phase are taken and analyzed.

The classical batch system is typical of university-scale apparatus. These units are generally best suited for the study of model compounds but are rarely suitable for slurries. Three categories of apparatus are used: (1) Burnett-type constant volume units for measuring pressure-volume-temperature relationships, (2) variable volume equilibrium cells for measuring vapor/liquid equilibria, and (3) perturbation chromatographs, which can be used to measure K -values and phase transitions.

The Burnett apparatus is used at Oklahoma State University (OSU) and at Rice University. At OSU, R. L. Robinson is studying pressure-volume-temperature relationships of He, H₂, CO, CH₄, CO₂, H₂-CH₄, H₂-CO₂, and CH₄-CO₂. At Rice University, Riki Kobayashi built a Burnett device and has completed shakedown testing.

Variable volume equilibrium cells are available at OSU and at Rice. At OSU, Robinson is investigating vapor/liquid equilibria in systems such as methane-ethane-propane-toluene-1-methylnaphthalene.

A perturbation chromatograph has been assembled by Kobayashi at Rice University. In addition, J. M. Prausnitz (University of California at Berkeley) is measuring vapor pressures of pure components, dew points of tars, and vapor/liquid equilibria near ambient conditions for selected cuts of tars and pure compounds.

The reactor flow vapor/liquid equilibrium system was used by Grayson and Streed to obtain vapor/liquid equilibria data on hydrocarbon distillate fractions. In this system, fresh feed is generated in situ by a controlled reaction such as hydrocracking of gas oils or reaction of coal with solvent and H₂, followed by phase separation and direct analyses. This is by far the best system to use for the complicated mixtures found in coal liquefaction. In fact, it is the only applicable method at high temperatures where thermal degradation is possible. No experimenters were found to be using a system of this type, but a proposal has been submitted by ORNL.

Applicable research projects in the vapor/liquid equilibria and the liquid/liquid area are shown in Table A.4. Details are provided in Sect. A.4.4, except where otherwise noted.

Table A.4. Summary of vapor/liquid and liquid/liquid separations research projects

Principal investigator	Institution	Relationships measured
H. C. Chao	Purdue University, West Lafayette, Ind.	Vapor/liquid equilibria data
C. H. Harrison	Bartlesville Energy Technology Center, Bartlesville, Okla.	Vapor/liquid equilibria data
C. L. Robinson	Oklahoma State University, Stillwater, Okla.	Pressure-volume-temperature and vapor/liquid equilibria data
R. Kobayashi	Rice University, Houston, Tex.	Pressure-volume-temperature and vapor/liquid equilibria data
J. M. Prausnitz	University of California; Berkeley, Calif.	Dew points and vapor/liquid equilibria data
R. Tarrer	Auburn University, ^a Auburn, Ala.	H ₂ solubility
G. M. Wilson	Wilco Research Co., ^b Provo, Utah	Vapor/liquid equilibria data in a flow system; pressure-volume-temperature data
J. M. Barryman	PVT, Inc., ^c Houston, Tex.	Vapor/liquid equilibria data in a flow system
J. R. Cunningham	Brigham Young University, ^c Provo, Utah	Vapor/liquid equilibria data
	Institute of Gas Technology ^b	Vapor/liquid equilibria and pressure-volume-temperature data

^aDiscussed in Sect. A.1.4, "Reactors/Dissolvers."

^bDiscussed in Sect. A.8.4, "Physical Properties."

^cDetailed discussion not provided because of insufficient information. Contacts for these institutions are James M. Barryman, PVT, Inc., P.O. Box 36272, Houston, Tex. 77036 and John R. Cunningham, Thermochemical Institute, Brigham Young University, Provo, Utah 84602.

A.4.3 Conclusions and recommendations

As noted in Sects. A.4.1 and A.4.2, the impact of vapor/liquid and liquid/liquid separations on the sizing and capital requirements of the portion of the plant downstream of the slurry letdown system is potentially very great. The reason for uncertainty in these areas is a lack of directly applicable design data. The void is emphasized by the fact that designers of coal liquefaction plants are currently using modifications of design procedures that were developed in the early 1960s for petroleum liquids.

Of the public research currently ongoing, the most directly applicable information for the SRCDPP is being produced by Chao and Greenkorn at Purdue University. Other possible contributors in this area are Wilco Research and PVT, Inc. All of these produce vapor/liquid equilibria data in feed-pot flow systems. Wilco has been working under contract with Exxon to develop data for the EDS process; institutional capabilities are provided in Sect. A.8.4. PVT, Inc., has, in the past, provided vapor/liquid equilibria data on SRC materials to Gulf.

The role of researchers working with batch systems (i.e., Harrison at BETC) is definitely important. There are distinct advantages to batch systems in that they are generally simpler and easier to operate and, therefore, less expensive. The problem with these systems is that the residence time at temperature is often on the order of hours. Therefore, if the temperature is high enough to cause significant breakdown of the fluids, valid data are impossible to obtain. Much useful data can, however, be taken at less severe conditions.

The classical batch system is most useful for pure components or synthesized mixtures. In this respect, the classical systems are useful for developing correlations to test on the actual coal mixture systems. These systems are generally available at universities, where the greatest interest lies in training students. Universities are interested in research with more easily characterized synthetic mixtures that lend themselves to theoretical interpretations and analyses.

As is evident in the discussion above and in Sect. A.4.4, there is an abundance of equilibria research currently under way in the country.

The problems with respect to support of SRC DPP include a lack of overall coordination of the research effort and a lack of data being taken on actual hot-fresh coal liquids. Therefore, a working group should be formed to oversee and guide vapor/liquid equilibrium research in the coal conversion area, and a reactor flow vapor/liquid equilibrium system should be built and operated to measure vapor/liquid equilibria for coal liquid/syngas systems.

Further recommendations and details of the proposed work are given in the following.

Summary of recommendations

1. Additional funding to the vapor/liquid equilibria program at Rice University would not enhance its applicability to the demonstration project. (See Sect. A.4.4 for details.)
2. Dr. R. L. Robinson's work at OSU should be continued as planned. It is recommended that he concentrate on measuring solubilities of CO and H₂O because of the work at Purdue. (See Sect. A.4.4 for details.)

Programs to be extended and/or redirected

1. R. Harrison's work at BETC should be extended and the following modifications made. The funding level should be increased from ~\$75,000 to ~\$175,000 per year to allow for more coal liquids and additional correlation of solubility data (either by BETC or a consultant such as Prausnitz). Solubility data for Wilsonville filtrate material should be measured. Solubility of oxides of carbon and light hydrocarbons should be measured in addition to hydrogen. (See Sect. A.4.4 for details.)
2. The activities at Purdue should be expanded to include measurement of *K*-values, not just solubility. This is a minor change and should not drastically affect program plans. DOE funding of at least some of this effort is recommended. (See Sect. A.4.4 for details.)

New projects To ensure successful coordination of data acquisition and correlation, a working group should be formed for determination and correlation of vapor/liquid equilibria for coal-derived liquids.

Representatives from industry, government laboratories, and universities should form the group.

A program should be instituted to form a base for vapor/liquid equilibria research for the upcoming coal conversion industry. The ongoing research in the feed-pot flow, batch, and classical batch systems described in Sect. A.4.4 and above should be continued. Such work is of great value in developing correlations, but it should be supplemented by experimental determination of K -values for actual coal-liquid systems. The second facet of the proposed work is the experimental determination of K -values in a reactor flow vapor/liquid equilibrium system (i.e., the coal liquid is generated in situ). The equipment components in this system include (1) a liquid or coal/solvent slurry feed tank and delivery system, (2) a gas supply system, (3) a reactor/dissolver, (4) vapor/liquid separators, (5) sample receivers, (6) on-line analytical equipment, and (7) support analytical equipment. The important advantages of the reactor flow system are minimization of effects of cracking and depolymerization and of sampling and aging effects due to taking a pilot plant sample, cooling it, and then reheating at the experiment site.

It is recommended that the proposed new experiment be located either at a national laboratory or an energy technology center. Either of these can provide large and diversified base of experimental expertise. They also provide the long-term continuity in manpower needed to form a coherent base for an experimental program that universities generally do not have. In addition, the job of obtaining high-quality, quantitative data is best left to small-scale experimenters, who can more precisely control conditions. However, pilot plants should be used to obtain data to check and verify any correlations obtained.

The funding for this program should be about \$750,000 per year for three to five years.

A.4.4 R&D efforts listed by institutions

Category: Vapor/Liquid and Liquid/Liquid Separations

Contractor: Bartlesville Energy Technology Center

Funding agency: DOE

Principal investigator: R. H. Harrison, FTS: 735-4321

Location: Bartlesville, Okla.

Funding level schedule: FY 1977, \$75,000; FY 1978, \$75,000; FY 1979, \$75,000.

Objectives:

To measure the solubility of hydrogen and possibly that of synthesis gas in selected process solvents.

Accomplishments:

Built an apparatus to measure solubility of H₂ in solvents. Measured solubility of helium in tetralin, hydrogen in tetralin; four-component mixture of 2-methyl naphthalene, p-cresol, 4-picoline and tetralin; SRC-II heavy distillate; and SRC-II mid-distillate. Data are obtained on vapor as well as liquid phase, and, therefore, K-values can be calculated.

Applicability to demonstration project:

Directly applicable.

Relevance to SRC or coal liquefaction technology:

Solubilities and K-values for H₂ and synthesis gas are necessary for almost any coal liquefaction project.

Relation of project to other programs:

This project uses less sophisticated equipment than do those at OSU or Rice, but it appears more relevant because process solvents are being used. This work complements the other projects discussed.

Capability to perform demonstration support work:

Excellent, but somewhat limited by batch system because of possible cracking and/or repolymerization. Also, data correlation work is minimal and done external to BETC (by the University of Florida).

Program plans:

To continue solubility measurements of H₂ and synthesis gas in various coal liquids such as H-Coal and EDS. The level of effort will continue at ~\$75,000 per year.

Impact of redirection/extension:

It is recommended that the funding level be increased to allow for additional correlation of the data. It is further recommended that solubility data be measured on filtrate from Wilsonville, which will be more representative of the material present in the slurry letdown system. In addition, the program should be expanded to include solubility data for oxides of carbon and light hydrocarbons.

It is anticipated that this additional work will require no more than \$100,000 per year increase in funding and result in insignificant changes in the schedule.

Category: Vapor/Liquid and Liquid/Liquid Separations

Contractor: University of California at Berkeley

Funding agency: DOE/FE

Principal investigator: J. M. Prausnitz, 415/642-3592

Location: Chemical Engineering Department, Berkeley, Calif. 94720.

Funding schedule: Completed June 1, 1979.

Objectives:

1. Dew points of tar vapors — This project is directed at developing a technique for estimating dew points of the gases evolved from coal gasification processes. It is necessary to be able to estimate the dew point of these streams to prevent fouling and eventual plugging of down-stream heat exchangers. It is relevant to off-gas treatment systems and vapor heat exchangers in coal liquefaction systems.
2. Solubility of gases in coal liquids — The objective of this work is to build and operate a device to measure the solubility of gases (H_2 , CO, CH_4) in coal-derived liquids. It is a batch system with short residence times (minutes). The device is operated at low temperatures and pressures. Solubility theory will be used to extrapolate to process conditions the results obtained at low temperatures and pressures.

Accomplishments:

1. Dew points of tar vapors — A method has been developed for the characterization of coal gasification tars to facilitate dew point calculations. The tars are fractionated into narrow-boiling-range pseudocomponents. Each fraction is then chemically classified with elemental analysis and with proton nuclear magnetic resonance photometry. Based on these chemical classifications, methods have been developed to estimate the vapor pressures and molecular weights required to predict tar condensation conditions.

A gas-saturation apparatus was built to measure vapor pressures at near-ambient temperatures in the range of 10^{-1} to 10^{-3} torr. Liquids such as *n*-octadecane ($C_{18}H_{38}$), *n*-eicosane ($C_{20}H_{42}$), 1-methyl-naphthalene, and 2-ethyl-naphthalene and solids such as anthracene, naphthalene, and phenanthrene have been tested. These vapor pressure measurements, coupled with chemical characterizations, are useful in dew point calculations.

2. Solubility of gases in coal liquids — The apparatus for measuring solubility of gases in coal liquids at near-ambient conditions has been assembled and shakedown has been completed.

Relevance to coal liquefaction:

Methods for estimating dew points, vapor pressures, molecular weights, and solubilities are useful information for gasification/liquefaction processes in general. Relevance to SRC could be enhanced if measurements were made with SRC liquids.

Relation of project to other programs:

These studies complement efforts of various researchers around the country in areas of vapor/liquid equilibria, vapor pressure measurements, and chemical characterization.

Capability to perform SRCDPP support work:

Support for the demonstration project is not probable from work currently under way at Berkeley. However, Prausnitz has the manpower (graduate students) necessary to analyze massive amounts of data and to test new theories and correlations on the data. In addition to his eminence in this field, he has expressed an interest in the work. This would be a relatively low-cost item because only salaries for graduate students and one professor, along with computer time, would be involved.

The vapor/liquid equilibrium apparatus is a batch system and therefore may be subject to thermal cracking and repolymerization. This apparatus is also limited in pressure capabilities.

Impact of redirection and extension:

Dr. Prausnitz's expertise in thermodynamics should definitely be utilized; he is most interested in modeling data taken by others and in proposing new theories to explain the behavior and interaction of particular fluids. It would be beneficial to use Dr. Prausnitz as a consultant to external experimental programs. These programs would obtain data for coal liquids similar to those obtained for petroleum fractions by Grayson and Streed. The experimental program could be performed by laboratories experienced in this work, and the theoretical aspects could be handled by one of the country's leading thermodynamicists. Prausnitz could also utilize the pure component data being obtained at universities for comparison and thus develop consistent correlations for heavy hydrocarbon coal liquids.

Category: Vapor/Liquid and Liquid/Liquid Separations

Contractor: Oklahoma State University

Funding agency: DOE

Principal investigator: Robert L. Robinson, 405/624-5280

Location: School of Chemical Engineering, Stillwater, Okla. 74074.

Funding level: FY 1977, \$81,600; FY 1978, \$63,000; FY 1979, \$51,800;
FY 1980, \$27,000; FY 1981, \$27,000.

Objectives for FY 1977-1979:

The thermodynamic properties of fluid mixtures are required for accurate design of equipment in which such fluids are stored, transported, or separated. The basic goal of the current project is to obtain data for components and conditions for which information is lacking. The mixtures studied will approximate those that occur in the processing of coal-derived fluids.

The research is divided into three major areas:

1. Design, construction, and operation of a Burnett-type apparatus for measurement of volumetric properties (pressure-volume-temperature-composition relationships) of gases. Measurement of the properties of the pure substances and selected mixtures of H_2 , CO, CO_2 , CH_4 , and H_2O at conditions from ambient to 700 K (800°F) and 13.8 MPa (2000 psia) or to the maximum temperature at which chemical reactions are unimportant.
2. Design, construction, and operation of a variable-volume, windowed, phase-equilibrium cell capable of operation of 700 K (800°F) and 13.8 MPa (2000 psia). Measurement of vapor/liquid equilibria in mixtures composed of selected light hydrocarbons heavy aromatic hydrocarbons and water.
3. Testing and/or development of predictive methods for estimation of volumetric properties and phase equilibria in mixtures of the type studied experimentally. Presentation of results in formats suitable for use in process design calculations.

Accomplishments:

1. Phase equilibrium measurements — Completed study of the system composed of methane, ethane, propane, toluene, and 1-methylnaphthalene. K -values were measured for each of the components at 378 K (220°F), 411 K (280°F), 444 K (340°F), and 478 K (400°F) at pressures between 0.69 and 13.8 MPa (100 and 2000 psia).

2. Volumetric property measurements - Completed studies include:

He	50, 100°C
H ₂	100, 200°C
CO	100°C
CH ₄	100, 200, 250°C
CO ₂	100°C
H ₂ -CH ₄	100°C
H ₂ -CO ₂	100°C
CH ₄ -CO ₂	100°C

All measurements were made at pressures to 14.2 MPa (140 atm).

Need for testing of results at larger scale:

Equilibria and volumetric property data are not a function of the size of equipment and, therefore, no scale-up is indicated.

Applicability to demonstration project:

Pressure-volume-temperature and vapor-liquid equilibria data are necessary for an accurate design of the demonstration plant. Results obtained to date are on pure component systems, which limits their direct application to design.

Relation of project to other programs:

This work complements the work at Purdue University and overlaps the planned work at Rice University.

Capability to perform demonstration support work:

OSU is a likely candidate for making pressure-volume-temperature and vapor-liquid equilibria measurements that would be complementary to measurements made in a feed-pot flow or reactor flow system.

In a batch system, many measurements on a fluid can be made in a short period of time over a wide range of conditions. This type of measurement is very useful if the chemical reaction region in the fluid is avoided.

Program plans:

The following work has been proposed to DOE for funding.

Objectives:

The overall objective of the proposed project is to produce data and correlations of value for the design, operation, and optimization of coal liquefaction processes. Specific objectives include:

1. Measurement of vapor/liquid phase equilibria in multicomponent systems composed of nonhydrocarbons, light paraffins, and aromatic solvents that will simulate systems occurring in coal liquefaction processes
2. Measurements on selected binary systems required to fix parameters in correlation models applicable to coal-fluid systems
3. Testing, extension, and development of adequate correlations for phase equilibrium predictions

Impact of redirection/extension:

Dr. Robinson's work should be continued; however, the level of funding listed is inadequate. A level of about \$100,000 per year would more reasonably cover these efforts. It is recommended that Robinson concentrate on CO and H₂O solubility data because of the work currently being performed at Purdue. Vapor/liquid equilibrium and pressure-vapor-temperature measurements on coal-derived liquids and associated vapors would be useful, and Robinson has expressed a reserved willingness to do these. However, since batch solubility data for coal liquids are currently being obtained elsewhere, the advisability of undertaking new efforts in this area is questionable.

Category: Vapor/Liquid and Liquid/Liquid Separations

Contractor: Purdue University

Funding agency: EPRI and NSF

Principal investigators: K. C. Chao and R. A. Greenkorn (317) 479-2593

Location: School of Chemical Engineering, West Lafayette, Ind. 47907.

