

ENGINEERING DEVELOPMENT OF THE CITIES SERVICE,
SHORT RESIDENCE TIME (CS-SRT) PROCESS

MARVIN I. GREENE

CITIES SERVICE RESEARCH AND DEVELOPMENT COMPANY
P. O. DRAWER 7
CRANBURY, NEW JERSEY 08512

History and Development of the CS-SRT Process

Cities Service Research and Development Company (CSR), the research arm of the Cities Service Company, has been developing a process scheme named The CS-SRT Process, for the non-catalytic, vapor-phase, hydrogenation of carbonaceous feedstocks. The initial and primary emphasis in our Energy Research Laboratory was to apply this technology to convert coal into pipeline quality gas and attractive byproduct yields of light aromatic (BTX) liquids.

A review of the literature early in 1974 led to our conclusion that the most productive area of process development would be that of short residence time hydrogenation. A brief literature review has been presented in our previous paper at the 1976 ACS (San Francisco) meeting (1).

In the summer of 1974, we designed a 1-4 lb/hr bench-scale unit capable of operation at temperatures and pressures that are scalable within present-day, commercial technology. A cold-flow model to test coal-hydrogen mixing injectors and to study coal-hydrogen slip velocities was designed and constructed in Autumn, 1974. Construction of the bench-scale unit began in December, 1974 and was completed in June, 1975 with the shakedown operations occurring in the Summer of 1975. The first complete material balance run was made in August, 1975. Since that time, over 125 runs have been made with a variety of feedstocks including lignite, bituminous and subbituminous coals, oil shale, tar sands and coal tars.

In September, 1974 work was initiated to estimate the economics in a conceptualized, commercial application of the CS-SRT Process for producing 250 MM SCFD of pipeline gas (and the associated byproduct BTX-liquids) from a mine-mouth plant in the Montana/North Dakota region. The results of this work pointed out the dramatic effect of benzene byproduct yield on lowering the cost of services for producing pipeline gas. In September, 1976, CSR retained the Foster Wheeler Energy Corporation to perform a preliminary engineering design and cost study of the CS-SRT Process to validate the results of the earlier economic study. The results of this study will be reported in a separate paper.

Further development of the CS-SRT Process is being undertaken in several programs supported jointly by Cities Service, Rocketdyne Division of Rockwell International, and ERDA. Experimental tests are continuing in the bench-scale unit to explore conditions for maximizing liquids yields and also for maximizing gas (methane, ethane) yields. Process flowsheet studies are also being made. The next step of development of the CS-SRT Process would require about a 6-inch diameter, pilot plant reactor capable of processing about 100 TPD coal. This pilot-plant would be one scaleup step away from a 12-inch diameter, single tube, commercial-type operation.

This paper summarizes the analysis of the bench-scale data obtained when processing a North Dakota lignite. Some conceptual commercial processing alternatives which appear quite attractive are also discussed.

Experimental Apparatus

The bench-scale unit, as described in our previous paper, utilized initially a dilute-phase, free-fall reactor with a movable quench probe. Coal was delivered to the hydrogen-coal mixing injectors under gravity flow from a star-wheel, volumetric feeder fed by a lock hopper. The feeder-hopper assembly sat directly on top of the reactor.

Since the initial testing, a coal transport system was developed to improve the running efficiency of the test program. This system also allowed for the remote location and safe access of the coal feeding equipment in an adjacent laboratory pressure cell. The transported coal was separated in a cyclone immediately upstream of the coal-hot hydrogen injectors and carried into the reactor. The "cold" hydrogen was recycled back to a diaphragm compressor. The compressor diaphragms were protected against entrained solids by use of a settling chamber and filters located on the suction-side of the compressor.

Several other reactor designs were also incorporated into the bench-scale unit since the initial testing. A description of these reactors is shown in Figure 1. In these "entrained-flow" reactors, a wider range of values of several important parameters is achieved over that of the free-fall reactor:

| | <u>Free Fall</u> | <u>Entrained-Flow</u> |
|----------------------------------|------------------|-----------------------|
| Coal Flux, lb/hr ft ² | 100-300 | 1500-35,000 |
| Gas Velocity, fps | 0.1-0.5 | 2-50 |
| Particle Velocity, fps | 0.5-8 | 2-50 |
| Gas Reynolds No. | 100-500 | 1000-6000 |
| Particle Residence Time, sec. | 0.10-4 | 0.05-10 |
| Vapor Residence Time, sec. | 2-30 | 0.05-5 |

