



# Liquid Fuels from Coal

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Edited by

**Rex T. Ellington**

Fluor Engineers and Constructors, Inc.  
Houston, Texas



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## Preface

This book represents the efforts of seventeen authors to bring to the general and technical public new information on a broad range of topics on coal liquefaction. When the papers were first presented at the 172nd National Meeting of the American Chemical Society, there were many requests for joint publication. As a result, the authors put forth the extra effort to assemble this book. These papers and other recent contributions represent a healthy situation for technical progress beyond 1976 and real progress for commercial plant construction in the next decade if those opposing all energy development can be enlisted to help solve, rather than continue to deepen, the national energy predicament.

In placing the results of their work before their peers, these authors help bring into eclipse the situation that has existed for much of the last generation regarding growth of coal liquefaction technical information beyond that of World War II, or earlier, vintage. The intervening period can be characterized as one of a low level of development, which occurred in private industrial projects, Government facilities, and universities, in decreasing order. Only limited publication has been made of the fundamental or the commercial results of work during this period. Even more limited publication has been made of basic interpretations of reactions and kinetic models for commercial plant design. A number of the papers in this book present results and/or extensions of results known for some time to people inside the various earlier projects. Contrary to some criticisms regarding recent expenditures to "reinvent the wheel," they often represent valid independent confirmations, improvements, extensions, and certainly a willingness to stand before their peers for judgment of the quality of their product. These papers represent contributions to the following major areas of coal liquefaction knowledge:

1. basic dissolution and liquefaction processes,
2. kinetics of certain reactions,
3. important secondary processes, and
4. results of improved analytical methods.

Each contribution is the subject of brief comment to assist readers with limited years of study of the field in interrelating various results and conclusions.

The Editor's comments are made primarily from the aspect of

designing and constructing plants to produce coal liquids. Assessment of academic contribution or sophistication of approach will be left to others. Groups involved in the design of the commercial plants are in a difficult position. Pilot plant and demonstration plant runs are unlikely to have covered all the conditions that must be considered for optimized plant design. Unless a process owner/licensor can supply the necessary design data and is willing to guarantee process performance, the engineering/construction firm will be very reluctant to guarantee that the final plant will perform to meet the design conditions. Development and publication of technical data for wide ranges of conditions is a sound way to soften these problems.

People charged with commercial plant design must make many selections in balancing flow sheets, optimizing energy efficiency, and minimizing the cost of entire projects. Basic decisions depend on the desired product slate, required depth of conversion of the coal feed, and the number of process steps to be employed. One of the papers<sup>1</sup> presented at the same ACS symposium as the others in this book was unavailable for printing because of prior publication. This paper addressed some of the basic flow sheet balancing in commercial plant design. In this case, it suggests objectives for solvents and products for the primary liquefaction unit. The thesis of Gleim is that heptane-soluble product is the most valuable because it would eliminate the downstream refining problems caused by the presence of asphaltenes. The detrimental nature of asphaltenes is concluded from the problems they cause with coking and catalyst deactivation in the upgrading of residual petroleum fuels. Rough material balances were developed to show that only 50 to 60% of the coal need be converted to heptane-soluble product to balance a plant. This product is separable by distillation. The remainder of the coal is required for energy and hydrogen production. The lesson suggested for the process developer is that efforts to push processes to the point of liquefying 80% or more of the coal fed may be well beyond the point of diminishing returns. The greatest benefit may come from optimizing primary products at substantially lower overall liquefaction.

Gleim's basic ideas merit pondering by process developers and plant designers in a number of ways. For example, the advisability of putting more coal through the reactor to obtain the desired amount of product must be balanced against deeper conversion of a lesser amount. This decision can be made soundly only by enough work to develop mechanical flow sheets, specify and price equip-

<sup>1</sup>Gleim, W. K. T. "Liquefaction of Coal without Catalyst Using Selective Hydrogenation."



ment, and estimate final installed cost. The work should be done early to preclude some very bad decisions.

Whether one considers coal as a polymer or a condensed ring structure, the temperatures that must be employed to obtain effective solution rates cause the thermal rupturing of linkages that creates the primary products of greatest interest. Much has been accomplished in determining what affects production of the primary products but much remains to be done to describe the interrelationships involved. Secondly, similar data are required for the conversion or upgrading of primary products. Until such progress is made, including rates of reactions, industry may be foreclosed on true design optimization for the product slate desired by a particular client.