Funding schedule and objectives: Table A.5.

Accomplishments:

1. Phase equilibrium in coal project — A flow-type equilibrium apparatus capable of measuring vapor/liquid equilibria and vapor pressures up to 700 K (800°F) and 27.7 MPa (4000 psig) has been installed, tested, and operated. Vapor/liquid equilibria of more than 30 binary mixtures, including a light gas (H_2 , CH_4 , or CO_2) and a heavy hydrocarbon of the type found in coal liquids, have been measured. Typical liquids include decane, benzene, tetralin, quinoline, and 9, 10-dihydrophenanthrene. High-temperature vapor pressures have been measured for each of the solvents used. Solubilities involve H_2 and CH_4 in EDS and SRC-II recycle solvents. The EDS liquids were 478 to 505 K (400 to 450°F) and 533 to 589 K (500 to 600°F) cuts, whereas the SRC-II liquids were 533 to 549 K (500 to 528°F), 589 to 607 K (600 to 632°F), and 644 to 672 K (700 to 750°F) cuts. Additional data at 703 K (430°C) were taken for the coal liquids. Solubility data were correlated with the augmented hard sphere model, Soave equation of state, and by extension of Chao-Seader correlation. Typically, equilibrium-saturated liquid and gas compositions and K -values are determined at four temperatures [462.8, 541.9, 621.8, and 662.3 K (189.6, 268.7, 348.6, and 389.1°C)] and at seven pressures [2, 3, 5, 10, 15, 20, and 25 MPa (20, 30, 50, 100, 150, 200, and 250 atm)].
2. Kinetics of coal liquefaction project — Operated two microbatch reactor systems (one capable of continuous operation) and a gas and liquid chromatograph. Developed a kinetic model to aid experimental analysis and classification of donor solvents.
3. Thermodynamics of fluid system — A total-pressure apparatus has been constructed to measure vapor/liquid equilibria for binary liquid mixtures. A solution model based on conformal solution theory has been used to correlate excess properties of binary liquid mixtures and the K -values of mixtures in which components differ greatly in size. Vapor/liquid equilibria data have been gathered for binary liquid mixtures (i.e., cyclohexane and n -pentane).

Tables A.5. Purdue University funding schedule and objectives

Funding agency	Amount	Period covered by award	Project title	Objectives
Electric Power Research Institute ^a	\$349,338	1/1/76-12/31/79	Phase Equilibrium in Coal	Measure phase equilibria of coal liquids
National Science Foundation ^a	99,300	9/1/76-8/31/79	Thermodynamics of Fluid Systems	Develop molecular structure correlation of organic liquids
Gulf Oil Company ^a	10,000	9/1/77-8/31/78	Kinetics of Coal Lique- faction	Study hydrogen-donor-acceptor reactions for coal liquefaction
	4,000	9/1/78-12/31/78		
School of Chem. Engineering, ^b	5,000	1/1/79-6/30/79		
Purdue Engineering Exp. to Purdue Research Foundation	15,000	7/1/79-6/30/81		
Environmental Protection Agency ^b	186,530	10/1/77-3/31/79	Assessment of Environment and Health Impacts of Control Options for Cadmium and Its Compounds	Conduct system study of pollutants from processes

^aK. C. Chao, principal investigator.

^bR. A. Greenkorn, principal investigator.

A gas/liquid perturbation chromatograph has been constructed to measure infinite dilution activity coefficients between 333 and 453 K (60 and 180°C) and to operate between 0.2 and 3.5 MPa (15 and 500 psig). A group-contribution molecular model has been developed to predict the properties of pure liquids and liquid mixtures. The model is applicable to polar and nonpolar materials.

Need for testing of results at larger scale:

Equilibria data preclude the need for testing on a larger scale. However, the results should be compared with routine vapor and liquid compositions on the slurry letdown streams at Wilsonville, Ft. Lewis, and/or Harmarville.

Applicability to demonstration project:

The correlation of the vapor/liquid equilibria data will probably not be completed in time for use in the plant design per se. But some of the data will be of great use to both the design and the interpretation of results from its operation. Correlating the vapor/liquid equilibria data from gas/heavy hydrocarbon systems will give an indication to the designer as to how well the Chao-Seader, Grayson-Streed, and similar equations, and modifications thereof, represent coal liquids. The solubility in coal liquids data can also be utilized by the designer. Comparison between their data and that of the pilot plants answers many questions, such as: (1) Is the developed correlation applicable to coal liquids? (2) Does the presence of solids and/or repolymerization in the slurry letdown system affect the equilibrium concentrations? (3) Does equilibrium actually exist in the separator tanks?

Relevance to SRC or coal liquefaction technology:

Vapor/liquid equilibria correlations are necessary for design of equipment (in any type of coal liquefaction process) in which gas and liquid phases exist simultaneously.

Relation of project to other programs:

This program complements other thermodynamic projects at OSU, Berkeley, and BETC. However, this program should be considered the primary source of data at this time.

Capability to perform demonstration support work:

The equipment is a continuous flow system (liquid rates from 500 to 2000 ml/h) with holdups in the equilibrium cell ranging from 12 to 30 s. This reduces, if not eliminates, the possibility of thermal cracking and/or repolymerization. The temperature and pressure can be consistently controlled to within 1 K (1°C) and 0.03 MPa (0.3 atm) respectively. The reproducibility of the data is excellent. Evidence to support the contention that equilibrium is actually achieved is as follows:

1. Sample compositions are independent of flow rates within the range of flow rates employed.
2. Data on hydrogen/benzene from the apparatus agree with results obtained by Connolly (1962) from a static apparatus.
3. Data on hydrogen/tetralin satisfy the Gibbs-Duhem equation.

The expertise at Purdue is respected in the academic and industrial community. The quantity, quality, and comprehensive analysis of the data make the institution a primary source of coal liquid vapor/liquid equilibria data.

Program plans:

1. Phase equilibrium in coal project — Vapor/liquid equilibria involving mixtures of light gas (H_2 , CH_4 , and CO_2) in heavy hydrocarbons or recycle solvents.
2. Kinetics of coal liquefaction — Donor kinetics of model compounds: tetralin/benzothiophene, benzofuran, indole, indene.
3. Thermodynamics of fluid systems — Vapor/liquid equilibria data will be taken on several binary liquid mixtures whose components are hydrocarbons with five to eight carbon atoms. Infinite dilution activity coefficients will be measured for heavy stationary phases.

Two new projects that have been proposed for funding are shown in Table A.6.

Impact of redirection/extension:

It is recommended that this work be continued essentially as planned. However, a minor change in data acquisition/analysis is indicated. The primary thrust of this program is to obtain solubility data, whereas designers require K -values. The procedure should be modified to include measurement of K -values as well as solubilities, particularly for coal liquids. This redirection is within the capabilities of the institution and should require no additional funding. DOE should consider partially funding these efforts because of the high quality and relevance to other DOE liquefaction projects.

Table A.6. Purdue University proposed projects

Funding agency	Amount	Period covered by award	Project title	Objectives
National Science Foundation ^a	\$250,761	9/1/79-8/31/82	Thermodynamics of Fluid Systems	Extend molecular model to sulfur, nitrogen, and water. Determine vapor pressure of organics.
Department of Energy ^a	545,588	6/1/79-5/31/82	Transport and Thermodynamic Properties of Coal Liquids	Measure and correlate transport and thermodynamic properties of coal liquids and model compounds

^aK. C. Chac and R. A. Greenkorn, principal investigators.

Category: Vapor/Liquid and Liquid/Liquid Separations

Contractor: Rice University

Funding agency: EPRI and DOE

Principal investigator: Riki Kobayashi

Location: George R. Brown School of Engineering, Houston, Tex. 77001.

Funding schedule:

<u>Funding agency</u>	<u>Amount</u>	<u>Period covered by award</u>
EPRI	\$327,000	12/1/75 to 12/1/78
DOE	\$247,973	5/1/76 to 5/1/79

At this time, all the funding has been spent, in addition to about \$250,000 worth of equipment furnished by Rice. The EPRI contract has been extended for one year with a funding of \$25,000. The DOE contract has been extended for two years at \$49,999 per year.

Objectives:

The objectives for all projects were the same: to study high pressure vapor/liquid equilibria, phase transition, adsorption, diffusion, viscosity, densities, and volumetric behavior of gases and liquids for pure compounds.

Accomplishments:

The installation and shakedown of one high-pressure perturbation chromatography apparatus and two static Burnett-isochoric pressure-volume-temperature apparatuses.

Applicability to demonstration project:

None, due to the anticipated length of time required for data generation and analysis.

Relevance to SRC or coal liquefaction technology:

If the forthcoming data are of sufficient quantity and quality, the program will be applicable to vapor/liquid equilibria correlations.

Relation of project to other programs:

Work performed at Rice University would complement other programs in this area.

Capability to perform demonstration support work:

The equipment is excellent and can be operated at temperatures up to 720 K (450°C) and pressures of 50 MPa (500 atm). However, these are static systems, and data taken should be in support of a dynamic system.

Program plans:

Objectives are as follows:

1. Measure vapor pressures of pure compounds (yet to be determined) from the triple point to the critical point
2. On the high-pressure chromatography apparatus: (a) phase transition studies in the hydrogen-2-methylantraquinone systems to pressures as high as 31 MPa (4500 psi); (b) *K*-value studies for light hydrocarbons, NH_3 , H_2S , CO_2 , CO , HCl , H_2O , and COS , at infinite dilutions in the hydrogen-2-methylantraquinone systems up to 31 MPa (4500 psi); (c) similar studies on selected gas mixtures such as hydrogen with methane or ethane
3. On the pressure-volume-temperature and vapor/liquid equilibrium apparatus: (a) obtain dew point and bubble point data on the hydrogen-tetralin system at 462.8, 541.9 and 393.2 K (189.6, 268.7 and 120°C); (b) obtain hydrogen solubilities in the hydrogen-2-methylantraquinone system and the homogeneous phase densities along with the vapor pressures of methylnaphthalene

Impact of redirection/extension:

Additional funding of this program would not enhance its applicability to the demonstration project.

A.5 Gasification of SRC Residues

A.5.1 Introduction

Current SRC designs include an oxygen-blown gasification plant to produce makeup hydrogen from coal liquefaction residues. Tests have been conducted with various SRC-I and SRC-II residue streams in the Texaco gasifier at the Montebello Research Laboratory. These have been short-term runs to date, limited under the current contract to less than a week for the maximum run type. Questions concerning long-term steady-state operation remain, with particular emphasis on gasifier refractory resistance to the very high molten slag loadings resulting from residue gasification. The question should be raised as to whether a heat sink (e.g., water wall) might be needed to form a solid, protective slag layer on the refractory surface for reliable, long-term operation. In this event, a major design change would be involved, and the ranking of the Texaco entrained-flow gasifiers relative to others for use in the SRC demonstration plants could change.

A.5.2 Related R&D programs

The most directly relevant program for supporting design efforts in SRC DPP for residue gasification is the Texaco Montebello program. This and a number of related programs are summarized in Table A.7. Additional details on several of these programs are listed by sponsoring institution in Sect. A.5.4.

A.5.3 Recommendations

With regard to the Texaco gasifier, the current DOE-Texaco contract, which was scheduled to expire in July 1979, calls for the following three types of tests (listed following Table A.7) under the specified conditions.

Table A.7. Gasification of SRC residues

Funding agency	Contractor	General description	Other project information	Applicability
Ruhrchemie AG & Ruhrkohle AG		1.66-kg/s (158-TPD) pilot plant Operated with coal/H ₂ O slurry since January 1978	Located in Oberhausen-Holten, West Germany. P = 4 MPa (580 psia). WHB used	Feed is different, and very little information has been published. Continuous runs to 300 h
DOE	Texaco, Inc.	Types 1, 2, and 3 tests (20 lb, 20 bbl, and 200 bbl, respectively) using coal liquefaction residual materials	Located in South El Monte, Calif. at Texaco's Montebello Research Laboratory. Principal investigator is A. M. Robin. Maximum test duration (current contract) is 5.6 d	Limited to date. Tests include type I for SRC ash concentrate and type II for SRC-II vacuum-flash drum bottoms (7.3-h run)
EPRI	Texaco, Inc.	Studies of H ₂ production from coal liquefaction residues	Located in South El Monte, Calif. at Texaco's Montebello Research Laboratory. Principal investigator is A. M. Robin. In reported work, various high-ash SRC-I and H-Coal residues were tested	Limited to date. Maximum run durations were 14 h (SRC-I case) and 19.3 h (H-Coal case)
EPRI	Texaco, Inc.	Establish detailed data base for gasification combined-cycle power production using the Texaco gasifier	For water slurries of Illinois No. 6 coal. Ongoing	Feed is different, and product is fuel gas, not H ₂
EPRI	Texaco, Inc. and Systems, Science & Software	Detailed steady-state and dynamic modeling of Texaco-type gasifier	Coal-fired case. Ongoing. Systems, Science, & Software principal investigator is T. Blake	Promising in areas of data evaluation and gasifier scale-up
EPRI	Under negotiation	Reliability prediction methodology for Texaco-based gasification combined-cycle power generation	Coal-fired case	Limited to later validation when data become available
EPRI	Westinghouse Electric Corp.	Develop refractories with improved high-temperature erosion/corrosion resistance	Includes slagging gasifier case, up to 2030 K (3200°F). Ongoing	Potential in the future

Table A.7 (continued)

Funding agency	Contractor	General description	Other project information	Applicability
EPRI	Stanford Univ.	Coal slag phenomena, experimental and analytical	Emphasis on open-cycle magnetohydrodynamics. Ongoing	Potential in the future
DOE	West Virginia Univ.	Steady-state modeling of Texaco gasifier fed with coal liquefaction residues	Principal investigator is C. Y. Wen (Chemical Engineering Dept.). Ongoing	Direct when the model has been validated
DOE	MIT	Properties of coal slags	Principal investigator is J. F. Elliott. Emphasis on volatilization of highly volatile species	In a general sense

Test type	Scope	Amount of feed (lb)	Feed rate (lb/h)	Cost per test ^a (\$)
1	Laboratory analyses, performance estimates, and pumpability testing	20	Not applicable	650
2	Short-term gasification tests	8000 (20 bbl)	600 to 1000	65,000
3	Longer term gasification tests; thermal efficiency analysis; further definition of optimal operating conditions	80,000 (200 bbl)	600 to 1000	195,000

^aFor CY 1979, including cost of reporting.

Note that the maximum test duration, based on maximum charge and minimum feed rate, is only 5.6 days. This contract, which is currently being renegotiated for an additional three years of testing, has resulted in valuable preliminary results. The follow-on contract for gasification of coal liquefaction residues is expected to provide for 7- to 10-day sustained gasification runs in addition to the types listed in the above table. The equipment needed to provide a continuous supply of molten residue to the gasifier is being installed. We recommend future single continuous runs of at least 15 days (preferably 30 days) to develop realistic information about gasifier refractory resistance to the high molten slag loadings involved. The cost of such runs is the subject of ongoing negotiations. It would obviously be higher than that for type 3 tests because of the increased duration recommended. To provide perspective, the ORNL SRC assessment team was told during a visit to EPRI that the West German group operating the larger Texaco pilot plant at Oberhausen-Holtien has estimated the cost for a 30-day test with a residue feed rate of 136 Mg/d (150 TPD) at about \$4 million.

A.5.4 R&D efforts listed by institution

Category: Gasification of SRC Residues

Contractor: Texaco, Inc.

Funding agency: DOE

Principal investigator: A. M. Robin, 213/699-9048

Location: South El Monte, Calif.

Funding level: Varies with number of tests conducted (see text).

Schedule: Contract through June 1979.

Objectives: To determine operational parameters for Texaco gasification of SRC-I, SRC-II, and other residues.

Accomplishments: Several type 1, 2, and 3 tests (20 lb, 20 bbl, and 200 bbl respectively) using SRC ash concentrate and SRC-II vacuum-flash drum bottoms have been conducted and the results published.

Need for testing of results at larger scale: Only at longer durations.

Applicability to demonstration project: Very direct.

Relevance to SRC or coal liquefaction technology: Direct.

Relation of project to other programs: Broad.

Program plans: Dependent on new (post-FY 1979) contract.

Impact of redirection and extension: Can be accommodated (plus or minus a few months). However, Texaco has not expressed a willingness to commit the Montebello facility to a long run (about 15 to 30 days). This is said to result from interference with other Texaco programs of such long runs, rather than from equipment limitations.

Category: Gasification of SRC Residues

Contractor: Texaco, Inc.

Funding agency: EPRI

Principal investigator: A. M. Robin, 213/699-0948

Location: South El Monte, Calif.

Funding schedule: Work completed.

Objectives: To quantitatively study H₂ production from coal liquefaction residues.

Accomplishments: Several tests using high-ash SRC and H-Coal residues have been conducted and the results reported. Maximum run duration was about 20 h.

Need for testing of results at larger scale: Longer duration tests needed.

Applicability to demonstration project: Very direct.

Relevance to SRC or coal liquefaction technology: Direct.

Relation of project to other programs: Broad.

Program plans: Dependent on future contractual arrangements.

Category: Gasification of SRC Residues

Contractors: Texaco, Inc., and Systems, Science, and Software

Funding agency: EPRI

Principal investigators: A. Lupa, Texaco; T. Blake, Systems, Science, and Software

Location: Texaco, Houston, Tex.

Schedule: Ongoing.

Objectives: To develop a detailed steady-state model for coal-fed Texaco gasifier (by Texaco); same for dynamic case by Systems, Science, and Software.

Accomplishments: First versions of the models have been developed and are being documented and tested.

Need for testing of results at larger scale: Probably none.

Applicability to demonstration project: Direct, in areas of data evaluation and gasifier scale-up.

Relevance to SRC or coal liquefaction technology: Broad.

Relation of project to other programs: Related to model development by C. Y. Wen at West Virginia University.

Program plans: To continue comparisons of model predictions with experimental data; model simplification is the primary goal.

Category: Gasification of SRC Residues

Contractor: Westinghouse Electric Corp.

Funding agency: EPRI

Location: Pittsburgh, Pa.