The vertical entrained-flow reactor is used generally for very short residence time studies (less than 200 milliseconds) whereas the helical entrained-flow reactors are used for longer residence times (c.a., 1-5 sec.). All of the reactors used in the bench-scale unit are fabricated from stainless steel 316 alloy. A "hot wall" pressure reactor was used as the basis of design to expedite the testing program. The life of the hot wall reactor, even at the small diametral ratios used, is limited because of operation at high stress levels. Therefore, careful watch is maintained on the instantaneous creep strength of each reactor and preheater vessel. Reactors are removed from service before the cumulative approach equals 25% of the rupture time. All reactors are operated in the confines of a high-pressure, explosion-proof, laboratory cell with lockout controls during testing.

Temperature and Residence Time Measurements

Temperature of the coal-hydrogen stream is an extremely important variable affecting the rates of hydrolysis under short residence time conditions. Most experimenters involved in continuous, two-phase, coal-gas reactions report reactant or reactor temperature as maximum reactor wall temperature. This latter temperature can vary quite markedly from the reactant temperature depending upon the size and mass of the reactor, coal throughput, exothermicity of reaction, heat losses, etc. Accordingly, we have developed several multi-dimensional, computerized, heat transfer models for estimating reactant temperatures from reactor wall thermocouple readings. We have also used equivalent isothermal temperature (EIT) calculations to characterize the non-isothermal reaction conditions. EIT has been found to be a good characterization factor in correlating trends of hydrolysis yields with temperature.

Particle residence time is another extremely important parameter affecting hydrolysis yields, particularly under short residence time conditions. In the case of the free-fall reactor, particles traverse the reactor under the influence of both gravity and entrainment by hydrogen. Char settling velocities were calculated using the Stokes equation for narrow-size ranges of particles. Corrections were applied for the wider-range size distributions used in the bench-scale operation by calculating a surface-volume average particle diameter. Based on studies by Wen and Huebler (2), corrections for gas entrainment, uneven particle distributions and particle cloud density were also made.

For the case of entrained-flow reactors, particle residence times are directly correlated with the superficial hydrogen velocities. For both cases, a comprehensive experimental (cold-flow) program is underway in order to measure accurately particle residence times in both free-fall and entrained-flow reactors.

Coal Preparation

Wet North Dakota lignite was received in drums from the Grand Forks Energy Research Center. Prior to drying, the lignite was reduced to 1/2-inch x 0 particle size using a rotary jaw mill blanketed with nitrogen. Drying was done either in a laboratory, fixed-bed drier at 15" Hg vacuum or in an industrial, fluid-bed dryer, both under nitrogen blanketing. In both cases, the maximum lignite temperature was limited to 230-250°F. Moisture was reduced from 35% to less than 5%. The lignite was then pulverized using either a Sweco ball mill or a Mikropul hammer mill. In both cases, a nitrogen atmosphere was maintained. An 18-inch, continuous Sweco sieve, fed by a Syntron vibrating feeder, was used to sieve the dried, pulverized lignite to specification.

Immediately prior to a run, the lignite charge to the unit was dried again, under vacuum, in the laboratory tray drier. An aliquot sample from a riffler was taken for every batch of coal fed to the bench-scale unit. The analysis of this sample was used as the basis for the material balance calculations for each run. A typical analysis was shown in our previous paper (1).

Material Balance Calculations

As a result of using hydrogen as both the preheating gas and the quenching gas, the coal-derived, reaction products are very dilute in hydrogen. Liquor and aromatic oils (boiling above BTX) are condensed using conventional indirect heat exchange. The lighter products, methane through BTX, are measured using gas chromatography combined with calibrated flow meters. Rotameters, orifice meters and turbine meters are used to measure gas flow. Redundancy in flow metering was incorporated into the design of the bench-scale unit in order to minimize uncertainty in calculation of the gaseous product yields.

All solid and liquid products are weighed after every run. The reactors were examined after each run and found to be free of deposits. The recovery system is purged with high pressure steam after every run in order to remove any hydrocarbons or char that does not readily disengage from the tubing surfaces, valves, receivers, etc. These hydrocarbons and char are recovered from the steam condensate by extraction with ether and are included in the material balance.