Certainly, with the comparisons available between low-temperature pyrolysis and solution, it is apparent that hydrogen or some other small free radical must be available to stabilize primary fragments as they are formed or condensation toward coke will occur. An effort to describe important characteristics of the solvent medium is described by Kang, Nongbri, and Stewart. This work focuses on the tetralin content of the solvent as a measure of its hydrogen donor capacity. Temperature effects observed suggest that when thermal cracking exceeds hydrogenation donor capacity, coke formation will become significant.

An often-asked question is whether the first stage of a two-stage conversion should be catalytic, if two stages are judged to be necessary. For a given slurry oil, one might consider an empty reactor, one with fresh catalyst, one with spent catalyst, or perhaps the limiting case of a reactor filled with catalyst support type material, which might be termed a "thermal" reactor. Such a first step would logically be followed by a catalytic reactor on straight primary product or demineralized primary product. The paper of Karr *et al.* treats two cases of the thermal reactor, i.e., one filled with alumina and the other with silica material. The primary product is treated over CoMo catalyst. Those concerned with reaction models should also find the paper of Karr *et al.* helpful in selecting primary, secondary, and tertiary products. It is proper to foresee a generalized model that would fit all of the reactor fillings mentioned above.

In the SRC process the solvent/slurry oil is fractionated out of the product oil and recycled without pretreatment. Since the hydrogen donor capacity of the solvent is very important in maximizing light product and minimizing coking, the characteristics of the straight-run solvent become extremely important. Tarrer *et al.* have examined the effect of the minerals in the coal on the hydrogenation and hydrodesulfurization of creosote oil containing primarily phenanthrene, naphthalene, acenaphthene, and anthracene. If the hydro-

generation of these components by the hydrogen in the dissolver can be increased, donor capacity can be increased. The demonstration that certain coal minerals do promote hydrogenation shows why the recycle of coal minerals to the reactor has been considered in some cases. Commercially, handling difficulties and relative ease of pre-treating the solvent may preclude such recycle.

Whatever the initial process of solubilization of the coal at commercially feasible reaction temperatures, catalytic processes cannot become effective until a reasonable concentration is generated of fragments that are small enough to be adsorbed on catalyst particles without steric hinderance. It is generally accepted that fragment molecular weights of less than 300 to 500 are necessary for such conditions to exist. Analyses of primary liquefaction products have shown the existence of large concentrations of a number of polycyclic aromatic compounds. Obviously, improved knowledge of the behavior of these compounds during catalysis would facilitate work on catalysts, reactors, and the process design of commercial plants for the upgrading of primary coal liquids. It is pertinent to solvent regeneration or donor capacity boosting. A basic approach to expansion of such knowledge is to employ a clean simple system consisting of one of the primary products, such as phenanthrene. The paper by Haynes *et al.* examines the hydrogenation of phenanthrene at severities that will cover most commercial upgrading conditions for one catalyst. Catalyst selection was not a part of the investigation.

As process design considerations involve more complex systems it is natural to examine the desirability of using catalysts to augment production of desired materials from primary products in the first and/or second stage reactor. Either location imposes a severe environment on the catalyst. As a consequence, properties outside activity come into play in catalyst selection. A review of basic criteria for selection of catalysts for coal systems is provided by Levy and Cusamano. The review confirms selections already in use and suggests others for test.

Moving beyond the fundamental considerations of catalyst selection, Kang and Johanson examine the behavior of the old standby CoMo catalyst under the conditions of the H-coal reactor. Deactivation with processing of well over two thousand pounds of coal per pound of catalyst is described. The factors considered are carbon deposition, sintering, metals deposition, and attrition. Information of this type is helpful in evaluation of different processing approaches for selected products.

A new variation in the use of catalytic first stage reactors is that employing synthesis gas rather than hydrogen as discussed in the paper of Fu and Batchelder. The basic objective is laudable. If syn-

thesis gas can be used for liquefaction, shift conversion and certain parts of gas clean-up may be eliminated from the process unit normally required to produce hydrogen. The cost savings may be significant. These authors show that with the same operating conditions, comparable amounts of hydrogen and synthesis gas are consumed and comparable amounts of oil produced. The basic catalyst was CoMo-alkali carbonate. Analysis of the product liquid is not detailed except in following the concentration of oil and asphaltene, one of the accepted primary products. Flow testing in a Synthoil unit is not reported yet.