Schedule: Ongoing.

Objectives: To develop refractories with improved high-temperature erosion/corrosion resistance.

Accomplishments: Preliminary correlation developed for erosion of refractories by synthetic (model) slags.

Need for testing of results at larger scale: Probable.

Applicability to demonstration project: Potential.

Relevance to SRC or coal liquefaction technology: Broad (future).

Relation of project to other programs: Complementary.

Program plans: Validation of refractory erosion correlation with natural coal slags.

Category: Gasification of SRC Residues

Contractor: West Virginia University

Funding agency: DOE

Principal investigator: C. Y. Wen, 304/293-2111

Location: Morgantown, W. Va.

Objectives: To develop a steady-state model for Texaco gasification of coal liquefaction residues.

Accomplishments: A report (*Entrained-Bed Coal Gasification Modeling*) that describes the model and initial data comparisons has been issued.

Need for testing of results at larger scale: Continue data comparisons.

Applicability to demonstration project: Direct.

Relevance to SRC or coal liquefaction technology: Broad.

Relation of project to other programs: Related to modeling efforts by Texaco and Systems, Science, and Software for the coal-fed case.

Impact of redirection/extension: Probably not needed.

A.6 Slurry Mixing

A.6.1 Introduction

The slurry mixer must take coal of a certain size, blend it with process solvent in fairly precise proportions, and feed this mixture to a fired heater (through a heat exchanger in the SRC-I design). The process recycle stream must be cooled to the mixer temperature, and the mixer effluent stream should be heated to provide feed to the fired heater at elevated temperatures. Mixing at pilot plant scale has been carried out at low temperatures and pressures [Wilsonville at ~ 330 K ($\sim 130^\circ\text{F}$) and Ft. Lewis at ~ 450 K ($\sim 350^\circ\text{F}$), both near atmospheric pressure].

However, demonstration plant design calls for elevated temperatures to reduce heat duties on heat exchangers and significantly increase the thermal efficiency of the overall process. These higher temperatures create a critical need for high-temperature blending data. For instance, if the mixing temperature can be raised to 500 to 530 K (450 to 500 $^\circ\text{F}$), the heat saving is considerable (SRC-I could eliminate feed slurry heat exchangers; SRC-II could remove recycle slurry exchangers). However, as temperatures are increased, the slurry goes through a plastic stage, with a sharp rise in viscosity that creates a serious problem for the mixing and pumping equipment and can cause plugging of lines and valves. Viscosity vs temperature vs residence time data are needed to predict and to avoid the plastic stage. Also, the type of mixing device for blending wet, crushed coal with recycle solvent at high temperatures has not yet been determined.

A.6.2 Relevant R&D programs

Additional data are needed for SRC demonstration plant designs because of the large potential for process savings vs the potential for process problems caused by the plastic properties of coal mixtures at elevated temperatures. This is clearly an area in which R&D, if properly directed, can make a valuable contribution in a short period of time. Considerations to be addressed are:

1. Slurry properties for 841- μm (20-mesh) and larger coal size
2. Viscosity changes with temperature

3. Viscosity changes with residence time
4. Gelling tendencies as a function of time and temperature
5. Effects of changes in feed coal and solvent characteristics
6. Effects of varying solvent/coal ratio
7. Agitation rate effects

The first consideration is important to current research only if a larger coal feed size is retained as a design option for demonstration plants. If not, it remains for longer-term research.

Currently, only one university and the two SRC pilot plants are doing work in this area. P. L. Walker, Jr., and others at Pennsylvania State are working a small project to determine the effect of coal agglomeration as a function of agitation rate. However, this continuing project, though both interesting and worthwhile, can have little impact on the SRCDPP slurry mix design.

The SRC-I plant at Wilsonville, Alabama, is physically limited in this area by equipment. This plant has previously operated at only 330 K (130 °F) and at a slight vacuum. Researchers there are planning to blend recycle solvent and a Kerr-McGee Critical Solvent Deashing unit solvent in a separate tank that has been heated to raise the mix tank temperature to 355 K (180 °F), its upper limit. They have installed steam coils on the slurry-mix tank to maintain this temperature. These conditions are not adequate for SRCDPP design, and cost and time considerations severely limit Wilsonville's contributions in this area.

The SRC pilot plant at Ft. Lewis has run tests with exit temperatures of up to 467 K (380 °F) in the SRC-II mode and up to 444 K (340 °F) in the SRC-I mode. More data should be forthcoming if planned mixing studies are carried out. Efforts to measure viscosity vs temperature and the effect of particle size on viscosity and solvent uptake in a bench-scale Haake viscometer are also under way. Under the Ft. Lewis contract, P&M has also proposed comparison of vortext and plug mill mixing systems. Ft. Lewis is the logical place to perform pilot experiments for the slurry-mix area, but it cannot be efficiently used for screening studies.

A.6.3 Conclusions and recommendations

The nature and number of parameters that require analysis before demonstration plant design necessitate pilot plant scale investigation; however, considerable assistance could be provided by supporting studies at laboratory scale. A number of experiments to determine viscosity vs temperature vs residence times for a distribution of particle sizes could be run more quickly at laboratory scale and used as input to determine the best operating conditions for the Ft. Lewis mixers, which have limited versatility and longer turnaround times. Also, a number of small-scale mixers could be screened so that only the best ones were recommended for testing at the pilot plant. The following are conclusions and recommendations:

1. The slurry mix temperature should be increased because of the very favorable effect of increased temperatures on process efficiency and thus on economics
2. Priority should be given to the solution of the most significant problem with the proposed operating conditions — the unknown temperature-time-particle size relationship for the gel transition range and the accompanying increases in viscosity
3. Mixing technology that would allow a higher exit temperature should be demonstrated at pilot scale
4. Supporting laboratory-scale studies should be started to provide
  - a. Viscosity vs temperature vs residence time data for a variety of particle size distributions
  - b. Screening of relevant types of mixing devices

All data should be obtained specifically to provide needed input for the pilot program to extend its capability to demonstrate devices in time to affect demonstration plant design.

A.7 Heat Exchangers

A.7.1 Introduction

The designs of heat exchangers used for heating and cooling slurries in the SRC-I demonstration plant are currently based on very limited data. The same is true of the recycle slurry cooler potentially required for the SRC-II demonstration plant. Heat transfer, rheology, fouling, corrosion, and erosion information would be directly useful in design efforts for the SRCDPP. Information on such thermal and physical properties as heat capacity, thermal conductivity, density, and viscosity, is included under the problem areas of "Fired Heaters" (Sect. A.2) and "Physical Properties" (Sect. A.8). Recommendations for acquiring additional properties data are also provided in those sections.

A.7.2 Related R&D programs

Excluding physical and thermal properties programs, only a few programs have been identified as offering information potentially relevant to heat exchanger design in the severe SRC demonstration plant services. These are summarized in Table A.8, with some additional details provided in Sect. A.7.4.

A.7.3 Recommendations and conclusions

1. A well-instrumented shell-and-tube or double-pipe heat exchanger should be installed on the dissolver outlet stream at the Ft. Lewis pilot plant. The data acquired would be relevant to the SRC-I demonstration plant design, in which a heat transfer fluid (such as Dowtherm A) is used to recover heat for use elsewhere in the process. A vertical orientation capable of both upflow and downflow operation appears to be preferable. For a 92.9-m² (1000-ft²) stainless steel unit, the cost of the optimum heat exchanger for relevant temperature and pressure conditions is estimated at \$55,000. Including installation and operating costs for several months, the total cost could be about \$100,000.

Table A.8. Summary of heat-exchanger research projects

Funding agency	Contractor	General description	Other project information	Applicability
DOE	Lawrence Berkeley	Alloy test program. Determine erosion/corrosion caused by coal slurry flows at 340 to 480 K (160 to 400°F) and V 3 to 12 m/s (10 to 40 fps)	Principal investigator, A. V. Levy, Berkeley, Calif. Through 1981 at \$130,000 per year.	Direct
NSF & AIChE	Univ. of Illinois	Characteristics of two-phase reactor flow. Emphasis on annular regime. Horizontal now and vertical later	Principal investigator, T. J. Hanratty. Phase distribution and heat and mass transfer for two-phase flows	Potentially useful
DOE	Argonne National Lab.	Flow-induced tube vibration in shell-and-tube exchangers.	Principal investigator, H. Halle. Subcontractor — Heat Transfer Research, Inc. Project near completion	Useful

2. Additional verification of existing multiphase flow-regime maps for vertical and horizontal tube flow is needed. (This is discussed in Sect. A.2.3, "Fired Heaters for Slurries.")
3. Tests should be conducted to determine shell-side fouling rates in rod-baffle exchangers relative to those in standard segmentally baffled units. Indirect (field) data indicate that such rates are lower in rod-baffle exchangers. Areas of application would encompass all dirty shell-side flows with the exception of concentrated liquid/solid mixtures. Two small heat exchangers, one with standard segmental baffles and the other with rod baffles, each with about 15 m^2 (160 ft^2) of stainless steel surface area and designed for 530 K/2.2 MPa (500°F/300 psig), would cost a total of about \$25,000. Including the heat exchanges, a one-year test program conducted, for example, by Phillips Petroleum Co. (Bartlesville, Okla.) or by HTRI (Alhambra, Calif.), might involve a total cost of about \$125,000.
4. A modest effort to extend past work on enhancing laminar-flow heat-transfer rates to industrial-scale equipment conditions seems warranted. This would apply primarily to tube-side flows of relatively clean but viscous liquids. A one-year test program conducted at the university level might reasonably be expected to cost about \$100,000 to \$150,000.

Increased emphasis should be placed on correlation of data available from future as well as current programs, which use conditions representative of SRC demonstration design heat exchangers.

A.7.4 R&D efforts listed by institution

Category: Heat Exchangers

Contractor: Argonne National Laboratory (HTRI, subcontractor)

Funding agency: DOE

Principal investigator: H. Halle

Location: Argonne, Ill.

Schedule: Continuing.

Objectives: To determine design criteria to prevent flow-induced tube vibration in shell-and-tube heat exchangers.

Accomplishments: See published reports.

Need for testing of results at larger scale: None.

Relevance to SRC or coal liquefaction technology: General.

Program plans: The program is nearing completion.

Category: Heat Exchangers

Contractor: University of Illinois

Funding agency: National Science Foundation, possibly American Institute of Chemical Engineers (Design Institute for Multiphase Processing)

Principal investigator: T. J. Hanratty, 217/333-1318

Location: Urbana-Champaign, Ill.

Funding level: \$60,000 per year, through CY 1979; about one-third in the two-phase flow area.

Objectives: To study two-phase, Newtonian, air/H₂O flows in horizontal pipes. Pressure changes, drop entrainment, and film thickness will also be studied.

Accomplishments: Early information is appearing.

Need for testing of results at larger scale: Considerable.

Applicability to demonstration project: Slight now; more direct later, if continued.

Relevance to SRC or coal liquefaction technology: General.

Relation of project to other programs: Complementary, if extended.

Program plans: Unclear as of December 1979.

Category: Heat Exchangers

Contractor: Lawrence Berkeley Laboratory

Funding agency: DOE

Principal investigator: A. V. Levy, 415/486-5822

Location: Berkeley, Calif.

Funding level: \$130,000 per year through CY 1981.

Schedule: 340 K (160°F) operation now; to 480 K (400°F) in fall 1979.

Objectives: To determine erosion/corrosion of various metals caused by coal-slurry flows at 3 to 12 m/s (10 to 40 fps) and 340 to 480 K (160 to 400°F) in a 50-mm-diam (2-in.) pipe loop and ancillary experiments.

Accomplishments: First conclusive results just becoming available.

Need for testing of results at larger scale: Probably none.

Applicability to demonstration project: Direct.

Relevance to SRC or coal liquefaction technology: Direct.

Relation of project to other programs: Complementary.

Program plans: Funding through CY 1981 anticipated; uncertain thereafter.

Impact of redirection/extensions: Directly supportive. Continuation coupled with expansion is desirable. Should include longer radius return bends in test program.

A.8 Physical Properties of Coal-Derived Materials

A.8.1 Introduction

Numerous researchers are conducting basic property measurements of coal-derived liquids and pure compounds identified in coal-liquid fractions. However, with the exception of the SRC pilot plant participants, very few researchers are measuring basic properties of coal-solvent slurries at conditions close to the SRC demonstration plant design. Such information, if obtained in time, could lead to significant improvements in the design of SRC process units, eliminate operational problems, and provide the necessary data base for the development of reliable engineering correlations for scale-up.

The R&D assessment team visited a selected number of institutions involved in physical property measurements to investigate the nature and extent of the research. Selections were made based on the relevance of the research at the institution to the SRC demonstration plant. However, some other institutions that could provide information useful to the SRC process and, in some cases, be directly supportive of the demonstration plant project were also visited. Table A.9 provides a summary of the R&D efforts in the physical property area. Apparently, it is at the pilot plants, energy technology centers, and national laboratories that much of the physical property R&D activity is conducted. Industrial research centers that may be developing this information typically consider it proprietary and it is not generally available. Universities are primarily involved in fundamental liquefaction research with most of the work based on model compounds; few are working directly on coal liquids and thus much of their work is not useful to the Demonstration Plants Program.

A.8.2 Relevant R&D programs

Although many aspects of the SRC process have been verified at the pilot plants, there remain uncertainties with regard to the design, scale-up, and operation of the individual units in the SRC process. Many of these uncertainties can be eliminated, or at least reduced, by obtaining the necessary basic property data on the SRC process streams. Most of

Table A.9. Summary of research on physical property data relevant to SRC demonstration plant

Physical property	Relevance	Institution	Description of research	Remarks
Viscosity	Direct	Pittsburg & Midway Coal Mining Co. (P&M), Merriam, Kan.	DOE funded. Autoclave experiments to measure viscosity of coal solutions at temperatures <673 K (<400°C) and moderate hydrogen pressures. Subsequently used a Dynatrol viscometer to measure viscosity at temperatures up to 673 K (400°C) and reactor pressures close to preheater operations	Can be directly supportive of SRC. A continuous flow reactor is also available to obtain rheological data in a continuous operating mode
Viscosity (and slurry preheater data)	Indirect	Argonne National Laboratory (ANL)	Slurry viscosity, heat transfer, flow regimes, and heat of reaction	Direct support of Synthoil Program. Final report ANL/CEN/FE-78-7, January 1979. Equipment and facilities could be used after modifications to obtain useful data on flow characteristics of gas-liquid, gas-coal-slurry mixtures under SRC conditions. Heat transfer coefficient measurements are also possible with the current setup
Viscosity	Indirect. Direct to SRC technology support	Auburn Univ.	Data on viscosity and peak-viscosity region in preheaters	Research is part of an overall program on the fundamental aspects of SRC process chemistry. Information is useful and supportive of SRC process technology
Viscosity	Direct	P&M, Ft. Lewis	Measurements of slurry viscosities and coking temperatures of heavy liquids; slurry preheater design data for several coal types	Direct support of SRC demonstration. A test program has been proposed on the new preheater being installed at Ft. Lewis, which will include development of correlations for various flow patterns under different conditions in the preheater. Viscosity measurements should be a key item in this proposed program

Table A.9. (continued)

Physical property	Relevance	Institution	Description of research	Remarks
Viscosity	Indirect	Wilco Research Co.	Measurements of hydrocarbon fractions viscosities, including fractions saturated with hydrogen	Direct support of Exxon Donor Solvent (EDS) process
Viscosity	Indirect	Brigham Young Univ. (BYU)	Measurements of viscosities at experimental conditions ranging to 700 K (800°F) and 21 MPa (3000 psia)	Direct support of EDS process
Viscosity	Direct support to SRC process technology	Oak Ridge National Laboratory (ORNL)	Bench-scale coal liquids flow system currently operating, measuring viscosity of coal solvent slurry liquefaction streams at SRC process conditions. Viscosity measurements are made with a pipeline and a falling-ring viscometer. A coal slurry preheater test section is also in operation for rheological and heat transfer measurements	Physical property measurements and slurry preheater studies can provide the necessary data for developing useful correlations for coal-solvent slurries at SRC process conditions
Density	Direct support to SRC process technology	ORNL	Density measurements on coal solvent slurry will be made in the bench-scale coal liquids flow system with a gamma absorption liquid density gauge, which is currently undergoing operational checkout. Accuracy is 0.002 specific gravity units over a 0.5 to 1.2 span	Data generated for SRC process streams should be useful for design
Density	Indirect	Wilco	Density measurements on hydrocarbon fractions up to 645 K (700°F)	Direct support of EDS process
Density	Indirect	BYU	Density measurements at severe experimental conditions	Direct support of EDS process
Solubility	Direct	Bartlesville Energy Technology Center (BETC)	Solubility of hydrogen in a heavy SRC-II distillate in the pressure range 5.1 to 25.1 MPa (725 to 3625 psig) and temperature range 398 to 673 K (125 to 400°C)	The effort is part of an ongoing project on thermodynamic studies on coal conversion processes. The program at BETC should provide useful data for SRC processes. Data are useful and directly relevant to SRC and of importance in coal liquefaction technology

Table A.9. (continued)

Physical property	Relevance	Institution	Description of research	Remarks
Solubility	Direct	Auburn Univ.	Solubility data for H ₂ , CH ₄ , and CO ₂ in SRC recycle solvent at process conditions	Data useful and relevant to SRC and liquefaction in general
Solubility	Direct	Purdue Univ.	Solubility of CH ₄ , CO ₂ , H ₂ S, H ₂ O, and NH ₃ in liquid recycle solvent from liquefaction processes	Important information, which could be used to develop useful correlations for vapor/liquid/equilibria in SRC process
Heat capacity	Mostly indirect; useful for SRC process technology	BETC	Heat capacity measurements have been made on SRC, H-Coal, and two distillates from CONOCO Coal Development Company. The data are the most extensive on coal-derived fluids	Specific heat measurements on coal-derived liquids over a wide temperature range are possible at BETC. The heat capacity laboratory is well instrumented and automated. The research is part of an ongoing program on thermodynamic studies of coal conversion processes
Heat capacity	Useful for SRC technology	ORNL	Heat capacity measurements on coal slurry streams will be obtained in a coal liquid flow system at SRC process conditions using a flow calorimeter	The system is instrumented; heat capacity instrument design is complete. Data should be useful for SRC process and will span a wide range of temperature and other variables
Heat capacity	Indirect	Wilco	Heat capacity measurements on hydrocarbon fractions up to 645 K (700°F)	Direct support of EDS process
Thermal conductivity	Applicable to SRC process technology	ORNL	Thermal conductivity measurements are to be based on the transient heated wire technique. Test liquid thermal conductivity is obtained from the temperature-time response of a thin wire undergoing constant electrical heating input while immersed in a body of test liquid. The slope of wire temperature vs log time plot is inversely proportional to liquid thermal conductivity up to the onset of natural convection	Thermal conductivity data on coal solvent slurry at SRC process conditions will be obtained at the coal flow test unit, along with other properties. The data will be useful in developing correlations. Pressure vessel for instrument constructed, electronics available, thermal conductivity unit under construction

