EXXON DONOR SOLVENT COAL LIQUEFACTION PROCESS

W. R. Epperly
L. E. Swabb, Jr.
J. W. Tauton

Exxon Research and Engineering Company
Florham Park, New Jersey

ABSTRACT

Exxon Research and Engineering is developing a donor solvent coal liquefaction process to produce low-sulfur liquid products from a wide range of coals. The primary goal is to achieve a state of commercial readiness by 1982 through an integrated program of laboratory and engineering research and development in conjunction with operation of a 250 T/D pilot plant. This presentation will discuss the basis for this development project and the status of the program.

INTRODUCTION

Exxon has been doing research on coal liquefaction for eleven years. Early in the program many different approaches to liquefying coal were explored and tested. The goal was to define a process that would be reliable and operable on any different kind of coal, and that would have a high probability for successful development. By late 1971 the Exxon Donor Solvent (EDS) process was defined and early in 1974 an aggressive research and development program was launched to bring the EDS process to commercial readiness. Since January 1976 the project has been jointly funded by Exxon, the Electric Power Research Institute, Atlantic Richfield Company, Phillips Petroleum Company, and the U.S. Department of Energy.

EDS PROJECT GOALS

The goal of the EDS coal liquefaction project is to develop the process to a state of commercial readiness, i.e., the technology will be available at the end of the project to design and build a full-scale, pioneer commercial plant with a reasonable and acceptable level of risk. The term "commercial readiness" has broad implications and for a new technology such as coal liquefaction depends on a number of factors. These factors include the nature of the new technology, the assessment of alternative development routes, the technical capability of the organization that will design the pioneer commercial plant, the level of risk in the pioneer plant that is acceptable to the owner, and the incentive for early commercialization. The evaluation of these factors, in combination with one another, will determine the stage of readiness for reaching commercial operability for similar technology.

EDS PROCESS DESCRIPTION

The EDS process is illustrated schematically in Fig. 1. The feed coal is crushed, dried, and slurried with hydrogenated recycle solvent (the donor solvent) and fed to the liquefaction reactor in admixture with gaseous hydrogen. The reactor design is relatively simple: an upward flow design operating at moderate conditions of 800-900°F and about 2000 psi total pressure. The reactor effluent is separated by a series of conventional distillation steps into a recycle solvent depleted of its donor hydrogen, light hydrocarbon -1000°F distillate, and a heavy vacuum bottoms stream containing 1000°F liquids, unconverted coal, and coal mineral matter. The spent recycle solvent is catalytically hydrogenated in a conventional fixed bed catalytic reactor. The light hydrocarbon gases are steam reformed to produce the necessary process hydrogen.

The heavy vacuum bottoms stream is fed to a FLEXICOKING unit along with air and steam to produce additional distilled liquid products and a low Btu fuel gas. This fuel gas is used in the furnaces required to operate the process. FLEXICOKING is a commercial petroleum process that employs an integrated coking/gasification sequence in circulating fluidized beds. In this process the unit is operated at low pressure (<10 psig) and intermediate temperatures (600-1300°F in the coker and 1500-1800°F in the gasifier). Essentially all organic material in the vacuum bottoms fed to FLEXICOKING is converted to liquid product or combustible gases. Residual carbon is rejected with the ash from the gasifier fluidized bed.

The EDS process has a number of distinct features. The process steps are based on demonstrated petroleum technology where applicable, and are reasonable and uncomplicated. The noncatalytic liquefaction and catalytic hydrogenation steps are separated. As a result the hydrogenation catalyst is exposed only distillate coal liquids. This results in very low catalyst deactivation rates and also allows direct control of the amount of hydrogen actually added to the coal through the donor solvent. The use of a properly tailored donor solvent has substantial benefits in process operability and product quality.

* "Service Mark"
Distillation to separate liquefaction products is the most cost effective method and provides direct control over the properties of the separated streams. This control is of critical importance when pumping high viscosity streams to the FLEXICOKING unit.

**EDS PRODUCT DISTRIBUTION**

The products from liquefying Illinois bituminous coals are shown in Fig. 2. One ton of dry bituminous coal will yield 2.6 barrels of liquid products—0.1 barrel of LPG, a barrel of naphtha and 1.5 barrels of fuel oil. These yields are in keeping with the constraint of carbon balance to produce process fuel and hydrogen. The naphtha is a good feedstock for gasoline production and the fuel oil can be used in stationary turbines for peak shaving generation of electric power and as heating oil and boiler fuel. The process also produces 78 pounds of sulfur, 12 pounds of ammonia and 221 pounds of ash per ton of coal feed.