Good reaction equations and rate data and kinetic models based on these are mandatory if commercial plant design is to be optimized and performance guaranteed. These tools are certainly required unless every plant is to be designed right on a set of pilot or demonstration plant data. Data for hydrodesulfurization rates for a Kentucky coal dissolved in anthracene oil are provided by Koltz *et al.* The rates varied with time and temperature.

Among the many rates that may become pertinent to process design is the rate at which hydrogen can be made available for reaction in coal solution reactions. To this end Guinn *et al.* determined the rate at which hydrogen could be absorbed by a Kentucky coal-creosote oil system. The results indicate that the rate limiting step is the chemical consumption of dissolved hydrogen, not the process of solution.

Part of a significant study of rates is reported by Whitehurst and Mitchell. It may overshadow many earlier investigations. Very short contact times and relatively complete product analyses are used to delineate fast early reactions and products. The results merit careful study for industrial process design. These include the rapidity of solution, early need for H-donor agent, delayed hydrogen consumption, and changes in product distribution. The reaction model with which the editor has had greatest comfort over the years is that related to Given. It suggests that depolymerization of the basic coal structure occurs upon solution yielding a molecular weight distribution peaking in the 5000 range. Thermal reactions then yield fragments with molecular weights in the 300 to 700 range. The Whitehurst and Mitchell work shows an early production of more than 2000 MW followed by rapid production of 300 to 900 MW material. They show rapid early yield of SRC-type product with little hydrogen consumption. With a push for deeper conversion, hydrogen consumption increases markedly.

One of the toughest engineering problems facing the industrial process designer is related to the mineral matter and undissolved coal in the reactor product. Somehow this material must be removed

from the final product. Distillation is not cheap, even when applicable. Among the approaches being explored are particle agglomeration, filtration, high gravity and magnetic fields, and antisolvent methods. Katz and Rodgers have tried to reform the inorganic particulates to facilitate their separation. Solvent dilution with process-generated solvent improved filterability only as much as a 40°C increase in temperature. Neither of these approaches or surface active agents yielded separability of the type needed in plants.

In another paper, Newman *et al.* explore use of a process-generated antisolvent to promote agglomeration of solid particles. With a short period of treatment filterability was improved substantially without need for filter-aid. Processes of this type may offer considerable promise.

Finally, Weintraub *et al.* tested a number of filtration approaches. None would yet seem to overcome the extreme operational and economic problems associated with commercial usage of filtration.

The keystone of reaction modeling and good material balances is in the existence of good methods for analyzing different plant streams. Many product streams in coal liquefaction are extremely complex and in years past analyses would take longer than the experiment. In many cases good reaction equations could not be prepared because analytical methods were not available or were prohibitively expensive. Recently analytical methods have taken such a leap forward that the opportunity for delineation of coal liquefaction reactions is enhanced greatly.

An extremely detailed procedure is reported by Dooley and Thompson using many procedures developed for the analysis of high-boiling petroleum fractions. The analytical system is based on physical and chemical separation followed by instrumental characterization. From this and other recent advances process developers should be able to select procedures that will realize full value from their reaction investigations.

Schwager and Yen employed solvent fractionation to obtain oil, resin, asphaltene, carbene, and carboid cuts. Color index, solvent elution chromatography, X-ray, NMR, and silica gel chromatography were employed to analyze the cuts.

Prather *et al.* discuss high-pressure liquids chromatography. With the suite of methods now available, it should be possible to prove or disprove old reaction concepts and follow the lives of important constituents in future work.

Finally, optical studies of residue particles from coal liquefaction are described by Mitchell, Davis, and Spockman. Petrographic examination of coals yields much information on source material and reactive parts thereof. Association of feed coal macerals with resi-

due components may provide yet another way to trace changes coal during processing.