Table A.9. (continued)

Physical property	Relevance	Institution	Description of research	Remarks
Enthalpy	Currently indirect. Useful for coal conversion technology	Colorado School of Mines	Enthalpy measurements have been made on samples of coal-derived liquids, principally Char-Oil Energy Development Syncrude from Western Kentucky coal, Char-Oil Energy Development distillate, a Synthoil distillate, and one SRC product. Measurements have been made in Freon boil-off calorimeter in the range of 290 to 700 K (65 to 800°F) and 0.1 to 13.8 MPa (15 to 2000 psia). Work is continuing on developing correlations for coal liquids	Research could be directly supportive of the demonstration project with some redirection, particularly if the measurements were made on SRC-I and SRC-II process streams. With minor modifications in the ancillary equipment, this could be accomplished
Enthalpy	Currently indirect	BETC	Enthalpy measurements are made on H-Coal distillates, distillates obtained from CONOCO Coal Development Co., and samples from SRC-II process by differential scanning calorimetry	Research could be directly supportive of the demonstration project, if redirected
Enthalpy	Direct	Wilco	Enthalpy measurements while heating coal slurry and hydrogen from 535 to 672 K (500 to 750°F). Attempting to simulate contact time, heat flux, etc., of actual process	New project directly supports SRC; studying effects of heating rate, H ₂ /slurry ratio, and slurry-to-oil ratio on enthalpy
Vapor/liquid equilibrium	Indirect, very useful if redirected	Purdue Univ.	Vapor-liquid equilibria measurements involving more than 30 binary mixtures containing a light gas (H ₂ , CH ₄ , CO ₂) and a heavy hydrocarbon of the type found in coal liquids have been made in a flow-type equilibrium apparatus capable of measuring vapor-liquid equilibria up to 700 K (800°F) and 27.7 MPa (4000 psig). Plan to obtain vapor-liquid equilibria data on mixtures of light gases and SRC, EDS recycle solvents	The vapor-liquid equilibria research at Purdue is extensive and could be very useful to SRC demonstration project if more data are generated on SRC mixtures

Table A.9. (continued)

Physical property	Relevance	Institution	Description of research	Remarks
VLE	Indirect; could be directly applicable if redirected	Institute of Gas Technology, Chicago, Ill.	Measuring phase equilibrium properties of Syngas-Benzene-Toluene-Xylene mixtures for application to high-Btu coal gasification plants. Equipment is a flow system capable of measuring phase equilibrium in pressure range of 0 to 20.7 MPa (0 to 3000 psia) and temperature range of 310 to 533 K (100 to 500°F)	IGT has given the modifications and associated costs to adapt the equipment to coal liquefaction (see Sect. A.8.4)
VLE	Direct	Wilco	Vapor-pressure and vapor-liquid equilibria measurements of pure compounds and hydrocarbon fractions, with light gases also, up to 727 K (850°F) for EDS. Vapor-liquid equilibria measurements related to high-pressure aqueous/sour-gas equilibria for SRC	Direct support of EDS in recent work. Now attempting to simulate process conditions for high-pressure aqueous absorption of ammonia from sour gas stream for SRC-II
VLE	Indirect	BYU	Measure vapor-liquid equilibria at severe experimental conditions	Direct support of EDS process
Emulsion characterization	Indirect	Illinois Institute of Technology, Chicago, Ill.	Bench-scale experiments on emulsion characterization, foam stability, interfacial tension, and surface shear viscosity on petroleum crude	Research is fundamental in nature but could be redirected to coal slurries. One of the few institutions involved in study of emulsions
Surface tension	Indirect	Wilco	Surface tension of hydrocarbon fractions and H ₂ -saturated fractions, up to 589 K (600°F)	Direct support of EDS process
Surface tension	Indirect	BYU	Surface tension measurements at severe experimental conditions	Direct support of EDS process

the available data are at conditions far removed from SRC demonstration design conditions. Correlations to extend the data to this range are either nonexistent or are based on petroleum-derived materials, which have questionable applicability to coal-derived materials.

The properties listed in Table A.10 have been identified as specific to the SRCDPP.

Slurry physical property data are being measured at the SRC pilot plants and at other liquefaction process installations, for example, the EDS facility at Baytown, Texas. The literature on slurry viscosity for the SRC process indicates that data are available up to 480 K (400°F) but are limited beyond this temperature. Gulf/P&M at Ft. Lewis and at Harmarville are two SRC locations at which slurry properties could be monitored over a wide range of conditions.

The Oak Ridge National Laboratory has one of the few programs on coal physical properties and slurry preheaters in which the relevant properties are being or will be measured on coal liquids in a flow system, under conditions close to SRC operating conditions. Viscosity measurements are being made at process conditions, although the shear rates have not yet exceeded 650 s^{-1} . The physical and transport properties (viscosity, density, thermal conductivity, and specific heat) should be suitable for general design purposes, including preheaters. The test setup at ORNL is described in Sect. A.8.4.

The coal liquefaction support studies conducted at ANL for the Synthoil process provide equipment and expertise that could be used for two-phase heat transfer and fluid-flow studies. The 450-kg/d (1/2-ton) coal test slurry unit built for the Synthoil Program at ANL can be operated at pressures of up to 27.7 MPa (4000 psig) and temperatures of up to 730 K (850°F). A brief description of the test module is provided in Sect. A.8.4. The test facility could be used to measure precise heat transfer coefficients at incremental sections, a capability particularly useful for predicting the heats of reaction along the SRC preheaters.

Exxon Engineering, Florham Park, New Jersey, has responsibility for determining which feed-slurry and product-stream physical properties are needed for the EDS Program. A thorough study has been made of the

Table A.10. Physical properties and operating ranges specific to SRC demonstration plants

Property	Operating range	Relevance	Remarks
Viscosity	400 to 730 K (250 to 850°F)	Improved characterization of viscosity, viscosity peaks, and gel formation in the front end of the process (SRC mixers, preheater, dissolver/reactor, and heat exchangers)	Critical for SRC demonstration plants and especially important for design, operation, and scale-up of front-end systems
	10 to 1000 s ⁻¹ shear rate		
	30 to 40 wt % coal (up to 50 wt % solids)		
	Swelling/nonswelling coals at design particle size up to 841 μ m (20 mesh)	Flow regime prediction, flow stability in three-phase operation in the SRC preheater, and pressure drop in the flow systems	
	Up to 17.3-MPa (2500-psig) pressure	Characterization of coal/oil solubility preheater, reactor, and slurry exchangers	
	Time dependence	Heat transfer coefficients in slurry preheater, reactor, and slurry exchangers	
Thermal conductivity	Same as for "viscosity"	Thermal properties in general will govern fouling in heat exchangers and coking in preheaters	Direct measurements on slurries desirable for design and scale-up although a weighted average thermal conductivity for the slurry would be estimated based on measured thermal conductivities of solid and liquid components at process conditions
Specific heat and enthalpy	Same as for "viscosity"	Net heat of reaction in the front end of the process	Very limited data exist for coal liquids
		Reaction kinetics in dissolver/reactor	
		Useful in estimating thermal load for optimum design of these units	
Viscosity (and fusion point) of vacuum-tower bottoms	Temperature range 530 to 700 K (500 to 800°F)	Affects liquefaction bottoms quality and feed to the gasifier	Critical to process operation and has to be correlated with feed properties
	10 to 2000 s ⁻¹ shear rate	Pressure drop in system	

Table A.10 (continued)

Property	Operating range	Relevance	Remarks
Solubility of gases (H ₂ , CO, CH ₄ , NH ₃ , etc.) in oil/coal slurry and vapor-liquid equilibria	20.7 MPa (3000 psi) total (H ₂ partial pressure up to 13.8 MPa, 2000 psi); 370 to 730 K (200 to 850°F) range	Critical for gas/liquid separators and feed to the fractionators in the processes. Also influences wastewater compositions. The data on hydrogen/coal liquid is also important in the slurry preheater and dissolver from a reaction energetics standpoint	Critical area in the SCRCDDPP

rheological properties of the EDS process feed and product streams (Quarterly Report, November 1978), and future work pertaining to SRC could benefit from the guidelines and experimental designs used for the EDS process. Exxon has reported a contract with Brigham Young University (BYU) for experimental determination of many properties in the EDS process. Density, viscosity, and surface tension data were and are being obtained at BYU on EDS process fluids over a range of conditions. Vapor/liquid equilibria studies are also being conducted by BYU on model compounds. Experimental conditions at BYU are stated to range up to 700 K (800°F) and 21 MPa (3000 psia). Results are to be presented at the National AIChE meeting, June 1980, in Philadelphia, by S. C. Hwang and C. Tsionopoulos of Exxon, J. R. Cunningham of BYU, and G. M. Wilson of Wilco. Subsequent work has also been performed by Wilco Research Company (headed by Grant Wilson, formerly with the Thermochemical Institute at BYU). Details of the programs at Wilco are provided in Sect. A.8.4. Other sources of property data contracted by Exxon are the Southwest Research Institute (density), Exxon's Baytown R&D Laboratory (viscosity) and Imperial Oil's Research Department in Ontario, Canada (heat capacity), in addition to Wilco and BYU.

Auburn University has conducted autoclave experiments on slurry viscosity changes during heat-up and dissolution of coal/solvent slurry. Although the measurements have been made on SRC-I coal liquids and under conditions comparable to the SRC process (up to 450°C), the data have not been obtained at in situ conditions. Typically, the samples were heated and exposed to the desired temperatures and shear rates, but the viscosity measurements were made at a low temperature (90°C). Viscosity data from autoclave experiments can differ from data obtained in flow systems because the heat-up rates are different in the two systems. This difference can significantly affect the slurry composition, particularly at high temperatures. Furthermore, the limits of measurable shear rates can differ in the two systems. However, the data generated at Auburn University should be useful and applicable to SRC processes.

The Institute of Gas Technology (IGT) in Chicago expressed interest in physical property and heat transfer measurements on coal-liquid slurries. They have acquired a slurry preheater (discussed in Sect. A.8.4) that

could be operated in the range of interest to the SRC process. The dimensions of the preheater and the operable heat flux are reasonable for scale-up purposes.

Extensive enthalpy measurements on coal liquids are being carried out at the Bartlesville Energy Technology Center. There is an ongoing project on thermodynamic studies of coal conversion processes in which heat capacity measurements of coal liquids and solubility of hydrogen in heavy process solvents are being measured. The center is well equipped and the technical staff has an extensive background in hydrocarbon research and characterization. The enthalpy and thermodynamic property measurements on coal liquids have definite applicability to the SRC process and are of considerable relevance to the demonstration plant.

The Colorado School of Mines is another institution with ongoing enthalpy measurements of coal-derived liquids. Professors Kidnay and Yesavage have designed, constructed, and operated an excellent Freon boil-off calorimeter operable at temperatures of 290 to 700 K (65 to 800°F) and pressures to 13.8 MPa (2000 psia). Data have been successfully generated on coal-derived liquids, but only one SRC product has been investigated. Correlations are being developed for the coal-liquids investigated and are being compared with correlations for petroleum liquids. There is a viscosity limitation with the current apparatus that could be corrected for future work.

The research on vapor/liquid equilibrium, solubility of hydrogen and other gases in coal liquids, and PVT data are discussed principally in Sect. A.4. To summarize here, the major program in this area is at Purdue University. Vapor/liquid equilibrium on coal gasification streams is measured by IGT, and their lab also has the capability of redirecting the research to liquefaction streams. (See Sect. A.8.4 for IGT institutional data.)

Fundamental research on emulsion characterization and stability, interfacial tension, and surface shear viscosity on petroleum crude fractions is being conducted at the Illinois Institute of Technology (IIT) in Chicago. Experiments are conducted in bench-scale laboratory systems; the applicability of data for larger systems is questionable. However, the emulsion and surface tension properties could be important

in the reactor/dissolver and in the slurry letdown system because of gas and liquid hold-up, foaming, and hydrodynamic patterns. The program at IIT could possibly aid in the solution of these problems.

A.8.3 Conclusions and recommendations

Continued laboratory and pilot plant efforts are needed to extend the current data base on physical properties of coal-liquid slurry streams at SRC process conditions. The pilot plants should provide a firm engineering data base for the demonstration plants and should also define solutions to operability and reliability problems associated with the individual process units in the demonstration plants. However, experience indicates that an important part of normal process development is a continued R&D effort in bench-scale systems and subsequent testing of the results in the pilot plants. Data on physical properties can also be acquired at institutions other than the SRC pilot plants. Some R&D programs (as indicated earlier in Table A.9) are already being pursued at the bench-scale level, and the research in almost all cases is directly useful in SRC technology development. In some cases, the results are also directly supportive of SRCDPF programs.

Data obtained from flow systems are generally more appropriate for design and scale-up purposes than are measurements made in autoclaves. There are only a selected number of institutions (see Table A.9) that are measuring, or have the capability to measure, basic properties in flow systems. Currently, the SRC pilot plants and ORNL are the only institutions obtaining data on slurry properties at SRC process conditions. There should be a continuation of these efforts over a two- to three-year period at current or higher funding levels. The ORNL study should be redirected specifically to SRC process streams, and the experimental conditions should be extended to cover the operating ranges cited in Table A.10. Data for selected cases from the ORNL setup should be checked periodically at the pilot plants to confirm their adequacy. A continuation of the efforts at ORNL (in conjunction with the fired heater study discussed in Sect. A.2.4) is planned through FY 1980 at a funding level of \$465,000.

A program could be initiated at ANL to develop heat transfer data in conjunction with the rheological data for three-phase flow. The equipment at ANL is well instrumented for studying on an incremental basis the variations in slurry heat transfer and flow patterns that have not yet been investigated. The net heat of reaction, heat of solution of coal, effects of H_2 partial pressure on the reaction kinetics in the preheater [particularly at higher velocities, above 5 m/s (15 fps), and high heat fluxes, above $31,500 \text{ W/m}^2$ ($10,000 \text{ Btu/h}\cdot\text{ft}^2$)], and limits of coking tendency on swelling and nonswelling coals in the particle size range up to 841 to $595 \mu\text{m}$ (20 to 30 mesh) are largely unknown at this time. Although these issues will be considered in the SRC pilot plants, the information obtained from the ANL setup should be coordinated with the pilot plant efforts for maximum benefit. The cost for modifications, shakedown, and restart of the setup at ANL for SRC conditions is estimated at \$500,000 over a nine-month period. Continued support will be necessary over a two- to three-year period at a funding level of about \$200,000 to \$250,000 a year to obtain relevant data for developing heat transfer correlations and characterizing the flow patterns at process conditions.

IGT has a coal slurry preheater that could be operated at SRC process conditions. Measurements of viscosity, pressure drop, and heat transfer pertinent to SRC slurry preheaters could be obtained in the test loop. The cost estimate provided by IGT for conducting heat transfer/rheology studies in their preheater is about \$400,000 over a two-year period. Design correlations for heat transfer in terms of fluid properties could be developed from such an effort. IGT has analyzed the data from SRC pilot plants and has made a detailed investigation of the applicability and limitations of available heat transfer correlations for SRC preheater design. This investigation was conducted as part of an ongoing project on the preparation of a coal conversion systems technical data book. The expertise and capabilities to conduct the study are available at IGT, and it could be one of the institutions considered for the slurry heat transfer/rheology investigation.

The enthalpy measurements at BETC should be continued at current funding levels, and more emphasis should be placed on SRC-derived coal liquids. The facility is well equipped to provide a sound data base on enthalpy/specific heat of SRC process streams. BETC is also one of the more qualified energy technology centers to serve as coordinator for ongoing and future enthalpy and thermodynamic property measurement programs on coal liquids. Several of the national laboratories could also serve this function. The enthalpy measurements at the Colorado School of Mines would be supportive of the demonstration plants if measurements are made on SRC-I and SRC-II liquid streams. However, the current funding level through FY 1979 is low (\$75,000) and should be increased to \$200,000 through FY 1981 to support such efforts.

The recommendations pertaining to solubility and vapor/liquid equilibrium measurements are described in Sect. A.4 of this report and are not repeated here.

In summary, the following recommendations are suggested in the area of physical property measurements:

1. Physical property data (viscosity, density, thermal conductivity, and specific heat) of coal slurries are inadequate for design and scale-up purposes of SRC front-end units. These properties should be measured under controlled conditions for a range of operating parameters. Institutions other than pilot plants should also be involved in such measurements because the data obtained from pilot plants are not always amenable for developing correlations, primarily because of interaction between system parameters.
2. The ongoing program at ORNL on physical property measurements is a sound approach to monitoring physical and transport properties simultaneously with reactions occurring in a flow system. The data obtained clearly will be useful for developing design correlations.
3. Heat transfer/rheology studies can be investigated in small-scale preheaters such as the ones available at ANL, IGT, and ORNL. See Sect. A.2 for additional comments.

4. Enthalpy data on coal-derived liquids from SRC process streams are inadequate and need to be measured and correlated with liquid properties. The inadequacies of petroleum-derived correlations should be identified and corrected, where possible, based on new data. The institutions currently involved in these measurements (e.g., BETC and Colorado School of Mines) should play a major role in this area.
5. The pilot plants should be involved in in situ measurements. However, their ability to obtain data for developing correlations is limited because of their relative inflexibility as compared with the institutions conducting bench-scale experiments. Nonetheless, the plants can serve an important role in testing the data and correlations from bench-scale efforts.
6. There is a need for coordination of the research on physical property measurements. A multifunctional research location, such as a national laboratory or an energy technology center, should play a coordinating role in this area.