A packed cryogenic condenser was developed to condense the residual BTX material from the ambient (70°F) hydrogen stream after G. C. analysis. The weighed amount of BTX liquids recovered from this condenser checked

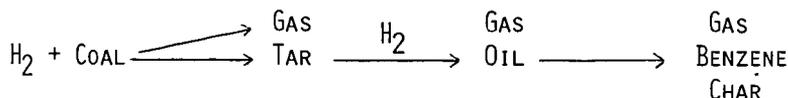
with the chromatographically-determined, BTX content of the gas stream entering the cryogenic condenser. Carbon balances of 90-105% are obtainable. Hydrogen consumption is calculated both 1) by difference of measured feed and product streams and 2) by elemental hydrogen balance between feed and product streams. Ash balances are 90-100%. The calculated oxygen balances are 100±10%. Nitrogen and sulfur balances are calculated by difference because of the very low quantities of these elements in lignite and lignite-derived, hydrolysis products. Gas yields are calculated based on coal fed to the reactor and corrected to 100% carbon balance.

CS-SRT Process Technology

Mechanism

The mechanisms involved in short residence time hydrolysis of coal are exceedingly complex. Coal consists of many types of structures each with different bond strengths. The initial coal conversion reactions are controlled by a thermal mechanism. Bond breaking will closely follow the rapid heating profile imposed on the coal. Weak bonds will rupture at the lower temperatures and decomposition products that are volatile will escape the particle under the driving force of a concentration or partial pressure gradient. As temperature is increased, stronger bonds will break and the volatiles will follow the same escape mechanism. These reactions are extremely rapid, of the order of milliseconds. Many of these devolatilized species are highly reactive and will undergo secondary decomposition reactions to form condensed and polymerized molecules generally classified as asphaltene and char. However, these asphaltene can be stabilized in the bulk phase and removed as single or double-ring aromatics by imposing the proper hydrogen partial pressure-temperature-residence time profile.

In terms of macro-reactions, the following simplified mechanism describes the vapor-phase, short residence time, hydrogenation process:



Although benzene is very stable, it too will decompose at elevated temperatures as shown by R. Graff and coworkers at CUNY (3). Extended residence times will also result in benzene decomposition as reported by Virk, et. al. (4). Normally, benzene decomposition in a hydrogen atmosphere would result in methane as the main decomposition product. However, it appears that in the case of benzene formation via short residence time coal hydrolysis, char particles catalyze the benzene-to-coke route of decomposition. Therefore, rapid quenching of the product vapors is necessary for the stabilization and recovery of aromatic liquids. The quantitative sequence of the in situ hydrogenation of tars to heavy oil to light aromatics is shown in Figure 2.

Coal Oxygen Reactions

Processing of lignite, because of its high oxygen content, provides some interesting observations about the mechanism of short residence time hydrolysis. The gaseous products obtained at very short residence time were rich in carbon dioxide while those obtained at longer residence time were depleted of essentially all carbon dioxide. This led to the conclusion that one of the initial reactions of short residence time

hydrogenation of lignite is the pyrolysis of carboxylic oxygen structures (decarboxylation). This agrees with the work reported by James Johnson at IGT (5). Once into the vapor phase, carbon dioxide hydrogenates to form carbon monoxide and water by the reverse water-gas shift reaction. Carbon monoxide will react further with hydrogen to form methane and additional water by the methanation reaction. These relationships are shown in Figures 3 and 4 which show the yields of water and methane respectively as a function of CO₂/CO ratio. The fact that these relationships were found in the 1/4-inch entrained-flow reactor and in the 1.1-inch, free-fall reactor, where the surface/volume ratio varied from about 50 to 500 in ⁻¹, tends to indicate that these vapor-phase reactions were not catalyzed by the SS 316 reactor wall. Equilibrium calculations of the reactor effluent also show that a) methane is approached from the carbon monoxide side of the steam-reforming reaction and b) carbon monoxide is approached from the carbon dioxide side of the water-gas shift reaction.

Coal Hydrogen Reactions

Another feature of the CS-SRT Process (as well as many other direct coal hydrogenation processes) is the utilization of coal hydrogen. Coal dehydrogenation increases continuously as hydrogenation severity and coal conversion are increased. During the initial pyrolysis stage of short residence time hydrogenation, the rate of coal dehydrogenation is increasing faster than hydrogen is being consumed. The rate of coal dehydrogenation appears to reach a maximum at about 30% carbon conversion as shown in Figure 5. At this point, approximately 50% of the hydrogen consumption is coming directly from the coal. This point also corresponds to the end of the pyrolysis mechanism and the initiation of the char hydrogenation stage. This is not surprising since 30% carbon conversion corresponds to 43% coal (MF basis) conversion which is identical to the ASTM volatile matter for lignite.