The second paper<sup>2</sup> ineligible for this book because of prior publication describes fast pyrolysis of small coal particles in an atmosphere of hydrogen. It is a logical extension of free fall hydrogenolysis of coal and oil shale done some years ago. A significant aspect of the work of Pelofsky *et al.* is that preliminary balances of commercial type flow sheets show that product yields of only about one-half the feed coal (lignite) are necessary to balance the plant flow sheet. Cracking severity can be varied to shift the proportions of liquid and gaseous product.

Most significantly, the liquid product is predominantly BTX with minor amounts of naphthalene, toluene, and anthracene, and the gaseous product is primarily methane and ethane. This product slate provides the potential for a relatively simple plant with only a few, high-priced products.

To conclude, coal technology is advancing in many areas. It is hoped that joint presentation of these papers will help show how the efforts fit together.

<sup>2</sup>Pelofsky, A. H., Greene, M. I., and LaDelfa, C. J., "Short Residence Time (SRT) Coal Hydrolysis".



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## THE ROLE OF SOLVENT IN THE SOLVENT REFINED COAL PROCESS

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A preliminary study of the effect of solvent upon SRC process performance was undertaken. The startup solvent and makeup solvent have a significant effect upon coal conversion. However, the use of a good startup solvent does not sustain good process performance under unfavorable process conditions and vice versa. Hydroaromatics, represented by  $H_\beta$  from NMR analysis, other than tetralin also possess hydrogen donor capabilities. These hydroaromatics could be better donors than tetralin. This conclusion was reached during a process study of the Black Mesa coal, a subbituminous coal. At the same hydrogen pressure, an increase in reactor temperature resulted in higher hydrogen consumption, lower coal conversion and higher dry gas ( $C_1$ - $C_3$ ) production. These observations were accompanied by a deterioration in solvent properties, (as shown by the lower tetralin to naphthalene ratio and lower  $H_\beta$  value) and significant amount of coke formation. These findings support a hypothesis that coke formation results when thermal cracking gets ahead of hydrogenation which is catalyzed by the hydrogen donors present in the solvent.

### I. INTRODUCTION

The coal-derived liquid used to produce a pumpable liquid slurry feed to hydrogenation systems is commonly referred to as slurry oil or pasting oil. The role of this material, other than as a transport medium, has been neglected until the last few years. The pasting oil used in Bergius Catalytic Hydrogenation consisted of fractions collected at various points in the downstream process. Some of these products of

carbonization and/or distillation were catalytically hydrogenated before being used. No specific analyses were made which provided information about a chemical basis for understanding hydrogen donor capability. Necessary solvent qualities that contribute to or enhance coal conversion, hydrogenation or desulfurization were not quantified. Pott-Broche used a hydroaromatic middle oil generated from coal or tar hydrogenation as their pasting oil and were able to achieve conversions of about 80% at pressures in the 1500-3000 psig range.

More recently, processes that depend upon the hydrogen donor capacity of the process solvent have been developed. Among these are the Consol Synthetic Fuel Process (CSF), Pamco Solvent Refined Coal Process (SRC), and Exxon Donor Solvent (EDS) Process.

The CSF and EDS processes depend upon special solvent production through fractionation and subsequent hydrogenation. The SRC process does not employ solvent preparation other than fractionation.

Interest has grown in the role of solvent in these three processes as bench scale and small Pilot Plant investigations have intensified. The application of analytical techniques toward identifying donor hydrogen was described by R.P. Anderson (1). More recently, donor reactions for desulfurization were described by G. Doyle (2).

The Exxon (EDS) process in operation since 1975 at the one-ton-a-day scale depends heavily upon knowledge of solvent quality and the ability to control it (3). A proprietary solvent quality index was reported by Exxon. A minimum quality index reported to be a function of liquefaction conditions was related to conversion and claimed to improve handling qualities of the products.

This work reports some rather dramatic bench scale coal processing data. Major effects are attributed to identifiable slurry solvent properties.

## II. THE ROLE OF SOLVENT

In conjunction with a process study undertaken at HRI under Electric Power Research Institute Research Project 389 to investigate SRC process operability and product yield structures for several coals of commercial interest, a preliminary analysis of the role of solvent upon coal conversion was carried out. The purpose of this project was to screen coals prior to their testing at the 6 T/D SRC Pilot Plant at Wilsonville, Alabama. The operation of this plant has been jointly funded by EPRI and Southern Services, Inc. Catalytic Inc. is the plant operator. The study was undertaken with two