A.8.4 Important R&D efforts listed by institution

Category: Physical Properties

Funding agency: DOE

Institution: Argonne National Laboratory

Principal investigator: J. Fischer, 312/972-4389

Location: Bldg. 829, Argonne, Ill.

Objectives:

The objectives of the program were: (1) design, construct, and test a liquefaction-heat transfer coefficient measurement test unit to handle 450 kg/d (1/2 ton/d) of coal slurry. The heat transfer test module was a substitute for the normal preheater and reactor used in coal liquefaction processes. (2) Obtain experimental data on flow characteristics for upward flow of gas-liquid-solid mixtures in vertical tubes simulating the conditions in the Synthoil preheater.

Accomplishments:

The program was completed in August 1977. A description of the test unit and its capabilities are provided below.

The test module includes a slurry make-up system, slurry feed system, six heating elements (stainless steel piping; 6.35-mm ID), ten cooling elements, slurry letdown tanks, and power, flow rate, and temperature instrumentation.

The limits of operability of the test unit are:

Coal slurry	0 to 40% coal ground to 74 μm (200 mesh)
Liquid flow	Up to 0.023 m^3/h (6 gal/h) of coal slurry, non-Newtonian
Gas flow	120 to 2150 mole/h (100 to 1800 scfh)
Reynolds number (based on H_2)	1000 to 15,000
Pressure	0 to 27.7 MPa (4000 psig)
Temperature	733 K (460°C)
Heat flux	Up to 2 kW, equivalent to 142 $\text{kW}/\text{s}\cdot\text{m}^2$ (45,000 $\text{Btu}/\text{h}\cdot\text{ft}^2$) for first heater; less for subsequent heaters. Maximum flux up to 20 kW for all sections

The process mixture flows up and down in alternating directions through the six heating elements (vertically oriented stainless steel tubes) and then through the cooling elements. Two types of cooling elements are used in the same test unit. The first is a 1.8-m (6-ft) vertical straight tube cooler, similar to the heating elements, but with a shell around the tube to contain the air flow. The second type consists of approximately 6.7 m (22-ft) of tubing coiled in a helix within an air-cooling shell and has an overall height of about 0.6 m (2-ft). Both cooling types use air for heat removal. Thermocouples have been installed at 0.3-m (1-ft) intervals in both the heating and cooling sections of the test unit (a total of 300 thermocouples), which enables detailed measurement of heat transfer.

The operability of the process development unit was demonstrated by ANL with base numbers being established for the heat transfer coefficient of hydrogen in the system. The system has been tested on a coal-derived oil but has not been tested on coal slurries. Data on pressure drop, liquid hold-up, and flow patterns resulting from the upward flow of gas-liquid and gas-slurry mixtures in cylindrical tubes have been obtained from the flow characteristics experiments. Heat transfer data on creosote oil under various operating conditions were also obtained and have been analyzed to give average heat transfer coefficient values in the system during both heating and cooling.

Need for testing of results at larger scale:

The physical property data from the present setup would be adequate for scale-up. However, heat transfer coefficients from this unit would need to be confirmed in large-scale units, particularly in the SRC preheaters currently being installed at Ft. Lewis.

Applicability to demonstration project:

This type of information can be useful to verify flow characteristics over the small temperature intervals associated with each segment in the SRC front-end units. The implications can be especially significant in SRC preheaters, particularly in predicting gel formation and peak viscosity of the coal slurry. Furthermore, the unit could be used to study the heats of reaction along the length of preheaters, which have not been established to date.

Relevance to SRC or coal liquefaction technology:

The unit could provide valuable data in the general areas of heat transfer and flow behavior in SRC slurry preheaters; the unit is applicable to SRC and other liquefaction processes.

Program plans:

The Synthoil Test Unit is well instrumented but has not been operated since 1977 because of lack of DOE support for the Synthoil Program. The Argonne National Laboratory is eager to run the unit on coal slurries for SRC conditions and has provided a preliminary estimate of \$500,000 and a time scale of nine months to restart the unit for SRC conditions and to implement the necessary modification and shakedown. Continued support would be necessary over a two- to three-year period at a funding level of \$200,000 to \$250,000 a year to obtain the necessary data.

Category: Physical Properties

Funding agency: DOE

Institution: Bartlesville Energy Technology Center

Principal investigators: W. D. Good, Fossil Energy and J. E. Dooley,
Petroleum and Natural Gas

Location: Bartlesville, Oklahoma

Funding/schedule and objectives: (See Table A.11.)

Accomplishments:

1. Measurements have been completed on heats of combustion and heat capacities of six chars of different origins and four fractions of Synthoil derived from West Virginia coal, three fractions of Synthoil from Utah coal, and liquid fractions from H-Coal and Conoco. An apparatus has been built for measurement of solubility of hydrogen in solvents. Shakedown measurements with helium in tetralin were completed, and the system is being used for hydrogen solubility measurements in tetralin and a four-component synthetic solvent mixture.
2. Vapor pressure measurements have been completed in solid and liquid states in the range 13 to 3440 Pa (0.1 to 25.8 mm Hg) by means of inclined piston dead-weight gauge. Enthalpies of sublimation and vaporization have been derived at the melting temperature and at 398 K (125°C) for these compounds.
3. Heats of combustion have been determined on alkyl naphthalenes and alkyl indanes and factors contributing to the high heats of combustion have been evaluated. The compounds have special application in jet fuel.
4. Compositional data on syncrude derived from the H-Coal liquefaction process have been compiled to provide information on effectively refining the syncrudes to useful products. Coal liquid products produced in-house in batch autoclaves and two distillates and a residue from the zinc chloride liquefaction process (Conoco Coal Development Company) have been analyzed. Coal liquids from the SRC-II process have also been characterized.

Table A.11. Funding and objectives for physical properties research
at Bartlesville Energy Technology Center

Funding agency	Amount per year (\$)	Period covered to date	Project title	Objectives
<i>Fossil energy</i>				
DOE (Advanced Research & Supporting Technology)	250,000	1976	Thermodynamic Studies on Coal Conversion Processes	Provide engineering process design data for heat capacity and enthalpy of liquids and chars from various coal liquefaction and gasification processes Correlate liquid properties with thermodynamic properties of pure components Measure solubility of hydrogen in selected process solvents
DOE (Basic Energy Science)	75,000	1977	Thermodynamic Characterization of Condensed Ring Compounds	Provide comprehensive thermodynamic information (vapor pressure and enthalpy) on major compound types present in syncrudes from shale oil, coal, and petroleum
Air Force Office of Scientific Research	72,000	1976	Thermodynamic Properties of Organic Compounds	Determine thermodynamics properties (heat of vaporization, heat of combustion) and density of compounds in relation to their molecular structure
DOE (Advanced Research & Supporting Technology)	150,000	1976	Characterization of Syncrudes from Coal	Determine composition data for syncrudes derived from various liquefaction processes and characterize them in terms of refining compatibility for upgrading
DOE (Advanced Research & Supporting Technology)	400,00	1977	Automotive Testing of Synthetic Fuels	Determine problems and opportunities in most advantageously matching synthetic fuels and engines
DOE	250,000	1977	Characterization Studies: Heavy Ends	Develop characterization and analytical methods for the heavy ends of petroleum, shale oil, tar sands, and heavy oils
DOE	161,000	1977	Refining Process Technology	Determine refining characteristics of synthetic crude oils, type and quality of finished products potentially obtainable from various syncrudes

5. Experimental work has been conducted to generate technical data on synthetic fuels, particularly properties and characteristics that influence their utilization in internal combustion engines. Tests have been conducted on gasolines derived from shale and coal-derived syncrude.
6. Detailed compositional data have been obtained on shale oil, tar sand liquids, and heavy oils to develop processes that upgrade these materials. Correlations are being developed that relate structural features of these liquids to their geologic origin.
7. The refining characteristics of coal-derived crude oils and the type and quality of products are being analyzed in a bench-scale hydrogenation unit. The differences in refining techniques and the methods of conversion between petroleum crude oils and coal-derived crudes are being identified in this study.

Need for testing of results at larger scale:

None, since the results can be applicable as presently obtained.

Applicability to demonstration project:

The enthalpy measurements on coal-derived liquids would be directly applicable to the demonstration project if more data are compiled and correlations developed, specifically on SRC liquids.

Relevance to SRC or coal liquefaction technology:

The programs described are relevant to liquefaction technology and are aimed at improving the present state of the art in this important area.

Relation of project to other programs:

The work at BETC and the enthalpy work at the Colorado School of Mines are complementary, although the programs at BETC are of a much broader scope, with direct emphasis on coal liquids.

Capability to perform demonstration support work:

BETC has the capabilities to be a central research facility to conduct measurements and coordinate efforts on heat capacity and enthalpy of coal liquids. BETC maintains one of the world's most complete source of data on crude oils and a consistent reference on properties of gasolines, diesel fuels, jet fuels, and heating oils.

Program plans:

Continue ongoing program at same funding level and redirect the enthalpy and correlation efforts to SRC liquids.

Category: Physical Properties

Funding agency: DOE

Institution: Colorado School of Mines

Principal investigators: A. J. Kidnay and V. F. Yesavage, 303/279-0300

Location: Golden, Colorado

Funding: \$75,000 - 1979

Objectives:

The objective of this program is to obtain enthalpy data on coal-derived liquids and to develop reliable and accurate correlations for enthalpy prediction.

Accomplishments:

A freon boil-off calorimeter, capable of measuring fluid enthalpies in the range of 290 to 700 K (65 to 800°F) and 0.1 to 13.8 MPa (15 to 2000 psia), has been designed, constructed, and tested on coal-derived liquids. To date, 436 enthalpy determinations have been made on six samples of coal-derived liquids from the COED and SRC-I processes. These experimental measurements have been tested with existing correlations for petroleum enthalpies with mixed results. The samples low in aromatics are in substantial agreement with the existing petroleum correlations, while those high in aromatics are poorly represented with existing correlations. Work is in progress to determine the properties of coal liquids that cause the deviations in correlations. Correction factors to incorporate in the available correlations are being developed.

Need for testing of results at larger scale:

The enthalpy correlation on SRC coal liquid streams would have to be tested under conditions close to SRC demonstration plant conditions. The experimental measurements could be extended to 755 K (900°F) and 27.6 MPa (4000 psia) with some modifications in the ancillary equipment (pumps and other components).

Applicability to demonstration project:

The project would be supportive of the SRC demonstration plants if more measurements are made on selected SRC-I and SRC-II process streams.

Relevance to SRC or coal liquefaction technology:

The enthalpy work is relevant and important to SRC and other liquefaction processes as well.

Relation of project to other programs:

The work is similar to that being conducted at BETC. However, there is no apparent conflict, and the programs complement each other.

Capability to perform demonstration support work:

The results indicate that the program is cost effective. Work needs to be redirected to suit the demonstration plant needs.

Program plans:

The research should be continued at a higher funding level (~\$200,000 over a two-year period) and redirected to SRC liquid streams.

Impact of redirection/extension:

Some modification in the ancillary equipment would be needed to extend the work to higher temperatures and pressure ranges. The manpower requirements would have to be increased to enhance rapid data generation.

Category: Physical Properties

Funding agency: DOE

Institution: Institute of Gas Technology (IGT)

Principal investigator: A. L. Talwalkar, 312/567-3755

Location: Chicago, Illinois

Schedule: May 1, 1979 to October 1980, \$570,000 per year.

Objectives:

Provide a single comprehensive data book for the design and evaluation of coal conversion systems.

Accomplishments:

Periodically published available data on coal conversion systems in quarterly and annual reports. With respect to coal liquefaction, IGT has compiled available data on slurry preheaters and physical properties of coal liquids.

Applicability to the demonstration project:

Can provide indirect support by way of compiling the available data, testing correlations, and identifying data limitations in SRC-I and SRC-II.

Relevance to SRC or coal liquefaction technology:

Direct relevance — specific data have been compiled and correlated with respect to SRC technology.

Relation of project to other programs:

The work involves surveying current programs in fossil energy and compiling information on coal conversion processes; hence, it complements most of the coal conversion programs.

Capabilities to perform demonstration support work:

- (1) Vapor-liquid equilibria studies on coal liquids,
- (2) Slurry preheater studies,
- (3) Center for compiling available and pertinent information on coal liquid streams from SRC pilot plants.

The technical staff at IGT has fossil energy experience in several multidisciplinary fields that can be useful to the SRCDDP.

Program plans:

The work on the coal conversion data book should be continued as scheduled. The current vapor-liquid equilibria support studies for coal gasification could be redirected to coal liquefaction streams. The modification and costs are provided by IGT as per the following attachment. The preheater could be used for basic heat transfer and flow property measurements as stated in the attachment.

ATTACHMENT PROVIDED BY IGTIGT Capabilities in Phase Equilibrium Studies
as Applied to Coal Liquefaction

IGT is presently measuring the phase equilibrium properties of syngas-BTX mixtures for application to high-Btu coal gasification plant designs and riser-cracking coal gasification plant designs. An important part of the experimental setup is Hewlett-Packard HP 5840A reporting gas chromatograph, which is used for both vapor-phase and liquid-phase analysis.

The equipment is capable of measuring phase-equilibrium behavior in the pressure range of 0 to 20.7 MPa (0 to 3000 psia) and the temperature range 311 to 533 K (100°F to 500°F). The equipment is closed loop (i.e., once the gas and liquid are charged into the system no further addition is required). Equilibration of the two phases is achieved by circulating the gas through the liquid phase with a high pressure-high temperature gas circulation pump until no change in composition of the gas phase is observed by analysis with the gas chromatograph.

Samples are obtained from the system with two high-temperature, high-pressure rotary sampling valves. This technique is used to minimize disturbance to the system equilibrium. Total volume of the phase equilibrium portion of the system is 0.1 liter. Sample volumes required for analysis are 30 μ l for the gas phase and 2 ml for the liquid phase. A representative sample of the gas phase is obtained by pumping the gas through the sample loop of the rotary sampling valve. Obtaining a representative sample of the liquid phase is more difficult, but good success has been obtained by withdrawing the liquid phase through the rotary liquid sample valve at flow rates of less than 1 ml/min. Pressure letdown of the liquid phase takes place downstream of the liquid sample valve to avoid flashing.

The volume of gas fed to the system is accurately determined with a calibrated high-pressure pump. The total moles of feed gas are determined by recording the temperature and pressure during feeding and correcting for compressibility. The total moles of liquid feed to the system is determined by weighing the difference in the liquid delivery vessel before and after the feeding and converting the difference to moles.

The accuracy of the experimental results is checked by performing a pseudo-material balance for each component. If we know the feed material composition, the equilibrium gas phase composition, and the equilibrium liquid phase composition, then a material balance for each component can be performed using the following expression

$$Z_i F = X_i L + Y_i G, \quad (1)$$

where F is the total moles of feed material, Z_i is the mole fraction of component i in the feed, L is the total moles of liquid phase, G is the total moles of gas phase, and X_i and Y_i are the mole fractions of component i in the liquid and gas phase respectively. However, L and G are unknowns; F is known, as is the fact that L plus G must equal F . Therefore, using this constraint we rearrange equation 1 to read

$$\frac{L}{F} = \frac{(Z_i - Y_i)}{(X_i - Y_i)} \quad (2)$$

Since L and F are constants for a specific set of conditions, Eq. (2) should be satisfied by each component if the liquid and gas phases are homogeneous and the analysis of the two phases is reasonably accurate. Therefore, using the feed, liquid, and gas compositions, L/F is computed for each component. If the calculated values of L/F are consistent within a reasonable error limitation, the values measured are assumed to be correct. Experiments to date with H_2 , CO , CO_2 , CH_4 , C_2H_6 , and C_6H_6 mixtures have demonstrated that the equipment and analytical techniques are performing well. Computed values of L/F for each component are generally within 5% of the mean value for L/F .

In the present equipment, up to three equilibrium points can be measured per day. This is because of the small sampling requirements of the system compared with other approaches. However, as with any other system, a certain amount of time is required for preparation, such as gas chromatograph calibration, cleaning of the equilibrium cell, and introduction of new material. The estimated cost per point with this equipment is \$250 for a large project.

Adaptation of equipment to Coal Liquefaction Studies

As stated previously, the limits of the present phase-equilibria apparatus are 533 K (500°F) and 20.7 MPa (3000 psia).

The only modifications required to adapt the present equipment to coal liquefaction studies are on the columns in the gas chromatograph. To analyze the heavier components, a 2% LAC 446 on Spherosil or equivalent column would replace the Parapak Q, and an activated carbon column would replace the mole sieve 5A. About two to three weeks would be needed to set up the chromatograph with these columns before experimentation could proceed. Estimated cost is only \$150.

If the liquid mixture is too complex to analyze by conventional gas chromatographic technique, gas chromatographic-mass spectrometric analysis of the liquid phase can be performed with existing equipment in the IGT analytical department.

Summary of Costs

The costs associated with modification and operation of IGT's phase equilibrium equipment to study coal liquefaction mixtures is as follows

Modifications and Initial Testing

Salary-related costs	\$4,000
Columns	<u>150</u>
	\$4,150

Operation

Number of data points:

1-10	\$10,000 + \$350 per point
11-50	\$10,500 + \$300 per point
51-100	\$11,750 + \$275 per point
>100	\$14,250 + \$250 per point

Coal/Oil Slurry Preheater Specifications

Operating pressure	13.8 MPa (2,000 psi)
Slurry discharge temperature	700 K (800°F)
Slurry composition	Up to 40 wt % solids
Average particle size	250 μm (60 USS)
Largest allowable particle size	841 μm (20 USS)
Average flux density	18,900-31,500 W/m ² (6000-10,000 Btu/h·ft ²)
Minimum inlet velocity	1.5 m/s (5 ft/s)
Slurry flow rate	0.95-1.58 liters/s (1.5-25 gpm)
Coil surface area	37 m ² (400 ft ²)
Coil material	28 mm ID (1-1/2 in. Sch. XX) Incoloy-800 pipe
Coil height	Occupies 25% of existing firebox height
Cost of helical coil	\$40,000
Cost of installation	\$120,000

Category: Physical Properties

Funding agency: DOE and NSF

Institution: Illinois Institute of Technology (IIT)

Principal investigators: D. T. Wasan and D. Gidaspow, 312/567-3009

Location: Department of Chemical Engineering, Chicago, Illinois

Funding schedule: DOE — August 1977 to July 1978, \$56,316; NSF — August 1978 to July 1979, \$62,000.