Furthermore, at carbon conversions greater than about 30%, hydrogen partial pressure has a pronounced effect on carbon conversion. The flux of volatiles escaping the coal particles decreases greatly above the 30% carbon conversion level. This results in increased hydrogen counterdiffusion into the char structure allowing more reaction between hydrogen and carbon sites. Higher hydrogen pressures greatly influence the diffusion of hydrogen into the particle. This is shown in Figure 6 and agrees with the work of James Gray and coworkers at PERC (6).

Rapid-Rate Carbon

One of the principal correlation tools of short residence time hydrolysis is the rate of conversion of rapid-rate carbon (7). The latter is a measure of the total theoretical amount of coal carbon that can be converted under "rapid" hydrogenation conditions. The rate itself is proportional to hydrogen partial pressure and the amount of rapid-rate carbon material remaining. Rapid-rate carbon is a function of coal type, particle size, temperature and pressure and is a difficult parameter to measure. Although this kinetic tool cannot be used as a predictive technique unless all of these functionalities are known, it can be used to demonstrate the relative effects of different hydrolysis data acquisition methods.

When processing a lignite at about 850°C, researchers (7) utilizing a captive sample technique for data acquisition reported a pseudo-rate constant of 50 atm⁻¹ hr⁻¹. Calculation of the pseudo-rate constant from free-fall reactor test data reported in the literature (8) results in a value of about 8 atm⁻¹ hr⁻¹ for lignite. Both sets of data were based on the assumption that the rapid-rate lignite carbon available for rapid hydrogenation is 100%. If the actual rapid-rate carbon for lignite is less

than 100%, then the respective pseudo-rate constants would both show corresponding increases.

The pseudo-rate constant for lignite at 850°C for the CS-SRT Process is 230 atm⁻¹ hr⁻¹, 5 to 30 times greater than that of the literature data, respectively. This CS-SRT pseudo-rate constant was based on data obtained with the turbulent, entrained-flow reactor equipped with a highly efficient, coaxial, coal-hydrogen mixing injector. Thus, it can be seen that in order to maximize carbon and thermal efficiencies in the conceptual CS-SRT reactor, it is imperative to utilize a reactor design based on data acquired from a bench-scale unit that has been operated under conditions closely scalable to the commercial reactor concept.

CS-SRT Conceptual Process

A conceptual plant design for producing 250 MM SCFD of pipeline gas and 6300 BPD of aromatic liquids has been developed. The design was based on data obtained when processing a North Dakota lignite in the bench-scale unit. Several alternative processing sequences have been examined but an optimization has not been undertaken. When more definitive test data become available, as would be generated in a pilot plant, a conceptual process optimization will be performed. The balance of this paper deals with the processing requirements about the reactor system including coal drying, coal feeding, SRT-reactor designs and quench. Details of the downstream processing, hydrogen production and offsites are presented in another paper at this meeting.

Design Basis

After careful analysis of the CS-SRT Process data and patents, the following reactor design basis was established for the conceptual plant:

1. "Slow" heating of coal to the incipient devolatilization temperature in order to increase thermal efficiency;
2. "Rapid" heating of coal from the incipient devolatilization temperature to hydrogenation temperature in order to maximize rate of conversion;
3. Use of the reactant, hydrogen, as the heat carrier in order to minimize the number of streams to be handled;
4. Total residence time less than two seconds;
5. Rapid quenching of product vapors to prevent secondary decomposition reactions;
6. Recovery of high temperature heat from the reactor effluent.

Coal Drying

Lignite drying on the commercial-scale typical of an SNG plant has an energy requirement of about 16 billion BTU/day which equates to about 5.5% of the heating value (HHV) of the lignite. In conventional drying methods, this heat is supplied from hot combusted fuel gas. Efficient lignite drying is a relatively new technology and conventional processes have been applied to reducing the moisture from 35% to only about 10-15%. In the case of the CS-SRT Process, any moisture entering the reactor will be evaporated at the relatively high SRT reaction temperatures. This heat duty will of course come from the hot recycle hydrogen stream. Therefore, a design specification of 3% lignite moisture was set in order to improve the reactor heat balance. Reduction to 3% using conventional drying technology may prove difficult due to the long residence times required in gas conveyor-type dryers.