**PROJECT FEATURES INTEGRATED R&D PROGRAM**

The EDS project features an integrated R&D program involving bench scale research and a number of pilot plants of different sizes. Fig. 3 illustrates the reproducibility of the curve. Liquefaction distillate liquid yields (expressed as weight percent of dry feed coal) is plotted against the residence time of the slurry in the liquefaction reactor. The data are for Illinois 107 coal at 840°F and 1500 psi hydrogen partial pressure. Each of the data points represents an average of four 24-hour balance periods of steady-state operation at the indicated conditions and with material balances of 98 to 102%. The solid line represents a least squares fit of the data from the 100 pound per day pilot plant. Data from the 1 ton per day pilot plant agree with this correlation. The dashed line is from a liquefaction kinetic model based on different levels of reactivity for different coal macerals tied together by a network of co-plotting chemical reactions. The agreement with the experimental data is excellent. In addition to reproducing these data for Illinois coal very well, the kinetic model is also valid for describing the liquefaction of a Wyoming subbituminous coal.

Use of a kinetic model and the high quality of confirmatory data obtained from considerably different size pilot plants leads to a high level of confidence in predicting the yields in the 250 ton per day pilot plant, which is now being built.

The 250 ton per day pilot plant is an important part of the integrated program. The size of the pilot plant was minimized while maintaining consistency with commercial plant scale-up practices in the petroleum industry. Coking in the slurry preheater, flow distribution and stability in the liquefaction reactor, and screening and entrainment in the vacuum distillation step were identified as critical areas.

Satisfying engineering scale-up and operability criteria for these areas determined the size of the unit. The design basis for the 250 ton per day unit has been confirmed from operations of the one ton per day pilot plant and this gives us added confidence in the successful operations of the larger unit.

Actual operations of the 250 ton per day unit, which will begin in late 1979, will be aimed at confirming engineering scale-up criteria, determining the reliability of the preferred operation conditions, defining practical operating limits for various process steps, and determining operating procedures in critical process areas to allow smooth startups, shutdowns, and transitions from one mode of operation to another. To achieve these objectives, the unit was designed with flexibility to accommodate 20 alternate modes of operation such as different coal feeds, different liquefaction reactor configurations and different coal concentrations. Analyses of the 250 ton per day pilot plant operations will provide an important part of the basis for a commercial plant design.

**PROJECT STATUS**

The present status of the EDS project can be summarized as follows:

1. The feasibility of basic process steps has been confirmed in laboratory studies in which over 30,000 hours of pilot unit operation have been logged. Liquefaction conditions for Illinois and Wyoming coals have been successfully defined in pilot plants processing both 100 pounds per day and one ton per day. In addition, the liquefaction conditions for two different lignites have been defined in the 100 pound per day pilot plant. These studies have investigated variations in reactor temperature, pressure, residence time, treat gas rates and composition, and solvent composition.

2. The FLEXICOKING process step has been successfully operated on vacuum bottoms from Illinois coal in a two barrel per day pilot plant. Operations are now in progress with vacuum bottoms from Wyoming coal. This scale of operations parallels studies used in the commercial development of FLEXICOKING for petroleum residua.

3. Flexibility to vary the product distribution by changing severity in the liquefaction reactor has been established. For example, the ratio of C2 - 350°F naphtha to 350°F fuel oil has been varied from 0.3 to 1.3.

4. Engineering studies utilizing results from operations of the one ton per day pilot plant have confirmed the critical

**REPRODUCIBILITY OF THE ORIGINAL PAGE IS POOR**
cal design basis for the 250 ton per day pilot plant.

(5) Comprehensive commercial plant study designs, involving 10 engineer years of effort, for Illinois and Wyoming coals, have been completed. These studies incorporated the latest laboratory data defining the process steps and included provisions for coal preparation, steam, fuel gas and power generation and product recovery. In addition, correlations have been developed which relate process results to operating conditions and economic models have been developed to select optimum plant configuration and to predict commercial plant economics. These correlations and models allow us to study the effect of process conditions on commercial plant investment and operating cost.

(6) Product utilization studies are in progress to define the preferred commercial outlets and the trade-offs, if any, which will be necessary to insure effective use. This work has primarily focused on end use testing, incorporation of product into existing fuel outlets, and product hydrotreating at different conditions. The coal naphtha is projected to make excellent gasoline components after catalytic reforming, and the 350°F to 1000°F and 350°F+ liquids meet current ASTM specifications for No. 4 and No. 0 fuel oils. Combustion testing in commercial equipment has been encouraging from the standpoint of the completeness of fuel evaluation. In addition, existing particulate emission standards were met without mechanical modifications to the equipment.

In conclusion, the outlook for successful development of the EDS process is excellent and small commercial plants could be on-stream in the late 1980's, assuming that adequate commercial incentives exist.
Fig. 1. Exxon Donor Solvent BDS Process

Fig. 2. Products from Bituminous Coal
Fig. 3. Liquefaction Liquid Yields for Illinois Coal