Objectives:

The objective of the program is to determine the feasibility of separating micron-size particles from synthetic oil mixtures and coal-derived liquids by electrokinetic separation techniques. Of more relevance to SRC is the effort in emulsion characterization.

Accomplishments:

1. Bench-scale experiments have been conducted on emulsion characterization and foam stability. Professor Wasan is one of the few individuals in the country who is looking at emulsion characteristics. Most of his work is on petroleum crude, which is extremely viscous and resembles asphalt. The objectives of his experiments are to predict the conditions of stable emulsion formation and to find a method to break these emulsions. Facilities to measure surface shear viscosity (as opposed to bulk viscosity), surface tension, and foam size are also available in his laboratory. With some modifications, most of these units could be used with coal slurries because of their similarity to petroleum crude. An instrument that can measure bubble growth, and also the actual bursting of the bubble, in an emulsion has been recently acquired in his laboratory. The data generated is fundamental but could be useful for SRC technology.
2. Fundamental settling (sedimentation) data for synthetic oil mixtures (alumina-xylene, alumina-tetralin, and alumina-tetralin-xylene), both in the presence of a high-voltage electric field and without the electric field, have been obtained as a function of particle size, dielectric constant, and zeta potential. Preliminary settling experiments were conducted on coal liquid samples from pilot plants (SRC, H-Coal, Synthoil, and Amoco).

The high viscosity of the coal liquids caused serious problems in the experiments. No settling was achieved at room temperatures, even with the application of high electric fields.

3. Based on surface charge characteristics of coal-derived particles, a technique called cross-flow electrofiltration has been developed at IIT. The experimental setup there has been used on H-Coal liquids and synthetic liquids, and the rates of filtration have been compared with those obtained from conventional rotary drum pressure filters. The maximum rate of filtration from the rotary filter for the H-Coal liquid at 480 K (400°F) is about 20% higher than that for the electrofilter at room temperature. At higher temperatures, a higher filtration rate can be expected in the cross-flow electrofilter because of reduced viscosity. The device was intended to demonstrate the principle, and with sufficient modification the filtration rates can be significantly improved.

A configurational modification of the electrofilter is being developed, based on a mathematical model that has been developed for settling behavior of non-aqueous slurries.

4. Sedimentation experiments on a synthetic liquid (alumina-tetralin) are being carried out in an experimental unit. The unit consists of a cylindrical stainless steel tube (schedule 80) surrounded by a cylindrical tube made of galvanized iron. Heat transfer oil flows through the concentric tube and heats the slurry in the stainless steel tube. The entire unit is mounted on a moving platform that can be raised or lowered to permit measurement of the settling rate as a function of distance along the column length. The instrumentation is not adequate for coal slurries, and substantial modifications would be needed to test the system on coal slurries.

Need for testing results at larger scale: A definite need exists.

Applicability to the demonstration project: Minimal.

Relevance to SRC or coal liquefaction technology:

The emulsion research is slightly relevant to SRC liquefaction technology.

Capability to perform demonstration support work:

The Chemical Engineering Department at IIT is not adequately equipped to conduct large-scale experiments. The capabilities and facilities are suited for basic research in solids/liquid separations and in emulsion characterization.

Impact of redirection/extension:

If redirected to include coal liquids, the IIT research could contribute to liquefaction technology, particularly in surface characteristics of particles in coal slurries and emulsion properties of coal slurries. The data would be basic but could be useful to liquefaction process developers.

Category: Physical Properties

Funding agency: DOE

Institution: Oak Ridge National Laboratory

Principal investigator: G. E. Oswald, 615/574-6825

Location: Chemical Technology Division, Oak Ridge, Tennessee

Funding schedule: \$200,000 in FY 1979, continuing through FY 1980, in conjunction with the fired heater study (see Sect. A.2.4), at \$465,000.

Objectives:

Measure physical properties (viscosity, density, thermal conductivity, and heat capacity) of coal-solvent slurry liquefaction streams at process conditions in a coal-liquids flow system (CLFS).

Accomplishments:

A bench-scale CLFS has been constructed and is being operated to measure viscosities of coal-solvent slurry streams at process conditions and to study the rheological and heat transfer characteristics of coal slurry-hydrogen flow in liquefaction preheaters. The CLFS is a small-scale coal-solvent slurry liquefaction facility with instruments capable of measuring process liquid stream physical properties at process conditions. Maximum process and physical property measurement conditions are 811 K (1000°F) and 31 MPa (4500 psia). Maximum slurry feed rate (positive displacement pump) is 4 liters/h; hydrogen is supplied by an air-driven booster compressor at rates up to 323 mole/h (270 scfh). The system includes the following: a preheater (2.77-mm-ID and 12.2-m-long), heated in a constant heat flux mode by electrical resistance heating tape; a reactor/dissolver (3.18-cm-ID and 76.2-cm-long), with a radiant heating furnace suitable for both catalytic and noncatalytic processing; a high-pressure, high-temperature vapor-liquid separator; a low-pressure, low-temperature vapor-liquid separator; and appropriate instrumentation for pressure and flow control and for material balance monitoring.

Physical property measurements are possible at several points in the system preheater inlet; preheater outlet; high-pressure, high-temperature vapor-liquid separator liquid discharge and product liquid. Viscosity measurements are made with two instruments, a falling-ring viscometer and a pipeline viscometer. Measurements with the falling-ring viscometer are based on time-of-fall through an annulus filled with the test liquid. Time-of-fall is correlated with equivalent Newtonian viscosity over a range of 0.0001 to 10 Pa·s (0.01 to 10,000 cP). Analysis of pressure drop-flow rate data taken at constant temperature with the pipeline viscometer generates

a rheogram, a plot of shear stress vs shear rate (τ_w vs S). Density measurements are to be made with a gamma-absorption liquid-density gage. Accuracy is 0.002 specific gravity units over a 0.5 to 1.2 span. Liquid heat capacity will be measured in a flow calorimeter based on differential temperature rise for known heat input and mass throughput. Thermal conductivity measurements are to be based on the transient heated-wire technique. Test liquid thermal conductivity is obtained from the temperature-time response of a thin wire undergoing constant electrical heating while immersed in a stagnant body of test liquid. The slope of a plot of wire temperature vs log time is inversely proportional to liquid thermal conductivity up to the onset of natural convection.

Operations were initiated in December 1978. The unit has successfully processed 35 wt % Illinois No. 6 coal/Wilsonville recycle solvent slurry in combined phase (hydrogen and slurry combined) preheating mode at up to 3.6 kg/h slurry feed rate, 711 K (820°F) preheater discharge temperature, and 13.9 MPa (2000 psig) hydrogen pressures. In combined flow, differential pressure measurements across the pipeline viscometer (located at the preheater exit) indicate a sharp rise at 566 K (560°F) in slurry viscosity, which appears to peak at 633 K (680°F) and then decreases as preheater discharge temperature increases. These results are presented as a differential pressure vs preheater discharge temperature plot.

A series of tests have been run using the pipeline viscometer to characterize the rheology of a 35 wt % coal/Wilsonville recycle solvent slurry with helium over-pressure. The tests were run at temperatures of 400-500 K (260-440°F) and at a pressure of 13.9 MPa (2000 psig). Results show the slurry to be Newtonian at a temperature of 400 K. At 478 K (400°F), the slurry was slightly non-Newtonian in nature, while at 500 K it became strongly non-Newtonian and exhibited pseudo-plastic behavior. At 500 K, the slurry had an apparent viscosity of 0.0066 Pa·s (6.6 cP) at a shear rate of 300 s⁻¹. These results are presented in both flow curve plots (τ_w vs $8V/D$) and rheograms. Additional tests at temperatures up to 727 K (850°F) will be done to more completely characterize the slurry.

Process liquids have been obtained from the H-Coal process and will be requested from the SRC processes for physical property measurements.

The gamma-absorption liquid density instrument will undergo operational checkout procedures in October 1979. The thermal conductivity instrument is under construction and the design of the heat capacity instrument has been completed.

Need for testing results at larger scale:

Present physical property measurements are adequate for scale-up.

Applicability to the demonstration project:

The physical properties will be applicable for specific process streams. The rheological characterization of preheater slurries, using the pipeline viscometer, are directly applicable.

Relevance to SRC or coal liquefaction technology:

Direct and very useful.

Relation of project to other programs:

Few programs attempt to measure physical properties under these conditions. Data obtained from this program will be complementary to future programs on physical property measurements of coal-liquid slurry streams in flow systems at process temperature and pressure. These data complement present efforts at SRC pilot plants.

Capability to perform demonstration support work:

The present capabilities and efforts of ORNL in physical properties research should be coordinated with the efforts at the SRC pilot plants and could be directly supportive of the SRC DPP.

Program plans:

The program has been combined with the fired heater study at ORNL and extended through FY 1980 at a funding level of \$465,000. It will include measurements on specific SRC process streams.

Impact of redirection/extension:

The physical property measurements at ORNL will be continued effort in support of SRC technology and should provide a comprehensive physical properties data base for design and scale-up of the various process units.

Category: Physical Properties

Funding agency: Industrial contracts

Institution: Wilco Research Company

Principal investigator: Grant M. Wilson, 801/374-6648

Location: 488 South 500 West, Provo, Utah 84601

Accomplishments:

Recently completed work related to coal conversion includes the following:

1. Vapor pressures of pure compounds and hydrocarbon fractions at temperatures from about 477 K (400°F) to about 727 K (850°F);
2. Vapor/liquid equilibria measurements on pure compounds and hydrocarbon fractions with hydrogen, methane, and hydrogen sulfide at temperatures to 727 K (850°F);
3. Viscosity of hydrocarbon fractions and hydrocarbon fractions saturated with hydrogen at temperatures to 589 K (600°F);
4. Surface tension of hydrocarbon fractions and hydrocarbon fractions saturated with hydrogen at temperatures to 589 K (600°F);
5. Heat capacity measurements on hydrocarbon fractions at temperatures to 644 K (700°F);
6. Density measurements on hydrocarbon fractions at temperatures to 644 K (700°F).

Most of this work was related to the Exxon Donor Solvent process, and some of this work is continuing. In addition, Wilco is presently engaged in two new projects relating to the SRC-II process:

1. Enthalpy measurements relating to the heating of coal slurry and hydrogen from 533 to 672 K (500°F to 750°F). Direct measurements are being made in an attempt to simulate the contact time, heat flux, and other conditions of the actual process. Parameters being studied are the effect of heating rate, hydrogen to slurry ratio, and slurry to oil ratio.

2. Vapor/liquid equilibria data relating to the high pressure aqueous absorption of ammonia from streams containing hydrogen, carbon, oxygen, light hydrocarbons, heavy hydrocarbons, cresols, salts, and acid gases. Direct measurements are being made in an attempt to simulate actual process conditions of pressure, temperature, and compositions. The effects of salt and cresols are also being studied.

All of this work is being done under contract with industrial firms. These contracts are often subcontracts of projects with the federal government.

Wilco is also engaged in various projects for the American Petroleum Institute and the Gas Processors Association relating to the production and processing of synthetic fuels and in other projects for the chemical processing industry. Projects vary in length from about one month to multiple-year projects.

To date, Wilco has not been engaged in any direct contracts with the federal government but would welcome the opportunity. Their capabilities are shown in Table A12.

Need for testing results at larger scale:

Physical, thermal, and thermodynamic properties are generally applicable irrespective of scale, although some properties do require care in adequately simulating process conditions.

Applicability to the demonstration project:

SRC efforts are directly applicable. Other activities may also be useful but would require careful interpretation to determine extent of applicability.

Relevance to SRC or coal liquefaction technology:

Highly relevant data gathering.

Relation of project to other programs:

Complements and supplements other programs in data acquisition. Wilco offers generally quicker response to specific data needs (within equipment capabilities) than other institutions, such as universities and government laboratories.

Table A12. Capability of Wilco, Inc., to perform demonstration support work

Type of measurement	Measurement method	Temperature range		Pressure range	
		K	(°F)	MPa	(psia)
Vapor/liquid equilibrium	Still ^a	255-533	(0 to 500)	0.001-3.4	(0.2-500)
	Rocked cell	77-533	(-320 to 500)	0.1-34.5	(15-5000)
	Flow cell ^b	77-755	(-320 to 900)	0.1-20.7	(15-3000)
	High-pressure rocked cell ^c	297-533	(75 to 500)	6.9-172.4	(1000-25000)
	Chromatographic	77-533	(-320 to 500)	0.1-3.4	(15-500)
Liquid/liquid equilibrium	Rocked cell	77-533	(-320 to 500)	0.1-34.5	(15-5000)
	High-pressure rocked cell	297-533	(75 to 500)	6.9-172.4	(1000-25000)
Simultaneous vapor/liquid and liquid/liquid equilibrium	Rocked cell	77-533	(-320 to 500)	0.1-34.5	(15-5000)
	High-pressure rocked cell	297-533	(75 to 500)	6.9-172.4	(1000-25000)
Solid/liquid or solid/vapor equilibrium	Rocked cell	77-533	(-320 to 500)	0.1-34.5	(15-5000)
	High-pressure rocked cell	297-533	(75 to 500)	6.9-172.4	(1000-25000)
Binary P-T-x data and derived y data	Still	255-533	(0 to 500)	0.001-3.4	(0.2-500)
	Rocked cell	77-533	(-320 to 500)	0.1-34.5	(15-5000)
Vapor pressure	Same methods and ranges as vapor/liquid equilibrium.				
Surface tension	Flow cell	77-589	(-320 to 600)	0.1-20.7	(15-3000)
Viscosity	Rolling ball	77-589	(-320 to 600)	0.1-20.7	(15-3000)
	packed column	311-727	(100 to 850)	0.1-20.7	(15-3000)
Enthalpy and heat capacity	Flow cell	77-700	(-320 to 800)	0.1-20.7	(15-3000)
	Static cell	311-672	(100 to 750)	0.1-20.7	(15-3000)
Density and pressure-volume-temperature properties	Rocked cell	77-700	(-320 to 800)	0.1-20.7	(15-3000)

^aCapable of operation when the vapor condenses to two liquid phases. Solid components can be charged by directly opening the still.

^bA direct measurement of the fraction vaporized of the inlet stream can be made. Also measurements with contact times at the cell conditions of only ten seconds or less are possible. This latter feature is very useful at high temperatures or where chemical reactions occur. Rates of chemical reactions can also be studied by going to very short contact times.

^cSpecial cells have been constructed of Teflon enclosed with metal to handle corrosive mixtures under pressure. Present capability with these cells is to about 3.5 MPa (500 psia) and temperatures from about 233 K to 422 K (-40°F to 300°F).

A.9 Fluid-Handling Components

A.9.1 Introduction

This broad problem area includes four common industry-supplied component groups that have been taxed in SRC applications beyond the state of the art and have caused concern about potential adverse economic effects. These component groups are (1) pumps, (2) compressors, (3) valves, and (4) metering equipment. In most cases, equipment is available that can perform the required functions but, because of SRC fluid properties, is subjected to unusually severe wear. Usually the result is frequent replacement or rebuilding of such equipment and, if switchable spare components are not installed, either loss of function or plant outage. However, in the case of metering equipment, reliable instruments for determining flow rates of SRC slurries are not available.

Beyond considerations of SRC process workability and product markets, attention must be shifted to the economics of maintaining the plant on line. Commercial-sized equipment replacement represents a large potential expense, even when component redundancy is such that total plant shutdown is not necessary. Therefore, a program to develop and test components for the SRC demonstration plants prior to initial startup is recommended.

A.9.2 Suggested program for testing slurry-handling components

Equipment, such as slurry pumps, valves, and metering instruments, needs to be tested under conditions as close as possible to eventual design. This requires that a continuous and adequate rate of flow of applicable SRC process material be maintained. Unfortunately, present test loops and pilot plants simultaneously do not satisfy the two essential criteria of continuous throughput capability and comparable slurry characteristics. Testing programs, such as those at Cresap and the Morgantown Energy Technology Center, do not meet fluid property conditions, and the pilot plants are too small to give realistic flow rates. Test loops have been proposed that would possess the needed flexibility to simulate SRC conditions at adequate scale, but costs for these are prohibitively high, approaching the cost of an entire pilot plant. For instance, an estimate of \$23 million (in 1976 dollars) was given by Stearns-Roger for a slurry pump test module.

A logical approach is to use either the H-Coal or EDS pilot plant in this regard, with H-Coal being the preferred location. The plant is sufficiently large and flexible to provide reasonable test conditions, as well as to offer advantages from a scheduling and cost standpoint. The testing of slurry handling components could be done at the 540 MG/d (600 t/day) input scale (boiler fuel mode), with only a minimum of extra equipment procurement, while process data were being routinely obtained. Operation of the H-Coal plant in the boiler fuel mode would cost roughly \$30-\$40 million per year of continuous running time, with costs being shared with other R&D programs using the facility.

A.9.3 Summary of recommended R&D for individual components

Suggested costs and schedules for the component testing discussed below are summarized in Table A.13.

Table A.13. Suggested funding and schedule summary^a

Area	Funding (thousands of dollars)			
	FY 1980	FY 1981	FY 1982	FY 1983
Pumps				
Evaluation of state-of-the-art pumps	100			
Advanced pump development	200	300	300	
Valve testing				
Prototype and state-of-the-art evaluation	50	100	200	100
Modeling studies	150	150	100	100
Instruments				
Continuation of instrumentation program	600	600	600	
Prototype development		200	200	200
Prototype test loop		800	120	120
Oxygen compressor				
Prototype development and testing	1000	800		

^aThese figures do not include costs of the SRC pilot plant and slurry testing facilities that are discussed in the following components sections.