To circumvent these problems, coal drying in the CS-SRT Process will make use of the large quantity of low level heat available in the form of 60 psi steam. This steam is associated with the inefficiencies in the

many processing steps in all coal conversion plants. In the CS-SRT Process, it is believed that 60 psi steam will account for about 5% of the heating value of the coal fed to the plant.

Furthermore, advantage is taken of the boiling point depression of water as a result of the azeotrope formed between water and benzene (156°F, 1 atm, 30 mol% water, 70 mol% benzene). Benzene is chosen since it is a byproduct of the CS-SRT Process thereby minimizing the handling of additional chemicals. Accordingly, wet lignite will be pulverized and slurried with benzene as shown in Figure 7. The slurry will then be fed to an ebullated-bed contactor where it is heated by an inert recycle gas stream. The gas stream will obtain its heat by indirect heat exchange with the 60 psi steam source. Proper design of the ebullated-bed, which falls within Cities Service's LC-Fining technology, will result in the vaporization of the benzene-water azeotrope. Condensation of the azeotropic vapors results in the recovery of a relatively pure liquid benzene stream and a liquid water stream. The benzene is recycled and this water, generated internally within the process, will be available for plant use in the gasifiers or shift reactors. This water can account for approximately 40% of the total process water needed in the CS-SRT complex and can result in water savings of about 2000 acre-ft/yr.

The "dried" coal-benzene slurry will be drawn from the ebullated-bed with less than 3% water content. The slurry will now readily be available for downstream processing in the coal feed section.

Coal Feeding

Since the CS-SRT Process utilizes a pressurized gasifier, it has the same problems regarding the feeding of coal into a pressure vessel as do other pressurized gasification processes, such as BI-GAS, HYGAS, and SYNTHANE. One major difference and advantage is that the CS-SRT Process produces benzene, a solvent that can be used to maintain a pressure seal. Therefore, the low pressure, lignite-benzene slurry from the coal drying section will be pumped to system pressure with a positive displacement injection pump. The benzene will be subsequently flashed away from the lignite in a drier near its critical point. Benzene content of the dried coal should be extremely low as a result of the purging effect of the benzene-free, fluidizing gas.

Lignite with less than 3% water and at system pressure will then be delivered to a dense-phase transport system (see Figure 8) similar to that described by Rocketdyne Division of Rockwell International (9). Laboratory tests have shown lignite dried from a benzene slurry to be free-flowing. It is believed that the small quantity of benzene remaining in the coal pores will enter the reactor and be carried through along with the product gases without undergoing decomposition.

Reactor Design

Rapid heating of coal may be achieved by mixing hot recycle hydrogen with the dense-phase, transported coal in an injector assembly at the inlet of the reactor. With properly designed injectors, mixing is rapid on the order of a few milliseconds. Once mixed the coal and hydrogen will traverse the reactor upwards under conditions of fast fluidization to allow intimate contact between hydrogen and coal particles. The temperature and flowrates of the hot recycle gas and coal respectively will be set to allow the CS-SRT reactions to proceed adiabatically and exothermically. The recycle hydrogen/coal ratio is a critical parameter affecting not only the reactor heat balance and reactor kinetics but also the cost of downstream processing equipment.

As a result of the rapid heatup and short residence time features of the CS-SRT Process, extremely high coal throughputs are possible, of the order of 2000 lb/hr ft³ of reactor volume. For the case of a 250 MM SCFD

plant (and associated BTX production), only three reactors will be required, each capable of processing roughly 200 TPH coal through a 3 1/2 ft inside diameter.

Multi-Tube Reactor Concept

The combination of operating at a minimum hydrogen/coal recycle ratio under exothermic conditions can result in excessive temperature rises. This could result in loss of valuable benzene due to the adverse kinetics favoring benzene decomposition. Furthermore, since coal is being transported in dense-phase in a series of tubes to a multi-element injector assembly, a multi-tube reactor concept appears plausible.

Accordingly, a multi-tube, high pressure reactor with internal heat exchange is being developed. High pressure steam generation (1500 psi) can be used on the coolant side to maintain an isothermal tube wall. However, by using a coolant, such as a molten metal, heat can be absorbed non-isothermally while allowing control of the temperature profile within the reactor tube. This heat can also be used for high temperature heat exchange elsewhere in the process. Control of the reactor temperature profile will afford the plant operator the ability to vary product spectrum, conversion and turndown.