1. Pumps. Recommended efforts for slurry pump development are concerned primarily with expanding the number of suppliers. Toward this end, it is proposed that evaluation of currently available commercial models be increased and a program to develop at least one advanced prototype be continued.
2. Oxygen compressor. Development of a prototype unit is under way, and funding for this effort should be provided as proposed if high-pressure centrifugal compression remains a demonstration design option.
3. Valves. Provision needs to be made for continued evaluation of promising new designs, with scale and SRC applicability of major concern. Development of a computer model that can be used as a tool in concept evaluation and possible sizing determinations is recommended. Visualization and cold flow studies used to reinforce computer model results should be included in a modeling effort.
4. Metering instruments. A development program is currently in progress, and its continuation is recommended, though with greater emphasis toward SRC applications. Continued pilot plant scale testing of promising instruments should be encouraged, as well as development of advanced meters.

A.9.3.1 Slurry pumps

Introduction. Slurry pumps are needed for a variety of applications in coal liquefaction plants, and they have been the source of many difficulties in pilot plants. Pump service lifetimes as short as a few hours have been experienced. However, the principles of successful pump application in slurry service are well understood, and careful application engineering should result in acceptable pump reliability.

For the near-term in slurry service, centrifugal pumps will be used for heads up to 1.38 MPa (200 psi) and reciprocating pumps will be used for higher heads. Some application principles that should be followed are:

1. Use very hard materials in slurry contact (i.e., impellers, cut-water, and wear rings in centrifugal pumps).
2. Limit impeller tip speed to 30 m/s (100 f/s) maximum.
3. Minimize secondary flow. It is highly desirable to operate a pump at its point of maximum efficiency. Where variable flow is needed,

consideration should be given to providing variable speed so that this criterion can be met. Reliable solid state variable frequency power supplies are now available and provide this capability.

4. Keep slurry away from seals. A major fraction of pump outages are caused by failure of seals. It is mandatory that slurry be kept away from them. This means that an adequate supply of clean flush liquid must be provided and as much attention given to the auxiliary seal flush supply system as to the pump.
5. Maintain adequate suction pressure. Reciprocating pumps need adequate pressure at the suction to function properly. An auxiliary pumparound loop that circulates slurry from the feed tank past the reciprocating pump section and back to the feed tank provides both adequate pressure and good mixing of the suction.
6. Use strict quality assurance in maintaining pumps. This should cover use of recommended materials throughout and satisfaction of criteria with respect to dynamic balance, runout, seal installation, and other parameters.

Relevant R&D programs. Slurry pumps conforming to the principles described and also meeting the requirements of the American Petroleum Institute standard (API-610) for pumps for high-pressure hot-oil service are offered by at least one manufacturer and have been procured for both the Wilsonville and Ft. Lewis SRC pilot plants. These pilot plant tests should enable evaluation of this design. Pumps of the same type have also been installed in critical slurry service in both the H-Coal and EDS pilot plants. Because of the larger size of these last two, probably they will provide more favorable test conditions for the pumps than the present smaller facilities. Other manufacturers are also considering offering such a product. Should this occur, prototypes should be purchased and installed in the pilot plants for evaluation.

Other R&D important to slurry pumping. The Rocketdyne Division of Rockwell International conducted a study for EPRI of ways to replace, with centrifugal pumps, the reciprocating charge pumps now in use. After considering various options, they proposed a two-stage module rated to

produce $9.5 \text{ m}^3/\text{min}$ (2500 gpm) at a head of 3.45 MPa (500 psi). Six modules would be connected in series to provide 20.7 MPa (3000 psi) discharge pressure. Their pump would be designed for much higher impeller tip speed [57 m/s (186 ft/s)] than the presently accepted practice. The EPRI study consisted of the design study plus some materials testing that was done at Wilsonville.

Design, construction, and testing of one of the modules have been proposed. Negotiations between DOE and Rocketdyne have been in progress for approximately one year; questions concerning patents and proprietary rights remain. The project cost is estimated to be \$800,000.

This pump concept is highly innovative and could represent a significant improvement in the technology, although its design deviates from accepted principles and involves considerable technological risk.

Recommendations and conclusions.

1. Testing of state-of-the-art slurry centrifugal pumps should be expedited, particularly at Ft. Lewis.
2. When other manufacturers offer pumps designed for high-pressure hot-slurry service, prototypes of these should be bought and tested. Funding at a level of \$250,000 should provide for the procurement and installation of two units. These would be installed as replacements in existing pilot plants, and test and evaluation costs would be absorbed in the pilot plant operating and maintenance costs.

Probably all slurry pumping requirements in the SRCDDPP could be met with state-of-the-art pumps. Some state-of-the-art pumps are used in existing small pilot plants, and more are being installed in the large EDS and H-Coal pilot plants, which should be operational in 1980. The experience in the pilot plants should be monitored to determine whether these pumps perform as well as expected.

In addition, since it is desirable to have more than one source of supply, similar pumps from other suppliers should be purchased, installed, run, and evaluated.

A.9.3.2. Oxygen compressor

Introduction and relevant R&D. Hydrogen needed for the liquefaction process is made by gasifying coal or residues from the liquefaction process using steam-oxygen blown gasifiers, followed by the water-gas shift reaction and necessary purification steps. It is advantageous to operate the gasifiers at relatively high pressures, and oxygen may be required at pressures up to 8.3 to 10.3 MPa (1200-1500 psi) for the gasifiers.

Centrifugal oxygen compressors for these discharge pressures require three sections and are beyond the present state of the art; the maximum discharge pressure from a centrifugal compressor presently in use is 6.5 MPa (940 psi). A Demag compressor is reported to have been tested briefly at 8.2 MPa, and a Sulzer circulator with a pressure ratio of 1.05 is operating at 10 MPa. Manufacturers of high-pressure centrifugal oxygen compressors express confidence that they can build compressors for this service.

Because the oxygen compressor is crucial to the high-pressure gasifier concept, it is important to demonstrate its successful and reliable operation. A compressor manufacturer (Mannesmann Demag Corporation) has offered to supply the high-pressure section (third case) of a centrifugal oxygen compressor to DOE, and testing of this compressor at the Engineering Technology Center (operated by the Atomics International Division of Rockwell International) is proposed. Funding for the test has been allocated and a contract to do the work is being negotiated by the San Francisco Operations Office of DOE. It is expected to take about 12 to 15 months to manufacture the compressor and to modify the test loop to accommodate it. Testing could not begin until late summer 1980 at best. The overall program is expected to require 18 months, allowing 3 months for testing. Funding for the proposed program is \$1.8 million.

Recommendations and conclusions. It is recommended that the program described above be carried out if demonstration plant design continues to require high-pressure centrifugal oxygen compression.

Manufacturers of oxygen compressors seem confident that they can supply the equipment needed, although it is beyond the present state of the art. A test to confirm this has been proposed and is in the process

of being arranged. At best, even preliminary results cannot be available before late summer of 1980.

Two design options are available if the above program is not funded:

1. Use state-of-the-art design for high-pressure oxygen compression, represented by one- or two-case centrifugal compression followed by reciprocating compressors, at additional capital and operating expense, or
2. Reduce the design oxygen delivery pressure to the maximum level currently proven in two-case centrifugal oxygen compressors, about 6.5 MPa (940 psi) potentially increasing other associated expenses.

Neither of these options carries the technical risk of designing for three-case centrifugal oxygen compression without prototype testing.

A.9.3.3 Valves

Introduction. Valve failures can be roughly divided into two categories related to function: loss of flow control and inability to interrupt flow when equipment isolation becomes necessary. Factors causing failure are usually related to slurry properties, which vary considerably within the process and are generally characterized as extremely erosive and corrosive. Hot SRC slurries contain varying concentrations of solids ranging from high solids content applications to relatively clean liquid streams. Solids contain some particularly erosive minerals, such as alumina, and particle sizes can range from submicron to one-eighth inch diameter. Corrosion occurs with erosion to accelerate the material deterioration. A particularly troublesome application has been the high-pressure letdown valve and its associated piping. As high pressure and temperature reactor effluent streams [e.g., at 730 K and 13.9 MPa (850°F and 2000 psig)] are letdown in pressure, light species are flashed to the vapor phase and cavitation may occur as pressure is subsequently recovered. This pressure cycle may occur as a result of the control action of the valve or from some other flow disturbance, such as a rapid change in direction. Moreover, hot stagnant slurries often exhibit a tendency to polymerize and form

deposits that freeze mechanical movements. The formation of deposits becomes a problem if a valve, such as one used to isolate a piece of equipment, is infrequently operated and deposits are allowed to accumulate in small cavities and crevices. Oversizing of control valves, a factor that aggravates the potential for erosive damage, has been commonplace in early pilot plant experience, and may, in part, be avoided by combining careful design practice with better understanding of coal liquid flashing behavior. Other factors contributing to failure, not directly related to slurry properties, are inadequate methods of trim attachment resulting in separation or breakage of tungsten carbide parts and inadequate quality assurance in assembly of valves.

Directly applicable R&D programs. The valve development efforts most directly applicable to demonstration plant design are those currently being carried on at the SRC process pilot plants. The fluid properties and flow conditions there are most like those encountered in a demonstration plant, with the exception of flow rate (a direct function of plant size which cannot be duplicated). Thus, available performance data for valves in SRC service is necessarily limited to small sizes, giving rise to speculation that increased flow rate through a properly sized valve might substantially improve reliability. This inherent scale improvement remains to be proven.

With over five years of experience in SRC equipment operation, Wilsonville¹ plant personnel have accumulated significant valve testing and development experience. They have done on-location design modifications to industry-supplied valves that have resulted in notable improvements in valve longevity, in some cases increasing changeout intervals from a few days to months. The Ft. Lewis² pilot plant has operated for a comparable period and has similarly been plagued by failures and persistent problems in both letdown and isolation functions. The Ft. Lewis plant has a higher throughput and is potentially the preferred data base for scaling decisions. However, specific valve-testing experience has been limited to relatively few models, especially in the high pressure letdown application. Both pilot plants, at this stage, are still primarily interested in obtaining process performance data; valve testing has been used in a limited number of locations and in relation to

failed units. The following brief equipment summary, therefore, mentions only a few types and is not meant to endorse any particular manufacturer.

The most successful valve in severe letdown service at both plants has been the modified Fisher DBAQ angle valve. Endurance figures given to date on this unit have been as high as 4000 h (for a Wilsonville valve), with 60 d being commonly mentioned. The design is basically a cage-guided globe-type valve that has undergone extensive in-plant design modification consisting of trim revision and material substitutions. Another design that has seen severe service but has not yet been as fully evaluated in SRC letdown work is the modified Willis choke. Recommendation for this design comes as a result of successful equipment experience at the IGT HYGAS³ coal gasification plant. Results of the work are reported in *Plant Engineering* (June 23, 1977) in an article entitled "Controlling Erosion in a High Pressure Letdown Valve." The concept involves variable area orifices (chokes) in series with a fixed nozzle to shift pressure-drop away from control elements and to dissipate erosive energy farther downstream where it does less harm. The valve has been installed at Ft. Lewis and is being evaluated. The valve has also shown promise in shutoff applications because it is less prone to experience gumming problems. Another design scheduled for testing at Wilsonville is a tortuous path configuration developed by Consolidated Controls. The concept uses gradual form losses and expansion to dissipate energy over a larger area with minimal pressure recovery. A number of different types of valves are being used for flow interruption at the pilot plants. Generally, free-flowing designs, which utilize solvent injection flushing systems to keep moving parts free, have given the most satisfactory service. Valves must close with a wiping action to prevent solid particle entrapment between mating surfaces and initial leakage across a large pressure gradient, which leads to rapid failure. It has also been necessary to install valves in dual arrangements, where one valve acts in a sacrificial manner during initial shutoff so that the other can be operated in the absence of a large pressure differential.

Related development programs. Related research and development work includes broad areas of basic fluid dynamics and metallurgy as well as specific testing of valves. Unfortunately, there is a lack of experience

with large valves in hot coal/solvent slurry streams, although test loops have been proposed. Industrial firms have not provided these facilities because of high cost and unsure markets for resulting product lines. A visit to Fisher Controls, one of the largest valve manufacturing firms, revealed test loops with water- and air-handling capabilities as well as static pressure testing, computer modeling, materials testing, and other facilities, but nothing comparable to mixed-phase SRC slurry testing.

Some testing experience has been gained from coal liquefaction work done at the Cresap,⁴ West Virginia pilot plant. The process differs significantly from that of SRC-I and SRC-II, so that results may not directly correlate with Wilsonville and Ft. Lewis experiences.

Equipment experience from H-Coal⁵ and EDS⁶ liquefaction schemes should be reviewed periodically and utilized in choosing equipment for SRC demonstration plants. Gasification projects dealing with hot, erosive, high-velocity flows likewise have relevant information to offer as input into SRC equipment decisions, although test conditions are different and the data must be carefully applied. Other industrial processes that experience similar equipment problems may provide information if proprietary considerations are not compromised. One difficulty, up to this point, has been in obtaining reliable data on specific valve types and failure rates from the above sources. A DOE approach toward solution of the problem might be to contract with a qualified institution to gather this information and make recommendations based on it.

Morgantown Energy Technology Center has done static and dynamic testing of valves for lock-hopper applications in two programs⁷ entitled Prototype Lock-hopper Valve Test Program and State-of-the-Art Lock-hopper Valve Test Program. While not applicable from a test conditions standpoint, the programs do exemplify a sound approach to testing methodology. The measurement and failure analysis aspects of the programs could serve as models for any future SRC valve testing programs. The idea of carefully documenting disassembly and inviting participation by outside experts in the evaluation and recommendation process appears to be particularly effective.

Much of the reported improvement in letdown valve performance can be attributed to material changes, specifically a shift to dense grades

of cemented carbide for trims. Erosion/corrosion coupons are used to gather specific material wear-rate information at SRC pilot plants. Other materials research is widespread, and literature is generally available from national labs, the Materials Property Council, the U.S. Bureau of Mines' Metallurgy Research Centers, EPRI, and others. A relevant example of open-literature information was compiled by Battelle-Columbus for EPRI and is entitled, "Application of Advanced Materials and Fabrication Technology to Letdown Valves for Coal Liquefaction Systems," Reports No. AF 305, March 1977, and AF 579, January 1978.

Recommendations and conclusions. Totally unacceptable failure rates were the rule in early pilot plant experience but have been reduced by improvements over the past few years. Continued pilot plant operation, up to finalization of specifications for demonstration plant equipment, will make possible continued improvement in these small valves. If suspected improvements with scale are assumed, reasonable standards of acceptability established, and sufficient redundancy designed into the demonstration plant, then the urgency for valve R&D seems somewhat reduced. Even taking this optimistic approach, however, valves will be so numerous and expensive in any proposed commercial-scale operation that investments in improving reliability now will ultimately be reflected in reduced maintenance costs and possibly increased plant availability later. Because of currently proposed operating schedules for the larger H-Coal and EDS liquefaction pilot plants, it might be reasonable to postpone major equipment decisions on development projects until early results of these projects become available.

Development of a computer model capable of predicting flow fields within various geometries would be a useful tool during the early design stages of many fluid components, including valves. Such a model could also serve to screen prototype designs and to aid in the sizing of valves. Cold-flow model testing and visualization of flow could provide valuable supplemental information as reinforcement to, or as a check on, analytical determinations.

For the present, communication should be maintained between DOE-funded projects to ensure that latest equipment developments become available to

decision makers as they occur, and that promising industry valve designs are tested under conditions as similar as possible to those of the SRC process and on a scale as large as available facilities will accommodate.

As SRC approaches commercialization, component performance and reliability will become more critical. The H-Coal and EDS pilot plant programs should provide the next level of assurance beyond the present SRC pilot plants, as component sizes in these projects are of adequate scale to clearly indicate trends for the future.

Installations referenced for valves

1. SRC — 6 t/d input; 18 bbl/d equipment output,
Southern Company Services, Inc.; Birmingham, Alabama.
Contract funding — \$8,700,000.
Schedule — through FY 1978, extended through January 31, 1981.
2. SRC — 50 t/d input; 150 bbl/d equivalent output in SRC-I mode.
Pittsburg and Midway Coal Mining Co.; Merriam, Kansas.
Contract funding — \$88,760,000.
Schedule — through FY 1980, extended through December 31, 1980.
3. HYGAS Process Pilot Plant.
Institute of Gas Technology; Chicago, Illinois.
Contract funding — \$22,800,000.
Schedule — FY 1976-1978, extended through FY 1979.
4. Cresap Test Facility — 20 t/d input; 70 bbl/d equivalent output.
Fluor Engineers and Constructors, Inc.; Los Angeles, California.
Contract funding — \$45,700,000.
Schedule — through 1980.
5. H-Coal project — 600 t/d input; 2000 bbl/d equivalent in boiler
fuel mode.
Hydrocarbon Research, Inc.; Trenton, New Jersey.
Contract funding — \$179,000,000.
Schedule — through FY 1982.
6. EDS project — 250 t/d input.
Exxon Research and Engineering Co.; Florham Park, New Jersey.
Contract funding — \$227,000,000.
Schedule — through FY 1982.
7. S-O-A and Prototype Lock-hopper Valve Test Programs.
Morgantown Energy Technology Center; Morgantown, West Virginia.
Contract funding — approximately \$4,900,000.
Schedule — FY 1977-1979.

A.9.3.4 Slurry flowmeters

Introduction. Reliable slurry flow-metering instruments are required for the coal/solvent feed, the recycle slurry, and the vacuum-bottoms streams in the SRC-II process and for the coal/solvent feed, and the solids/liquid separation feed streams in the SRC-I process. Several conventional meters, such as quadrant-edge orifice plates, venturi meters, and target meters have been tested with varied success in the pilot plants, and they are currently used primarily as flow indicators. Problems with changes in the orifice dimensions caused by erosion and coking, difficult calibration of the system for slurry flow, and plugging of pressure taps with solids have been recurrent.

Opinions vary as to how critical the slurry-metering problem may be during demonstration plant operations. Some individuals indicate that material balance determinations based on available flowmeter measurement, pump feed rates, and vessel inventories may be adequate. Others conjecture that the plants cannot be operated smoothly without reliable slurry-metering instrumentation. That instruments of high reliability are not currently available for coal conversion systems appears to be the almost unanimous opinion of pilot plant operators and developers.