In one conceptual case, molten sodium is used to 1) control the reactor temperature profile and 2) limit tube wall temperature in contact with coal-hydrogen reactants to 1200°F. Other molten metal systems, such as molten lead or a 22% sodium/78% potassium eutectic, are also under consideration. The reactor design might consist of 12 alloy heat transfer tubes containing the reactants at system pressure. The tubes are immersed in the molten metal environment, also at system pressure, within a refractory-lined pressure vessel. The tubes will be about 12-inches in diameter and fit within the 3 1/2 ft. i.d. vessel. The major advantages of this reactor design are believed to be:

1. High coal throughput per volume of reactor;
2. Excellent turndown (8-100% full capacity);
3. Excellent reactor temperature control;
4. Overcomes heat transfer limitations of conventional pressure vessels;
5. Potential low risk to scaleup from pilot operations.

Molten Metal Heat Exchange

High thermal efficiency in the CS-SRT Process may be achieved by recovery of the high temperature heat of the reactor effluent. However, this has to be accomplished within the constraints of rapid quenching. This problem has been recognized and solved in the associated technology of ethylene production via steam cracking (10,11). In the latter reference, indications are that molten metal is used to rapidly quench steam cracker effluent while at the same time recovering high temperature heat. Quenching requirements for ethylene production are even more stringent than for the CS-SRT Process, of the order of 10-15 milliseconds compared to about 100 milliseconds.

A molten metal heat exchanger system has been conceptualized and applied to the CS-SRT Process. The bulk of the heat stored in the molten metal results from heat recovery in the reactor effluent quench exchanger. This heat can then be exchanged to provide the duties for recycle gas preheating, coal moisture removal and coal-benzene slurry drying. These total duties are equivalent to about 7.5 thermal efficiency points.

Our calculations have shown that this concept is feasible from both a process and a metallurgical standpoint. The concept utilizes molten metal technology already developed for the Clinch River breeder reactor.

Studies of the economics and optimization of this molten metal technology to the CS-SRT Process are continuing.

Conclusion

Bench-scale testing with a North Dakota lignite has shown that the CS-SRT Process scheme may be an attractive process for converting coal to pipeline quality gas, ethane and BTX liquids. Production of BTX liquids (and ethane feedstock for ethylene production) in the CS-SRT Process is a key factor in lowering the overall cost of services for producing SNG from coal. As a result of the rapid heatup, short residence time and rapid quench features of the process, a simple, high throughput reactor concept may be utilized without suffering loss in conversion and undesirable, secondary-decomposition reactions.

In order to maximize carbon and thermal efficiencies in the conceptual CS-SRT reactor, it is imperative to utilize a reactor design based on data acquired from a bench-scale unit that has been operated under conditions closely scalable to the commercial reactor concept.

Flowsheet studies of the CS-SRT Process have resulted in identifying several promising processing steps applicable to coal gasification:

1. Drying of coal using azeotropic benzene vaporization in an ebullated-bed contactor;
2. Multi-tube, high pressure, entrained-flow reactor concept with internal heat exchange and low scaleup ratio to commercial operation;
3. High temperature, heat recovery using molten metal heat exchange.

The next step of development of the CS-SRT Process would require about a 6-inch diameter, pilot-plant reactor capable of processing about 100 TPD coal. This pilot-plant would be one scaleup step away from a 12-inch diameter, single tube, commercial-type operation.