Slurry-metering instruments that have been tested in the coal conversion pilot plants and their major problem areas are:

1. Quadrant-edge orifice plates — erosion and plugging of impulse lines and dimensional changes due to erosion and coking.
2. Venturi meters — same problems as with quadrant-edge orifice plates. Flow coefficients are not reproducible at low Reynolds numbers.
3. Target meters — erosion of the linkage was the most intractable problem.

The H-Coal Process Development Unit in Trenton, New Jersey, has reported some success using venturi meters where sizable slurry flows are to be metered. The impulse lines there are purged with petroleum-base oil as compared with those at the Ft. Lewis pilot plant where high pressure H_2 or N_2 (low-pressure service) is used.

Relevant R&D programs. At the present time, the most relevant R&D programs in progress are those at the Ft. Lewis and Wilsonville SRC pilot

plants and the Instrumentation and Process Control for Coal Conversion Program at Argonne National Laboratory. A significant slurry-metering program at the Cresap Liquefaction Test Facility was planned, but the plant was shutdown in FY 1980 and placed in standby so that it can be started if future testing is required. Bench-scale studies at Lawrence Berkeley Laboratory indicate that advanced instruments, such as ultrasonic flowmeters, may have promise for SRC application. Information on these programs is summarized here and is described in detail in the following institutional write-up ("R&D efforts listed by institution").

The Ft. Lewis Pilot Plant

The Ft. Lewis pilot plant will start to test slurry-metering instruments by the use of a skid-mounted test stand that will provide for testing up to six meters in series on small slip streams (v1%) in the following locations:

1. Coal slurry circulation loop
2. Dissolver effluent
3. Lummus deashing settler underflow
4. Vacuum-flash feed

The following flowmeter types will be tested:

1. Target meter
2. Universal venturi
3. Quadrant-edge orifices
4. Elbow meter
5. Thermal meter

Successful candidate meters will be tested in the pilot plant on both SRC-I and SRC-II slurry streams.

During the plant shutdown in September 1979, Ft. Lewis scheduled installation of an ultrasonic flowmeter (developed by ANL) in the feed line to the new fired heater. Details of the meter are provided in the sections discussing ANL.

Wilsonville Pilot Plant

The Wilsonville pilot plant will continue testing of slurry-metering instruments available from vendors, but they currently do not plan developmental studies such as those underway at ANL and elsewhere.

Argonne National Laboratory

The instrumentation and controls program at Argonne National Laboratory (ANL) will be continued if funding is provided for FY 1980 and beyond. A gamma densitometer using cross-correlation techniques will be tested in the near future in their Solids/Gas Flow Test Facility. A neutron activation meter will be studied in a small-scale cold solids/liquid circulation loop. Development of acoustic- and capacitive-type mass flowmeters will be continued, but currently ANL has no plans for high-temperature studies on SRC slurry flow metering in demonstration plant piping. An ultrasonic flowmeter was provided to Ft. Lewis for installation. Operation is based on the Doppler effect.

Lawrence Berkeley Laboratory

A bench-scale study at the Lawrence Berkeley Laboratory (LBL) has had some success with a Techsonics ultrasonic flowmeter in a circulating loop containing coal/kerosene slurry at low temperatures. However, the pipe diameter appears to have a significant effect on the reliability of the measurement in the 25- to 50-mm (1- to 2-in.) diameter range. This work will continue with plans for heating the system to 480 K (400°F) to simulate more closely the SRC operating conditions.

Other important R&D studies. Programs for developing geothermal instrumentation at Sandia Laboratories and Brookhaven National Laboratory (BNL) have developed meters that may have promise for SRC application. Sandia has developed an impeller-type instrument for metering two-phase flow at high temperature (275°C) and pressure. A magnetic flowmeter on slurry materials used in geothermal applications has been successfully tested at BNL. These meters should be examined for possible application to SRC systems. In addition, other meters available from industrial vendors, such as the ANNUBAR flow sensor, gyroscopic flowmeter, a rotating field capacitive meter, and a TRW, Inc., slurry flowmeter used in magneto-hydrodynamic (MHD) applications, should also be considered.

Recommendations. Recommendations for current and new projects are discussed in the paragraphs that follow.

Recommendations for current projects

Continuation of the efforts to develop slurry-metering instrumentation at the Ft. Lewis and Wilsonville pilot plants is strongly recommended. Instruments that show promise in the small-scale test stands should be tested full scale in the pilot plants, and additional provisions should be made for testing these instruments at reasonable flow rates in pipe with as large a diameter as possible. The current efforts in the pilot plants have been directed toward testing of conventional meters, such as orifices and venturis. Additional studies should be implemented for developing and testing the following advanced types of meters in the pilot plants as they become available:

1. Acoustic/ultrasonic
2. Capacitive cross-correlation
3. Gamma-ray cross-correlation
4. Neutron activation

Extension and/or redirection of current projects

The current efforts concerned with developing advanced instrumentation, particularly at ANL and LBL, should be extended into and beyond FY 1980. However, a significant fraction of the efforts should be redirected toward the development of slurry-metering instruments for SRC application with specific attention paid to the following:

1. High-temperature operation, to 700 K (800°F)
2. High-pressure operation, to 13.8 MPa (2000 psi)
3. High solids concentrations, to 50% solids
4. The effects of scale-up in pipe diameter on the measurement of slurry flow rates.

In addition, promising candidate meters developed at ANL and elsewhere should be tested in the pilot plant loops under actual SRC operating conditions with an accelerated schedule to meet the startup requirements for the demonstration plants. The Ft. Lewis program for testing an ANL-developed ultrasonic flowmeter is consistent with this recommendation.

The cost of continuing a portion of the instrumentation program for slurry meters is estimated at about \$600,000 for three years, and prototype instruments will probably cost about \$200,000 each. Therefore, a projected

cost per year would be about \$800,000, assuming that one prototype is purchased each year.

Recommendation of new projects

A dedicated facility is needed for testing of slurry flowmeters under actual SRC plant conditions and at a scale that will provide realistic information for the demonstration plants. The largest systems that will be available for such tests will be the H-Coal and EDS pilot plants. Exxon plans to investigate the applicability of venturi flowmeters. The two key technical concerns are the low Reynolds number and the highly erosive service. The test program will monitor flowmeter accuracy over a range of flow rates, with regular inspections to determine erosion rates. If the meters prove unsuitable, testing of other meters already screened for applicability, at Ft. Lewis for example, should be instituted.

If testing cannot be accomplished in the H-Coal pilot plant (preferably operating in the boiler fuel mode) or the EDS plant, development of a loop with adequate capacity for testing slurry meters in pipes of sufficient diameter to simulate demonstration plant operations should be considered. Coal liquids from the SRC-I and SRC-II plants could be circulated in this loop to simulate plant operations. The cost of this loop will be high. For example, a slurry loop for testing pumps over a range of 0.03 to 0.15 m³/s (500 to 2500 gpm) was estimated at \$23 million in 1976. Therefore, it is recommended that efforts be made to use pilot plants such as the H-Coal plant for testing of large slurry-metering instruments.

Industrial vendors have developed mass flowmeters that show promise for slurry-metering applications and should be tested for SRC feasibility. They include:

1. Magnetic flowmeters — Vendors: Taylor Cybron Corp., General Electric Corp., and Westinghouse.
2. ANNUBAR flow sensor — A newly developed meter that determines flow as a function of the pressure in the vortex created by a bar-shaped obstruction positioned in the flowing stream. Dimensions of the bar are not critical to the flow calibration. Vendor: Dietrich Standard Corp., Box 9000, Boulder, Colorado.

3. Gyroscopic flowmeters — Utilizes the Coriolis effect to meter mass flow through a U-shaped tube. However, since thin tubes must be used for this system, it may not be practical for high pressure-thick tube application. Vendor: Micromation, Inc., Denver, Colorado.
4. Rotating field capacitive meter — A capacitive meter that features an electronically rotated field for determining the relative volume fraction of each phase in a two-phase system. This could be used to meter the solids-liquid ratio in slurry streams. Vendor: Auburn International, Inc., Danvers, Massachusetts.
5. TRW, Inc. meter — A slurry flowmeter designed for use in MHD applications.

R&D efforts listed by institution

Category: Slurry Flowmeters

Funding agency: DOE

Institution: Argonne National Laboratory

Principal investigator: N. M. O'Fallon, Program Manager; Instrumentation and Control for Coal Conversion

Location: Argonne, Illinois

Schedule:

Assuming continuation of the program at ANL, the following meters will be tested in loops currently being installed:

1. A gamma densitometer using cross correlation is installed in the Solids/Gas Flow Test Facility;
2. A neutron activation meter is installed in a small solids/liquid circulation loop. Acoustic and capacitive-type meter development will also continue, but ANL currently has no plans for high-temperature studies on SRC slurry flow metering in demonstration-scale piping;
3. An ultrasonic flowmeter is scheduled for installation at Ft. Lewis in September 1979.

Objectives:

To develop instrumentation and process control systems for coal gasification and liquefaction plant application.

Accomplishments:

The Instrumentation and Process Control for Coal Conversion Program has been responsible for the development of mass flow instruments of the following types:

1. Acoustic flow measurement
2. Capacitive flow and density measurement
3. Activation flow measurements using pulsed neutrons
4. Gamma densitometer mass flow measurement

Cross-correlation techniques are under development that utilize any one of the above techniques to measure local fluctuations in the flow patterns that are superimposed on average values. The same fluctuation can be detected at a station downstream from the first station such that a comparison of the two signals as a function of time-delay will show the maximum correlation when the time-delay equals the transit time of the medium between the two stations. Encouraging results have been obtained using the cross-correlation technique on capacitive flow and density measurements installed in a toluene/coal slurry line at the HYGAS Pilot Plant.

Argonne National Laboratory personnel believe that neutron activation, acoustic, gamma densitometer, and capacitive techniques have the best chance for success in SRC applications, but the problems of scale-up to large diameter pipes, the effects of high temperature and pressure, and the effects of high solids content have not yet been addressed.

Operation of the ultrasonic flowmeter at Ft. Lewis is based on the Doppler effect. In order to use the transducers that were originally designed for low-temperature operation, standoff rods are required to serve as waveguides and to isolate the transducers from high process temperatures. Argonne National Laboratory has developed a transducer for 900°F operation, but the frequency is too high for use with slurries. If the Ft. Lewis tests are successful, development of a suitable high-temperature transducer is possible. Ft. Lewis estimates that Argonne National Laboratory would require about seven months for such development.

Category: Slurry Flowmeters

Funding agency: DOE

Institution: Cresap Pilot Plant

Principal investigator: A. G. Draeger

Location: Cresap, West Virginia

Schedule:

The Cresap Pilot Plant was placed on standby at the end of FY 1979. Therefore, without plans to continue operation during FY 1980 and beyond and revisions to provide SRC process conditions, it cannot be recommended that the facility be used for the development of SRC slurry-metering equipment.

Objectives:

To provide a facility in which coal liquefaction equipment and unit operations can be tested at pilot scale under a realistic liquefaction environment.

Accomplishments:

Cresap Pilot Plant has installed two flowmeter loops capable of testing the following meters:

1. Quadrant-edge orifice plate
2. Eccentric quadrant-edge orifice plate
3. Venturi meter
4. Flow target meter

The test loops are installed at two locations in the pilot plant. The first is in Section 3000 on the high temperature extract containing low solids content. The solids content is about 6%; the temperature range, 590 to 640 K (600 to 700°F); and the pressure, about 1.9 MPa (260 psig). The second loop is in Section 500 on the high temperature deashed extract. Here, the solids content is about 1 to 2%; the temperature range is 520 to 620 K (480 to 650°F); and the pressure is about 0.86 MPa (110 psig). Operation of these test loops would provide data on instruments suitable for the Consol Synthetic Fuel (CSF) process. However, since the process conditions differ widely from those used in the SRC-I and SRC-II processes, the results will probably not be applicable. For example, the SRC processes utilize a much deeper extraction depth (extent of dissolution) so the dissolution streams from the CSF and SRC processes are not comparable in solids content, size distribution, and composition.

Category: Slurry Flowmeters

Funding agency: DOE

Institution: The Pittsburgh and Midway Coal Mining Co.

Principal investigator: J. H. Ward

Location: Ft. Lewis, Washington

Objectives:

To successfully establish in a pilot plant [with coal capacity of 18-36 Mg/d (20-40 t/d)] the conversion of coal to clean fuel products using the SRC-I and SRC-II processes.

Accomplishments:

Normal coal feed rates for the SRC-I system are 36 Mg/d (40 t/d) and 18-27 Mg/d (20-30 t/d) for SRC-II. The major difference between the processes with respect to slurry metering is concern with metering the recycle of hot slurry back to the dissolver in SRC-II and metering the mineral separation feed in SRC-I.

The quadrant-edge orifice has been the most successful meter, but problems have been encountered with dimensional changes because of erosion and coking of the orifice plate. Testing of an ultrasonic flowmeter in FY 1980 is planned. See Argonne National Laboratory institutional discussion for details.

A slurry-metering test stand has been designed by The R. M. Parsons Co. for installation at the Ft. Lewis SRC Pilot Plant. It will operate on a small slip stream (~1%) of the slurry and test the following flowmeters:

1. Quadrant-edge orifice
2. Venturi meter
3. Target meter
4. Thermal meter
5. Elbow meter
6. Segmented wedge

There are also plans to test advanced concepts, such as the ANL acoustic meter when it becomes available in the required temperature range of 400 to 600 K (300 to 600°F). Details of the Ft. Lewis program for slurry metering development through December 31, 1980 (Task 5) are given below:

The contractor shall conduct a slurry flowmeter test program. The objective is to develop correlation curves for, and evaluate the accuracy, reliability, and wear characteristics of a variety of slurry flowmeters in several types of slurry service. The test program will be conducted using a skid-mounted, fluid mechanics test stand. This stand will provide for testing as many as six meters in series and will be equipped with a weigh tank for on-line calibration. Flow streams in the pilot plant where tests are to be conducted include:

<u>Area</u>	<u>Stream</u>
01	Coal slurry circulation loop
02	Dissolver effluent
03	Lummus deashing settler underflow
04	Dirty vacuum-flash drum feed

Meters to be tested include:

1. Target meter
2. Universal venturi
3. Eccentric, segmented, or quadrant-edged orifice plate
4. Segmented wedge
5. Elbow meter
6. Thermal meter.

Category: Slurry Flowmeters

Funding agency: DOE and EPRI

Institution: Southern Company Services, Inc.

Principal investigator: H. E. Lewis, 205-669-6747

Location: Wilsonville, Alabama

Schedule:

Wilsonville plans only testing of available instruments and will leave development to ANL and others.

Objectives:

This program seeks to broaden the data base for the design and economics evaluation of commercial SRC-I plants. Specific objectives are to evaluate and to develop reliable components for these plants.

Accomplishments:

The SRC-I Pilot Plant is designed for a nominal rate of 5.4 Mg/d (6 t/d) of coal that requires low flow rates that are difficult to measure. The following slurry-flow instruments have been tried with varying degrees of success:

1. Target meters
2. Thermal meters
3. Quadrant-edge orifice

The best results have come from the quadrant-edge orifice meters, but the plates must be replaced every three months due to erosion. Also, problems have been encountered in keeping the instrument lines purged of solids. A quadrant-edge meter was used primarily to check for high or low flow; weigh tanks were used for basic material balance purposes.

Category: Slurry Flowmeters

Funding agency: DOE

Institution: Sandia Laboratories, Brookhaven National Laboratory

Principal investigators: A. F. Veneruso, A. J. Dunbar

Schedule:

Although the flow instruments under development for geothermal measurements may not be applicable to SRC processing, it is recommended that liaison be maintained with these programs because both systems have common problem areas, such as high temperatures and pressures, two-phase flows, and corrosive/erosive environments.

Objectives:

To develop instrumentation for use at 548 K (275°C) in pressures up to 48 MPa (7000 psi) to be used for geothermal applications.

Accomplishments:

A high-temperature impeller-type instrument for metering two-phase flows in geothermal systems has been developed by Sandia. The associated hybrid electronics for 275°C operation are presently under development. Electronic components that are also under development include:

1. Resistors
2. Capacitors
3. Transistors
4. Thick-film hybrid microcircuits
5. Ceramic vacuum tubes
6. Transducers for acoustic sensing

Brookhaven National Laboratory has successfully tested a magnetic flowmeter (manufactured by Taylor Instrument Co.) on slurry materials used in geothermal applications. These slurries were recycled materials fed to the drill bits and containing wastewater plus additives required for the drilling operation.

Category: Slurry Flowmeters

Funding agency: DOE

Institution: Lawrence Berkeley Laboratory

Principal investigator: A. V. Levy, FTS 451-5822

Location: Berkeley, California

Funding: FY 1979 - \$132,000.

Schedule:

Plans are to raise the temperature in the loop to 480-590 K (400-600°F) and use anthracene oil as the liquid to more closely simulate SRC conditions. The testing of other slurry flowmeters or increasing the system capacity to test the meter in larger pipe sizes is not planned at present.

Objectives:

To determine erosion/corrosion behavior of materials flowing at representative coal liquefaction system conditions. Also to measure the slurry flow velocity in the loop.

Accomplishments:

A slurry test loop incorporating 50-mm (2-in.) pipe has been installed and includes an ultrasonic slurry flowmeter purchased from Techsonics Corp., Houston, Texas. The meter was originally calibrated for 0-4 m/s (0-12 ft/s), but was modified to meter velocities of 0-20 m/s (0-60 ft/s). It employs the Doppler effect and has been moderately reliable ($\pm 10\%$) when installed in 50- or 76-mm (2- or 3-in.) diam pipe. When used on 25- or 38-mm (1- or 1.5-in.) diam pipe, results are not reproducible. Operation is currently at 333 K with coal/kerosene slurries of about 25% coal. Separate tests are also made to determine the viscosity of the slurry using a Rheomat viscosimeter manufactured by Contraves Co. It may be possible to determine the slurry density from viscosity measurements, thereby permitting the determination of mass flow rates, because the ultrasonic meter measures only the average slurry velocity.

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