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Figure 1

COMPARISON OF REACTORS UTILIZED IN ENERGY RESEARCH LABORATORY

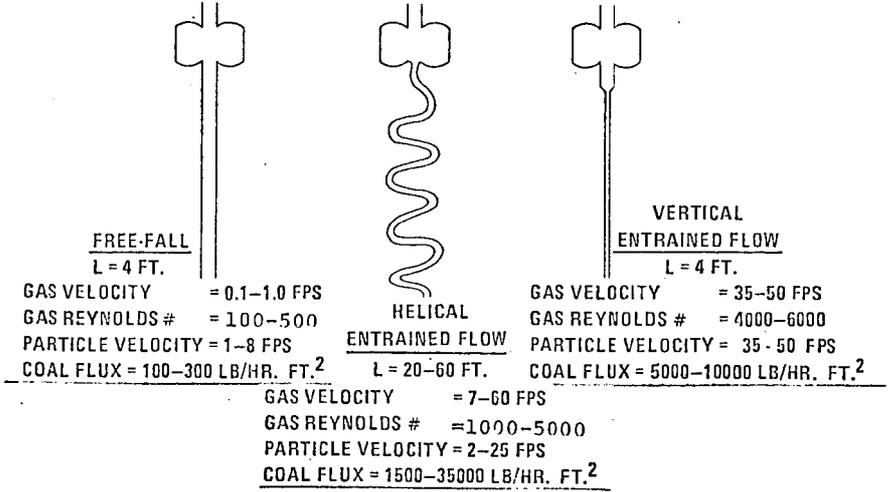


Figure 2

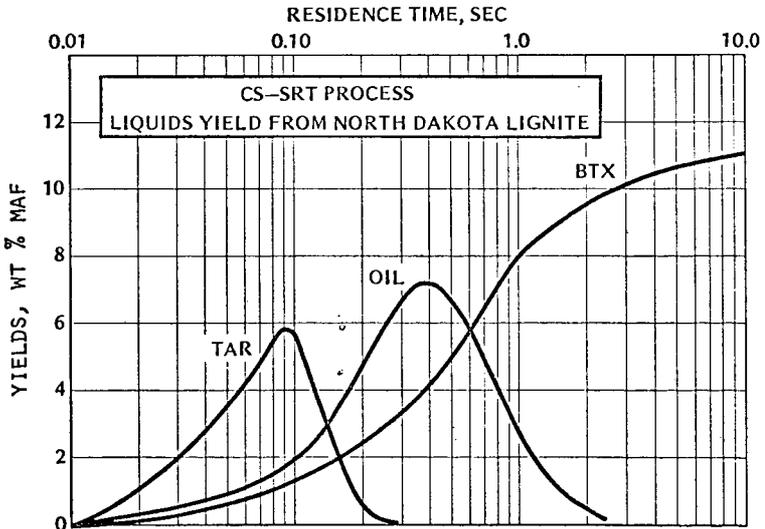


Figure 3

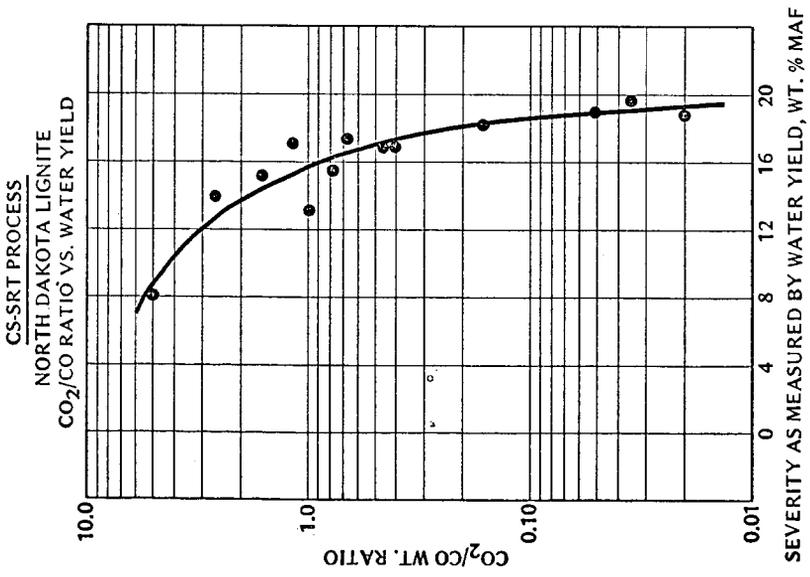


Figure 4

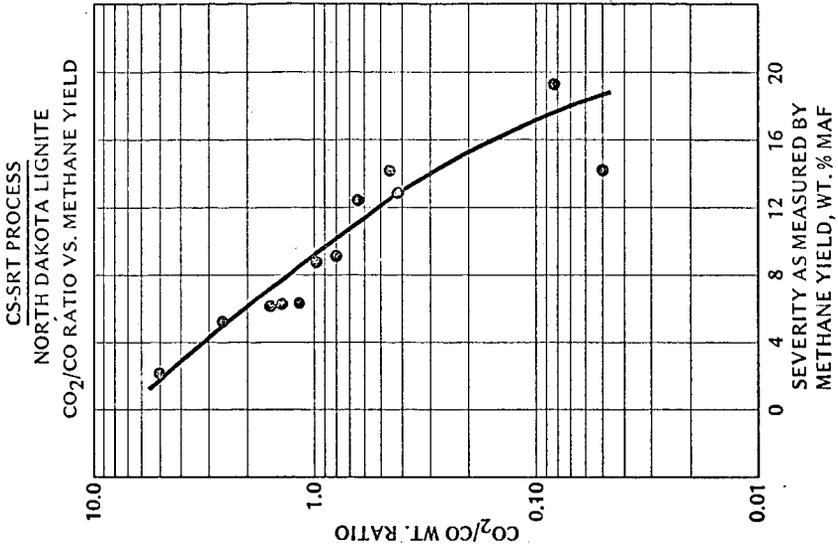


Figure 5

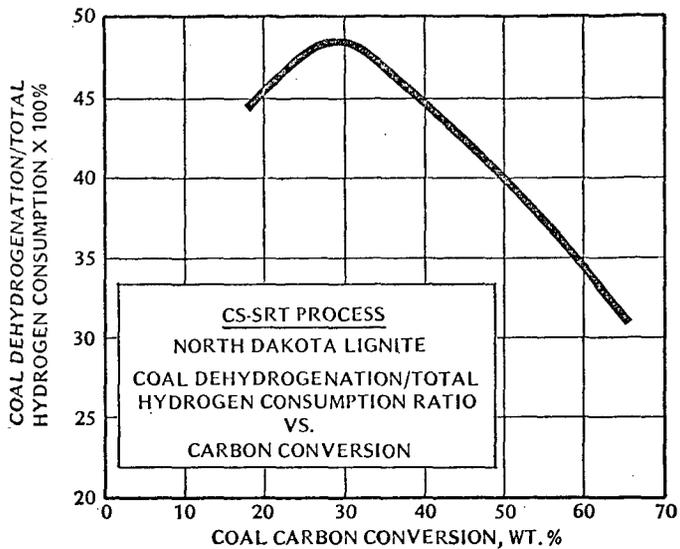


Figure 6

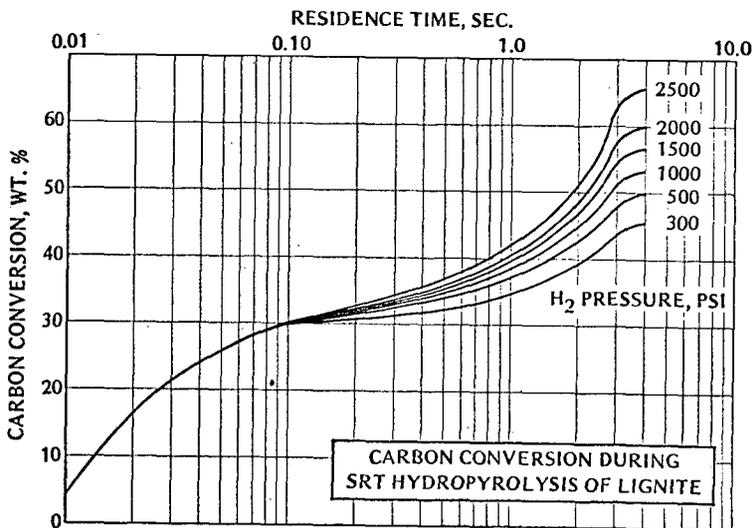


Figure 7
CS-SRT PROCESS
 Coal Drying

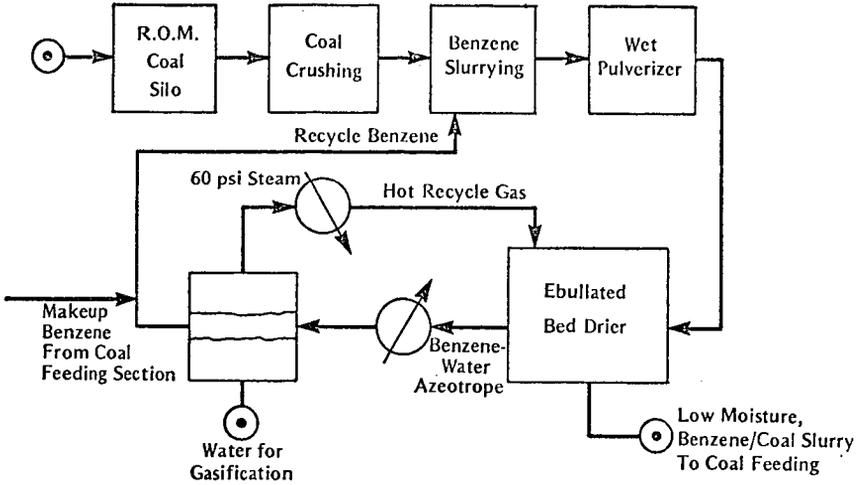


Figure 8

